THE QUEEN MARY.

BeWS-14

2019

14th International Workshop On Beryllium Technology

Long Beach, California, USA

24-25 October 2019

Organized under the auspices of
The IEA Technology Collaboration Program on Fusion Materials (FM TCP)

Proceedings of the 14th International Workshop on Beryllium Technology (BeWS-14)

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Proceedings of the 14th International Workshop on Beryllium Technology (BeWS-14)

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The 14th International Workshop on Beryllium Technology (BeWS-14) was held on 24-25 October 2019 at the Hotel Queen Mary in Long Beach, California, U.S.A., in conjunction with the 19th International Conference on Fusion Reactor Materials (ICFRM-19 in La Jolla, California). The workshop has generally been held once every two years since 1993.

These Proceedings of the BeWS-14 have been compiled from the documents generated by the workshop organizers, combined with the abstracts, manuscripts, and presentation files submitted by the participants in the event.

The BeWS-14 was organized by Be4FUSION LLC, with major technical support from KIT (Karlsruhe Institute of Technology) in Germany. Participants came mainly from the U.S., Germany, Japan, France, UK, Spain, Portugal, Korea, and Kazakhstan.

Keywords: Beryllium, Fusion Engineering, Irradiation Effects, Beryllide Intermetallic Compounds, Plasma-Facing Materials, Neutron-Multiplier Materials, Health and Safety, Chronic Beryllium Disease, Beryllium Sensitization

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Introduction & Executive Summary

The 14th International Workshop on Beryllium Technology (BeWS-14) was held on 24-25 October 2019 at the Hotel Queen Mary in Long Beach, California, U.S.A., in conjunction with the 19th International Conference on Fusion Reactor Materials (ICFRM-19 in La Jolla, California). The workshop has generally been held once every two years since 1993.

The objective of the workshop for scientists and engineers around the world is to share the results of their efforts to develop technology in the application of beryllium in fusion energy research and related fields, including health and safety. The BeWS-14 was organized by Be4FUSION LLC, with major technical support from KIT (Karlsruhe Institute of Technology) in Germany. Participants came mainly from the U.S., Germany, Japan, France, UK, Spain, Portugal, Korea, and Kazakhstan. Due to visa issues for entry into the U.S., there were no participants at this workshop from China or Russia.

The BeWS-14 program agenda consisted of three keynote presentations, which were followed by five technical sessions. There were 49 registered and paid attendees at the BeWS-14, an increase from the 35 attendees at the BeWS-13 in Japan in 2017.

The keynote presentations began with an historical retrospective on the BeWS series, given by Dr. Glen Longhurst (Idaho National Lab, retired). He was followed by Dr. Milan Zmitko of F4E, who reported on the status of Be neutron multiplier materials for the ITER Test Blanket Module (TBM) program. The final keynote was presented by Dr. Hiroshi Kawamura, formerly of JAEA in Japan, who presented his ideas on recycling of irradiated beryllium for use in fusion research reactors, which had its peak activities in 2007-2012. Drs. Longhurst and Kawamura are two of the original founders of the Beryllium Workshop, and the BeWS-14 marked the first time they had attended together in many years.

The five technical sessions featured presentations on Alternative Blanket Concepts and Beryllides, Beryllium Materials under Extreme Conditions, Industrial Fabrication, Modeling, and EH&S (Environmental, Health & Safety). EH&S topics had particular emphasis in the program due to strong interest and concerns at ITER.

The Prof. Mario Dalle Donne Memorial Award (MDDMA) was established at the BeWS-11 in Barcelona to recognize researchers with outstanding achievements in beryllium-related research. This year, the BeWS International Organizing Committee (IOC) members unanimously agreed that Dr. Jae-Hwan Kim of QST in Japan was the deserving recipient, based on his many outstanding achievements in beryllium-related research over the past decade. Special lifetime contribution awards were presented to Drs. Longhurst, Kawamura, and Anton Möslang of KIT.

The final BeWS-14 program is included near the beginning of these Proceedings to introduce the abstracts and presentations that follow. A list of the registered participants may be found in the Appendices of this document. It should be noted that in cases where the presentation title herein is different than what is listed in the program, that is because the author changed it prior to delivering it at the workshop.

Chris DORN
BeWS-14 Chair
Oxford, June 2021

BeWS-14 Final Technical Program

The program was organized by the BeWS-14 Technical Chair, Dr. Pavel Vladimirov of KIT in Germany. This is the version of the program distributed to the participants. Some presentation titles changed by the time of the workshop.

Title:	The 14th International Workshop on Beryllium Technology
Location:	Hotel Queen Mary, Long Beach, CA, USA
Date:	24-25 October 2019

24-Oct-2019

Start	End	Time	Item	Presenter	Session	Chair
8:30 AM	8:40 AM	0:10	Welcome	Christopher Dorn	**0_Welcome**	
8:40 AM	9:00 AM	0:20	Evolution of the BeWS-Series	Glen Longhurst	**1 Key Notes**	C. Dorn
9:00 AM	9:20 AM	0:20	Current Status in Development and Qualification of Beryllium Materials for the EU HCPB	Milan Zmitko		
9:20 AM	9:40 AM	0:20	Recycle Business Challenge of activated Beryllium Neutron Reflectors used at the Research Reactors	Hiroshi Kawamura		
9:40 AM	10:10 AM	0:30	Break & Exhibition			
10:10 AM	10:30 AM	0:20	On beryllium for tritium breeding blankets: Resource availability, alternatives and determining the optimum route to commercial fusion	Richard Pearson	**2_Alternative blanket concepts & beryllides**	H. Kawamura & G. Longhurst
10:30 AM	10:50 AM	0:20	Overview of beryllium and beryllide R&D as neutron multipliers in QST	Masaru Nakamichi		
10:50 AM	11:10 AM	0:20	Development, characterization & modelling of neutron multiplier materials at KIT	Pavel Vladimirov		
11:10 AM	11:30 AM	0:20	R&D on Vanadium Beryllides as an advanced NM for DEMO Fusion applications	Jae-Hwan Kim		
11:30 AM	11:50 AM	0:20	Microstructure and properties of intermetallic Be12Ti fabricated by arc melting or hot isostatic pressing	Ramil Gaisin		
11:50 AM	12:10 PM	0:20	Hot extrusion of Be-Ti powder	Aniceto Goraieb		
12:10 PM	1:40 PM	1:30	Lunch			
1:40 PM	2:00 PM	0:20	Radiation induced formation gas bubbles in beryllium after neutron irradiation up to 6000appm helium production	Michael Klimenkov	**3_Beryllium materials under extreme conditions**	P. Vladimirov & M. Nakamichi
2:00 PM	2:20 PM	0:20	Experimental investigation of radiation damage effects in beryllium: updates on recent results obtained on proton, neutron and He-ions irradiated samples	Slava Kuksenko		
2:20 PM	2:40 PM	0:20	Investigation of high-dose irradiated beryllium microstructure including STEM-EELS analysis of He/T bubbles	Nikolai Zimber		
2:40 PM	3:00 PM	0:20	Tritium release and retention behavior of beryllium and titanium beryllide irradiated up to high neutron doses	Vladimir Chakin		
3:00 PM	3:30 PM	0:30	Break & Exhibition			
3:30 PM	3:50 PM	0:20	Deuterium retention on Be-C-O films: in-situ versus ion implantation loading	Eduardo Alves	**cont'd**	
3:50 PM	4:10 PM	0:20	Oxidation of neutron irradiated Be pebbles	Marta Dias		
4:10 PM	4:30 PM	0:20	Compatibility of beryllium & beryllides with structural materials	Rolf Rolli		
4:30 PM	4:50 PM	0:20	Comparison of the thermal shock damage on beryllium induced by electron beam and laser	Benjamin Spilker		
4:50 PM	4:50 PM	0:00	ADJOURN			
Total		**8:20**				

7:00 PM **Conference Dinner**

25-Oct-2019

Start	End	Time	Item	Presenter	Session	Chair
8:30 AM	8:50 AM	0:20	Overview of the US Beryllium Industry	Keith Smith	**4_Industrial Fabrication**	V. Chakin & C. Dorn
8:50 AM	9:10 AM	0:20	Status of the BP1 Pebble Production	Keigo Nojiri		
9:10 AM	9:30 AM	0:20	Studies for production of billets and articles from tantalum and titanium beryllides	Yevgeniy Frants		
9:30 AM	9:50 AM	0:20	Simple process for mass production of metal Be true sphere	Yuri Natori		
9:50 AM	10:20 AM	0:30	Break & Exhibition			
10:20 AM	10:40 AM	0:20	Hydrogen coverage regimes on (0001) Be surfaces	Christopher Stihl	**5 Modeling**	M. Zmitko
10:40 AM	11:00 AM	0:20	Beryllium Lifetime Analysis for Research and Test Reactors	Gary Solbrekken		
11:00 AM	11:20 AM	0:20	Valence electron structure of the beryllides using soft X-ray emisssion spectroscopy	Keisuke Mukai		
11:20 AM	11:40 AM	0:20	TEM analytical study and ab initio simulation of impurities in beryllium	Pavel Vladimirov		
11:40 AM	1:10 PM	1:30	Lunch			
1:10 PM	1:30 PM	0:20	Beryllium: A Review of Uses, Potential Health Effects and Impacts of Regulatory Activities	Theodore Knudson	**6_Safety**	E. Alves & J.H. Kim
1:30 PM	1:50 PM	0:20	Comparison of collection efficiencies of Whatman n°1 filter paper and Ghost wipes for loose beryllium surface contamination	Mirjana Damjanovic		
1:50 PM	2:10 PM	0:20	A Review of Beryllium Combustibility and Explosivity and Potential Risks in Fusion Energy Production	Marc Kolanz		
2:10 PM	2:30 PM	0:20	Relationship between surface contamination and airborne particulate of hazardous materials	Beth Walker		
2:30 PM	3:00 PM	0:30	Break & Exhibition			
3:00 PM	3:20 PM	0:20	Identifying Risks and Diagnosis of CBD	Kathryn Creek	**cont'd**	
3:20 PM	3:40 PM	0:20	Key Elements of a Successful Beryllium Control Program	Kathryn Creek		
3:40 PM	4:00 PM	0:20	Surficial Levels of Beryllium and the Relationship to Worker Exposures and Potential Health Effects	Marc Kolanz		
4:00 PM	4:20 PM	0:20	Overview of the BeYOND workshop series	Aniceto Goraieb	**7_BeYOND**	C. Dorn
4:20 PM	4:30 PM	0:10	Closing remarks	Christopher Dorn	**8_Closing remarks**	
4:30 PM	4:30 PM	0:00	ADJOURN			
Total		**8:00**				

BeWS International Organizing Committee

Region/Country	Name	Organization
Asia/Japan	Masaru NAKAMICHI	National Institutes for Quantum and Radiological Science and Technology (QST)
Europe/Germany	Pavel VLADIMIROV	Karlsruhe Institute of Technology (KIT)
Kazakhstan	Yevgeniy FRANTS	Ulba Metallurgical Plant JSC
North America/U.S.A.	Chris DORN	Be4FUSION LLC
Russian Federation	Radmir GINIYATULIN	Federal State Unitary Enterprise Efremov Scientific Research Institute of Electrophysical Apparatus (NIIEFA Efremov)

BeWS-14 Sponsor Organizations

The workshop could not have gone as well as it did without the support of the following sponsors:

Materion Brush Inc. Elmore, Ohio, U.S.A.	NGK Insulators, Ltd. Handa, Aichi, Japan
KAKEN Inc., Laboratory & Test Center Mito, Ibaraki, Japan	KIT - Karlsruhe Institute of Technology Eggenstein-Leopoldshafen, Germany
KBHF - Karlsruhe Beryllium Handling Facility Eggenstein-Leopoldshafen, Germany	Be4FUSION LLC Upland, California, U.S.A.

Session 1: Keynote Presentations

Evolution of the Beryllium Workshop Series

G. Longhurst (INL & Southern Utah University, USA) et al.

Evolution of the Beryllium Workshop Series

Glen R. Longhurst[1] and Hiroshi Kawamura[2]

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[2]*Chiyoda Technol Corporation, Tokyo, Japan*

Abstract

The IEA International Beryllium Workshop series is an outgrowth of a meeting of specialists held at the Idaho National Laboratory in 1989. At a subsequent satellite meeting to ICFRM-5, a decision was made to apply to the International Energy Agency for sponsorship of a continuing workshop series. Meetings have been held since then at nominally two-year intervals in various places around the world. Though initially focused on issues relevant to fusion, the ITER project in particular, the scope of the workshops has expanded to include issues of beryllium technology associated with fission reactors as well. Participation in these workshops has been more or less consistent from one workshop to the next. Technical issues and concerns identified in the first meeting have continued to be pursued. New forms of beryllium have been produced including pebbles, and different compounds such as beryllides and intermetallics have been explored as a means of improving performance in the nuclear environment. There is still much to do including finding pathways for disposal and/or recycling of irradiated beryllium.

Keywords: Beryllium Workshops, History and Evolution, Beryllium Nuclear Technology

1 Background

Beryllium has been used in nuclear applications virtually from the beginning of the development of peaceful applications of nuclear energy. It forms an integral part of many research fission reactors where it serves as a neutron reflector and multiplier in research reactor cores. More recently in fusion technology it has been planned on for its excellent qualities as a plasma-facing material and for its neutron reflection and multiplication characteristics in the tritium breeding blanket.

Processes for manufacturing beryllium have developed over the years as efforts have been made to increase toughness and ductility by using powder metallurgy techniques. Issues arising from its use in nuclear applications relate to swelling and alteration of mechanical properties due to neutron irradiation. Various techniques for compaction and sintering have been explored. To minimize issues of swelling and cracking of beryllium under irradiation, the production of beryllium in small pebble form has been explored. Another concern is finding ways of forming and joining beryllium to substrates that will survive the nuclear environment. Because of nuclear transmutation products such as tritium and long-lived activation products from uranium and other naturally occurring heavy metals, disposal and possible recycling of irradiated beryllium are also of significant concern in both fission and fusion applications. Beryllium is toxic to some individuals and beryllium dust is pyrophoric, so there are health and safety concerns associated with its use.

2 Workshop Genesis

As with many sensitive technologies, centers of excellence and capability for beryllium in nuclear applications are somewhat isolated and localized. In the mid-1990s there was a growing sense that there should be communication and collaboration between these groups if the goals for beryllium in this industry were to be realized. The first gathering of specialists interested in nuclear applications of beryllium, especially for fusion, was held at what is now the Idaho National Laboratory on 11 September 1989. This was the grandparent, so to speak, of the BeWS series we now have. There were 22 participants. Names and institutions of these persons are listed in Table 2.1.

Table 2.1: Participants at the first meeting of specialists interested in fusion applications of beryllium, held 11 September 1989.

D. Baldwin	Battelle Pacific Northwest Laboratory
C. K. Dorn	Brush Wellman, Inc.
H. Matsushima	Brush Wellman (Japan), Ltd.
R. A. Anderl D. E. Ardary S. Brereton L. C. Cadwallader J. G Crocker J. S. Herring D. F. Holland G. R. Longhurst L. G. Miller D. Mousseau	EG&G Idaho, Inc.
H. Kawamura T. Kurasawa H. Yoshida	Japan Atomic Energy Research Institute
T. Suzuki	Kawasaki Heavy Industries, Ltd.
I. Hitani	Mitsubishi Atomic Power Industry, Inc.
K. Nishida	NGK Insulators, Ltd.
J. M Beeston	Private Consultant
R. Causey	Sandia Livermore National Laboratory
K. Ashibe	Toshiba Corporation, Research and Development Center

The main objective of that meeting was to bring together persons with experience and interest in research that was being conducted on beryllium, particularly aspects related to its use in nuclear facilities. Ten presentations were made on topics of discussion that included health and safety aspects of working with beryllium, production of beryllium pebbles, storage and release characteristics of the radioactive tritium bred in the beryllium by neutron transmutations, swelling and mechanical property changes caused by the irradiation, and storage and disposition of irradiated beryllium.

As a follow-on to the 1989 meeting in Idaho, and to broaden the participation in these discussions, it was decided to hold a satellite meeting to the Fifth International Conference on Fusion Reaction Materials (ICFRM5) at Clearwater Beach, Florida. That meeting was held as a Beryllium Technology Workshop on November 20, 1991.

The objectives of that workshop were to (a) bring researchers, developers, designers, and managers together; (b) get the "flavor" of work in progress on beryllium at the various institutions; (c) identify areas where additional effort is most needed; and (d) communicate the results of these discussions to participants and others with financial or technical resources to pursue the tasks identified [1].

Forty-two persons attended including representatives from major beryllium manufacturers and research laboratory personnel from laboratories in Canada, England, France, Germany, Japan, and the United States. Designers from major fusion projects attended as did private consultants.

Twelve formal presentations were made. Following the prepared presentations there was a group discussion of the areas where additional research was needed or planned and of the relative importance to fusion of those areas. That discussion resulted in the following as the most important needs, in order of priority.

1. Dimensional stability under irradiation
2. Changes in mechanical properties, notably ductility and creep resistance under irradiation
3. Thorough understanding of tritium interactions with beryllium

Other important issues, not ranked, included manufacturing capabilities and functional integrity of such forms as pebbles and low-density blocks, means to dispose of or recycle irradiated beryllium, bonding of beryllium to substrates and plasma-spraying, alternative alloys and intermetallics for improved performance. Personnel protection during installation and maintenance was another issue of concern.

Perhaps the most significant action at this workshop was a conversation that took place following the group discussions. Prof. Mario Dalle Donne of Kernforschungszentrum Karlsruhe, Dr. Hiroshi Kawamura of Japan Atomic Energy Research Institute ORAI, Dr. Vladimir Shestakov of Kazakh National State University and Dr. Glen Longhurst of the Idaho National Engineering Laboratory met together to consider a proposal of Prof. Dalle Donne that application be made to the International Energy Agency (IEA) to sponsor regular meetings such as this one as an official international meeting series. Prof. Dalle Donne volunteered to lead in making that application, working with acquaintances who were part of the IEA. That proposal was prepared and presented to the IEA. It was accepted under the IEA Implementing Agreement for a Programme of Research and Development on Fusion Materials, and the Workshop on Beryllium for Fusion Applications was born. This 1991 workshop at which the decision was made to apply to the IEA has been referred to as BeWS-0.

At that time, the main focus of the workshop series was on fusion applications of beryllium, notably those associated with tokamaks and with ITER in particular. It was decided that the International Organizing Committee (IOC) for the Beryllium Workshop series would be comprised of representatives from the then four major ITER parties: the European Union, Japan, the Russian Federation, and the United States. The principal responsibility for the various workshops in the series would rotate among the members of the IOC who would then recruit a local organizing committee from institutions falling functionally within those respective ITER parties. It was also decided that it would be most convenient if the Beryllium Workshops where held convenient in time and location to the International Conference on Fusion Reactor Materials (ICFRM). It was the intent that following each Workshop meeting, a proceedings publication would be produced containing the written versions of the papers presented at the Workshops. That goal has not always been achieved. In some years only the presentation files themselves have been available.

3 Beryllium Workshops

3.1 Venues and Participation

The first IEA sponsored International Workshop on Beryllium for Fusion Applications (BeWS-1) was held 4-5 October 1993 at Kernforschungszentrum Karlsruhe, Germany. Since then, twelve additional meetings have been held at nominally two-year intervals, generally in concert with the ICFRM. Table 3.1 lists the dates, numbers of attendees, numbers of papers presented, numbers of institutions contributing to those presentations, and the locations of these meetings.

Table 3.1: Dates, locations, and participating institutions for the IEA Beryllium Workshop meetings.

Meeting	Year	Attendees	Papers	Institutions	Location
BeWS-0	1991	42	12	12	Clearwater Beach, FL, USA
BeWS-1	1993	52	20	23	Karlsruhe, Germany
BeWS-2	1995	45	37	26	Jackson Lake Lodge, WY, USA
BeWS-3	1997	68	38	38	Mito, Japan
BeWS-4	1999	69	38	41	Karlsruhe, Germany
BeWS-5	2001	46	27	31	Moscow, Russia
BeWS-6	2003	54	49	48	Miyazaki, Japan
BeWS-7	2005	45	27	33	Santa Barbara, CA, USA
BeWS-8	2007	56	22	27	Lisbon, Portugal
BeWS-9	2009		16	24	Almaty, Kazakhstan
BeWS-10	2012	64	30	28	Karlsruhe, Germany
BeWS-11	2013	41	35	20	Barcelona, Spain
BeWS-12	2015	44	30	44	Jeju, South Korea
BeWS-13	2017	35	29	46	Chiba, Japan

In addition to the technical discussion, each meeting has featured a cultural experience at or near the venue of the meeting. Consistently, workshop banquets have been held for participants and guests. Frequently these banquets have featured entertainment representative of the culture and history of the region. There have also been tours of culturally significant places such as shrines, museums and centers for arts and crafts.

3.2 Changes

The name and scope of the BeWS series has changed slightly over the years since its inception. BeWS-1 was titled, "IEA Implementing Agreement for a Programme of Research and Development on Fusion Materials Workshop on Beryllium for Fusion Applications." The name for BeWS-2 was shortened to "2nd IEA International Workshop on Beryllium Technology for Fusion." That name continued through BeWS-6, but it was further modified for BeWS-7 to "7th IEA International Workshop of Beryllium Technology." This was in consequence of the broadening of the scope of the meeting to include non-fusion applications, notably those in fission reactors and other fields of interest.

The IOC voted at the BeWS-10 meeting to create an award for excellence in beryllium research. Called the Mario Dalle Donne Memorial Award (MDDMA), commemorating workshop founder, Prof. Mario Dalle Donne, the award recognizes outstanding achievement in the field and is intended to stimulate further excellence. There are two parts to the award. One is a crystal pyramid with the recipient's name and recognition engraved on it.

The second is the recipient's name engraved on a plaque attached to a travelling trophy that is retained by the recipient until the next BeWS meeting (Figure 3.1). The award was first given at BeWS-11 in Barcelona, Spain and has been awarded at each meeting since. The first award was to Dr. Masaru Nakamichi of the Secretariat of Nuclear Safety Commission, Japan. The second and third were received by Dr. Vladimir Chakin and Dr. Jörg Reimann, respectively, of the Karlsruhe Institute of Technology (Figures 3.2 and 3.3).

Figure 3.1: Travelling trophy for the Mario Dalle Donne Memorial Award.

Figure 3.2: Dr. Vladimir Chakin receiving the Mario Dalle Donne Memorial Award at BeWS-12 [2].

Figure 3.3: Dr. Jörg Reimann receiving the Mario Dalle Donne Memorial Award at BeWS-13 [3].

4 Technical Progress

As previously noted, major concerns or uncertainties related to beryllium use in fusion applications identified in the 1991 meeting (BeWS-0) were

- Dimensional stability under irradiation
- Changes in mechanical properties, notably ductility and creep resistance under irradiation
- Thorough understanding of tritium interactions with beryllium
- Manufacturing capabilities and functional integrity of such forms as pebbles and low-density blocks
- Means to dispose of or recycle irradiated beryllium
- Bonding of beryllium to substrates
- Plasma-spraying
- Alternative alloys and intermetallics.
- Personnel protection during installation and maintenance

As the BeWS series has progressed, the specific categorization of papers has changed slightly from one meeting to the next, but in general the papers presented fall within the following groups. The numbers in parentheses following those topical areas are the total numbers of papers falling generally into those areas that have been presented in the first thirteen Workshops.

1. Applications for Advanced Machines (40)
2. Production and Characterization (96)
3. Chemical Compatibility and Corrosion (25)
4. Forming and Joining (30)
5. Plasma and Tritium Interactions (51)
6. First Wall Issues (31)
7. Neutron Irradiation Effects (53)
8. Health and Safety Issues (20)
9. Disposal and Recycling (8)

The scope of the first two groups is rather broad. Developing production techniques for foams, pebbles, beryllides, and intermetallics and establishing their properties and performance characteristics all fall within the second group for example. It should be noted that in several instances the technical scope of a paper could have fallen into more than one category. No effort was made here to resolve those commonalities. Each paper was assigned one category in the development of these numbers.

Figure 4.1 is a display of the numbers of papers in each category broken down by category and workshop in which they were presented.

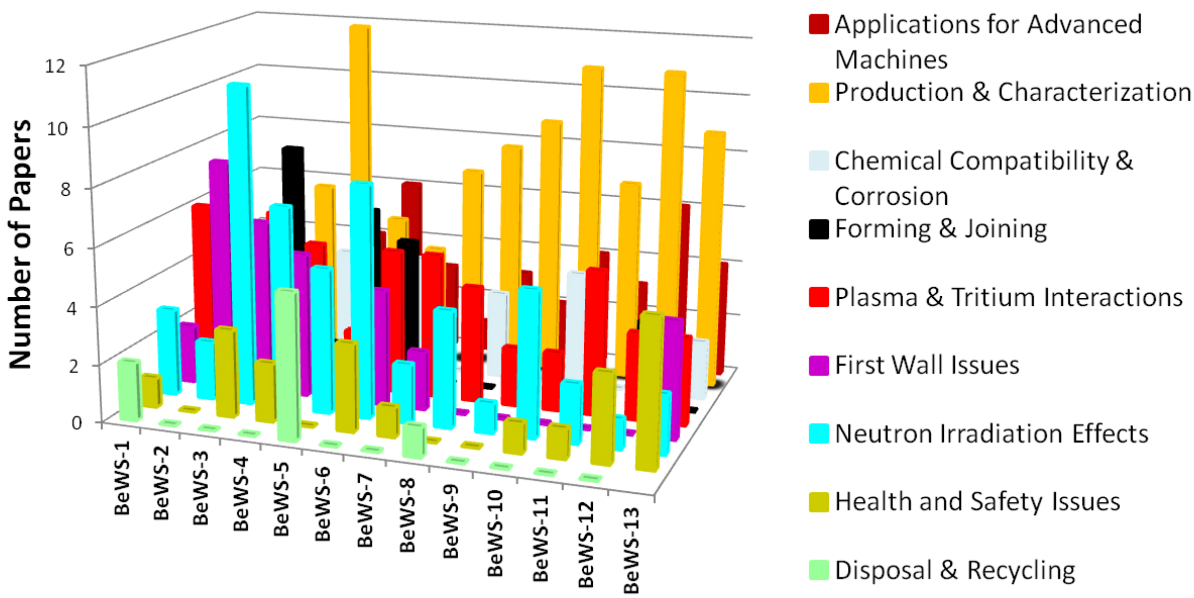

Figure 4.1: Activity of beryllium technology research as gauged by the numbers of papers presented at the various BeWS workshops in nine different research categories.

Several observations may be made regarding these data. First, as may be expected, the level of activity in all of the categories varies from year to year. One contributor to that is the long-term nature of some research efforts which do not produce results on predictable schedules. Also, works are performed by various institutions, some of which move into the research area and others leave.

By far the category of greatest activity is the Production & Characterization area. Considerable effort has gone into producing different forms of beryllium including pebbles, beryllides, and intermetallics and in characterizing the mechanical, thermal, and material interaction performance of these. Second place is a virtual tie between Neutron Irradiation Effects and Plasma and Tritium Interactions. These two areas both involve the response of beryllium to perturbations from the outside such as particle collisions, gas production and swelling, and intense heat loads. Considerable testing has been done in fusion-environment simulators and in nuclear reactors to evaluate the response of various beryllium forms and compositions to these environments.

The areas where the least amount of work has been reported are Health and Safety research and Disposal and Recycling of irradiated beryllium. Although both are important, programmatic urgency is probably less for these categories compared with the need to find technological solutions to design and performance problems involving beryllium components to meet development needs of ITER and other future machines.

The following discussion highlights a few selected accomplishments that have been reported just to illustrate progress that has been made.

4.1 Neutron Irradiation Effects

4.1.1 Dimensional Stability

It had been known for many years that beryllium subject to neutron irradiation experiences swelling in consequence of the (n,2n) and other reactions that produce ^{4}He, ^{3}H and other products. While the (n,2n) reaction is highly desirable for neutron multiplication, a principal reason for using beryllium in nuclear applications, the associated swelling of beryllium parts in large fission reactors results in the need for periodic core internals change outs which are costly and time consuming. Swelling in fission applications was sufficiently well characterized to allow rough estimates of component lifetime, but because of the substantial difference in neutron energy spectrum between fission and fusion environments, there was considerable uncertainty as to the extent and rate at which beryllium will swell under fusion conditions.

There is still a lack of information on irradiation effects on most grades of beryllium. It is now known that hydrogen isotopes and helium tend to collect in small bubbles in metallic beryllium. Swelling of irradiated beryllium is mainly due to the formation of such bubbles, whose volume is determined by what is effectively surface tension at the interface between the gas molecules and the matrix. As the bubble size increases, the pressure of the gas within the bubble decreases resulting in a greater volume per gas molecule. Hence, anything that can inhibit the migration of gas atoms produced by transmutation to coalesce into gas molecules in bubbles would improve swelling resistance. Small grain size and the presence of finely distributed pinning points, such as BeO inclusions, would help retard swelling [2].

As of the last workshop, BeWS-13, there were still no comparison data for swelling in irradiated S-65, TGP-56FW, and CN-G01, beryllium grades under consideration for ITER [3]. It is known that swelling takes place faster at higher temperatures, due in part to the greater mobility of gas atoms produced by the nuclear reactions to move through the lattice to bubble nucleation sites. Grades with the smallest grain size and highest BeO content were found to have the lowest amount of high temperature swelling [4]. Grain boundaries and inclusions also tend to pin the flow of dislocations in the material making it stiffer and more resistant to swelling. Swelling mechanisms as then understood were incorporated into the ANFIBE code, developed by Scaffidi-Argentina et al. [5] in 1998, but code results remain to be validated under fusion relevant conditions.

4.1.2 Changes in Mechanical Properties

In 1991, beryllium was considered for use as tiles on plasma-facing surfaces, as pebbles or blocks inside containers made of other materials in breeder blankets, and as evaporated coatings such as had been used in the Joint European Torus (JET). Particularly in designs involving manufactured shapes, mechanical properties such as thermal expansion coefficient, Young's modulus, ductility, yield and ultimate strength, and thermal conductivity are important in design and effect performance and service lifetime. The effects of the fusion neutrons, plasma ions, and thermal environments on these properties of beryllium generally and on the various grades of beryllium were largely unknown at that time.

An extensive study of irradiation effects on five different grades of beryllium, three manufactured by hot extrusion and two by hot isostatic pressing was reported by Chakin et al in 2001 [6].

Mechanical property changes of beryllium pebbles were studied in the HIDOBE-02 experiment in the High Flux Reactor (HFR) at Petten, Netherlands between 2005 and 2011 [7]. Constrained and unconstrained beryllium pebbles made using the rotating electrode process ranging from 1 mm to 2 mm in diameter were irradiated at average temperatures from 370°C to 650°C for 1,247 full power days, resulting in damage doses of 21 to 37 displacements per atom (dpa). Post-irradiation hardness was found to decrease by about 50 percent at the highest irradiation temperatures. Pebbles irradiated and tested for creep at lower temperatures showed lower creep rates and crack formation compared with higher strain rates and no crack formation in pebbles irradiated and tested at higher temperatures.

One interesting discovery was that when vacuum hot-pressed S-65 Grade beryllium is used for plasma facing surface tiles, the orientation of the tile face relative to the direction of axial compression makes a difference. The tensile strength and elongation in the direction of compression are lower than in the transverse direction. Thus, by making sure the normals to the tile faces are in the transverse direction relative to the compression axis, cracks that develop will tend to result in effectively new castellations rather than allowing layers to break free of the tile and fall into the plasma chamber [8].

4.2 Tritium Interactions

It was earlier noted that ^{3}H (tritium) is one of the products of neutron transmutation in beryllium. In general, there is about an order of magnitude less tritium produced than helium depending on the local neutron energy spectrum. Some tritium will be absorbed from the plasma by plasma-facing beryllium. Because of its radiotoxicity, tritium is of concern in accidents, in storage after irradiation, and possibly during maintenance. While the He (both ^{3}He and ^{4}He) is insoluble in the beryllium and must exist in bubbles or as interstitial atoms, it was not known whether there is a beryllium hydride or how the tritium exists in beryllium. Knowledge of its release characteristics on heating the beryllium was just beginning to come forth. Now it is known that most of the tritium resides as hydrogenic molecules in the helium bubbles [9,10], though some also may be associated with hydroxides near inclusions.

4.3 Manufacturing Capabilities

4.3.1 Pebbles

At BeWS-5, held in 2001, several study results on beryllides were presented by the Japanese team. Then, an energetic study on new beryllium materials more relevant to fusion than to fission was started [11, 12]. $Be_{12}V$ and $Be_{12}Ti$ have been considered as attractive material forms for neutron multiplication in tritium breeding blankets because of their higher melting temperature, greater resistance to oxidation, low neutronic activation, and lower retained gas inventories than either Be or their alloying elements alone. However, they are brittle [13]. Among the developments since BeWS-0 was successful demonstration of the manufacturing of $Be_{12}V$ pebbles using the Rotating Electrode Method. $Be_{12}V$ single phase pebbles demonstrated a lower chemical reactivity than Be pebbles, and retained tritium is two orders of magnitude less than that of Be [14].

4.3.2 Joining

The present ITER design calls for approximately 700 m^2 of First Wall panels clad with small castellated beryllium tiles that must be brazed to a CuCrZr substrate. Significant progress has been made in developing the brazing process. Testing reported in 1991 showed considerable scatter in results [15]. Braze alloys have been optimized and the heating cycle has been changed from electron-beam heating to induction heating to minimize the thickness of the brittle intermetallic layer that forms at the braze joint and reduce re-crystallization in the beryllium. This results in greater strength and the ability to better withstand thermal and mechanical shocks [16].

4.4 Disposal and Recycling

It is necessary to develop beryllium reprocessing technology for effective resource use and to aid in disposing of irradiated beryllium. From that point of view, preliminary reprocessing tests were performed using un-irradiated and irradiated beryllium by Kawamura et al. in 1995 [17]. After a workshop on beryllium disposal and recycling held at the INEL in 2002, it was concluded that at that time there was in the United States no identified pathway to disposal for irradiated beryllium [18]. Most of the irradiated beryllium from fission reactors is held in temporary storage. Some beryllium components from research reactors at the Idaho National Laboratory had been buried as low-level waste in the Radioactive Waste Management Complex there. Routine surveys found tritium plumes above the locations where these items were buried, an indication that they were corroding and releasing incorporated tritium and other hazardous radionuclides into the soil. One approach to containment of these releases was encapsulation in a paraffin wax product [19]. Work in Belgium has addressed a number of disposal techniques including cementation, bituminization, vitrification, phosphatization, and direct disposal [20]. Several proposals have been made for developing one or more processes to remove the long-lived radioisotopes and tritium from the beryllium. A prominent one involves reacting irradiated beryllium with chlorine to produce a chemically pure vapor which is then reacted to regenerate the metal [21], and work is continuing in that direction [22] but the process remains to be fully implemented.

5 Conclusion

The Beryllium Workshop series began as an informal meeting of specialists in beryllium technology pertaining to nuclear applications. Through the efforts of Prof. Mario Dalle Donne and others, the meeting series was accepted for sponsorship by the International Energy Agency (IEA). Thirteen workshops in this series have been held commencing in 1993 and coming at nominally two-year intervals, typically near in location and in time to the International Conference on Fusion Reactor Materials.

These workshops have provided a valuable forum for sharing results of work in this field. They have also pointed desirable directions for research and development of the science and technology supporting the use of beryllium in nuclear applications, especially in fusion. Participants come from many countries, principally those represented or participating in the international ITER program. Attendance at the workshops has varied but is typically in the range of 40 to 60 persons. The number of institutions with persons contributing as authors or co-authors of the papers presented is approximately the same. Publication of the proceedings of these workshops is a valuable and useful archive for the accomplishments of the participants.

Besides providing a valuable technical forum, workshop participants have been treated to outstanding cultural experiences in the venues where workshops have been held. The organizers are to be congratulated for the great service they have performed.

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Evolution of the Beryllium Workshop Series

Glen Longhurst – Emeritus Faculty, Southern Utah University
Hiroshi Kawamura – Chiyoda Technol Corporation

Before BeWS there was a Specialists Meeting at the INL in 1989

Objective — Bring together persons with experience and interest in research that was being conducted on beryllium, particularly aspects related to its use in nuclear facilities.

Ten presentations were made on

- Health and safety aspects of working with beryllium
- Production of beryllium pebbles
- Storage and release of tritium bred in the beryllium by neutron transmutations
- Swelling and mechanical property changes caused by the irradiation
- Storage and disposition of irradiated beryllium.

Twenty two participants came from

- Battelle Pacific Northwest Laboratory
- Brush Wellman, Inc.
- EG&G Idaho, Inc.
- Japan Atomic Energy Research Institute
- Kawasaki Heavy Industries, Ltd.
- Mitsubishi Atomic Power Industry, Inc.
- NGK Insulators, Ltd.
- Sandia Livermore National Laboratory
- Toshiba Corporation, Research and Development Center

Beryllium Technology Workshop

Held 20 November 1991 at Clearwater Beach, Florida

Objectives (continued from 1989 Specialists Meeting)

- Bring together researchers, developers, designers, managers
- Get the "flavor" of work in progress
- Identify areas where work was most needed
- Communicate results to potential performers

Forty-two persons attended

Meeting was effectively BeWS-0

Needs Identified at the Workshop

1. Dimensional stability of Be under irradiation
2. Changes in mechanical properties due to irradiation
3. Understand tritium interactions with Be
4. Manufacturing capabilities for diverse Be forms
5. Means for disposal or recycling of irradiated Be
6. Bonding Be to substrates
7. Alternative alloys and intermetallics

The most important outcome was the decision to apply to the IEA for sponsorship of a regular workshop series.

Mario Dalle Donne — Hiroshi Kawamura — Vladimir Shestakov — Glen Longhurst

Working with three others, Prof. Dalle Donne recommended applying to the International Energy Agency (IEA) for their sponsorship of regular meetings.

The Beryllium Workshop Meeting Series

MEETING	YEAR	ATTENDEES	PAPERS	INSTITUTIONS	LOCATION
BeWS-0	1991	42	12	12	Clearwater Beach, FL
BeWS-1	1993	52	20	23	Karlsruhe, Germany
BeWS-2	1995	45	37	26	Jackson Lake Lodge, WY
BeWS-3	1997	68	38	38	Mito, Japan
BeWS-4	1999	69	40	41	Karlsruhe, Germany
BeWS-5	2001	46	27	31	Moscow, Russia
BeWS-6	2003	45	49	48	Miyazaki, Japan
BeWS-7	2005	45	27	33	Santa Barbara, CA
BeWS-8	2007	56	22	27	Lisbon, Portugal
BeWS-9	2009		17	24	Almaty, Kazakhstan
BeWS-10	2012	64	30	28	Karlsruhe, Germany
BeWS-11	2013	41	35	20	Barcelona, Spain
BeWS-12	2015	44	30	44	Jeju, South Korea
BeWS-13	2017	35	29	46	Chiba, Japan

The Workshop name has evolved

- BeWS-1 Workshop on Beryllium for Fusion Applications
- BeWS-2 2nd IEA International Workshop on Beryllium Technology for Fusion
- BeWS-7 7th IEA International Workshop on Beryllium Technology

Mario Dalle Donne Memorial Award

- Conceived at BeWS-10 to recognize excellence in beryllium research
- Awards presented
  - BeWS-11 – Dr. Masaru Nakamichi
  - BeWS-12 – Dr. Vladimir Chakin
  - BeWS-13 – Dr. Jörg Reimann

Beryllium Workshops have many benefits

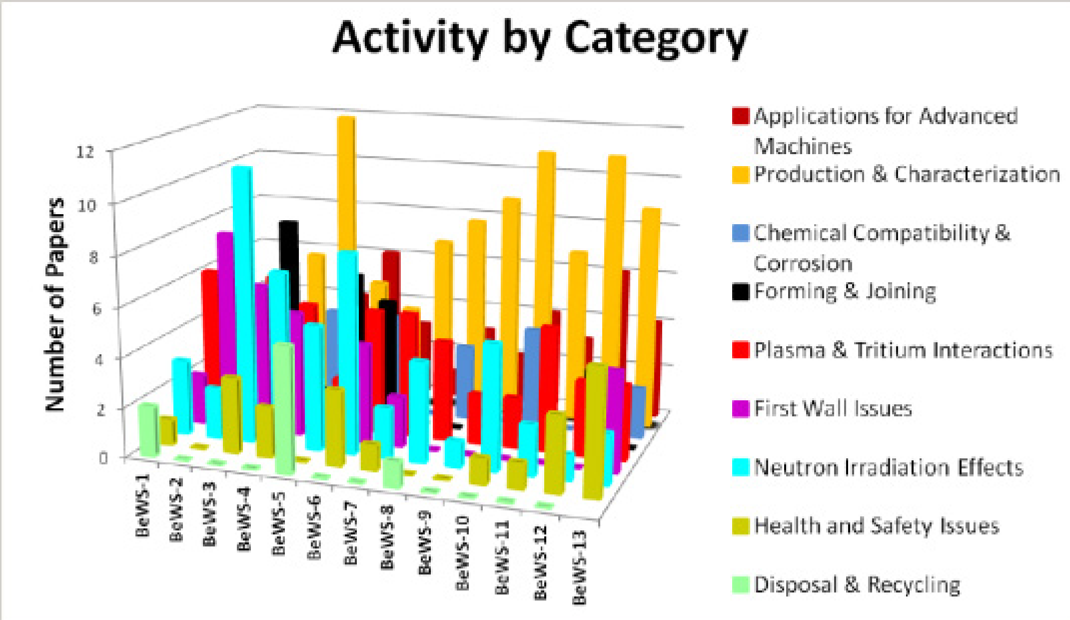
- Approved international forum for exchange of information, results, ideas and concerns
- Opportunity for informal collaboration among researchers with common interests and objectives
- Held at various venues around the world
  - Allows shared responsibility for arrangements and facilities
  - Shadowing ICFRM meetings helps minimize travel costs and time away from work for participants
  - Participants get cultural experience in many different lands

Memories of some past workshops

Video

Workshop papers have generally been grouped into the following categories

- Applications for Advance Machines
- Production and Characterization
- Chemical Compatibility and Corrosion
- Forming and Joining
- Plasma and Tritium Interactions
- First Wall Issues
- Neutron Irradiation Effects
- Health and Safety Issues
- Disposal and Recycling



General Observations

- Considerable progress has been made in the last thirty years in exploration of new concepts and ideas.
- Though BeWS participation has varied from year to year, general activity levels remain about the same.
  - Most attention has been given to manufacturing and characterization of new forms of beryllium including beryllides and intermetallics.
  - Least attention has been given to disposal and recycling
- The need for research and technology advancement continues in support of both fusion and fission.
  - Experiments and analyses have added new understanding.
  - Many of the issues raised as concerns in 1991 remain concerns.
  - There is greater emphasis lately on machines beyond ITER.

Thank you for your attention

Best of luck and success in your future work and meetings

Current Status in Development and Qualification of Beryllium Materials for the EU HCPB Test Blanket Module
M. Zmitko (F4E, Spain) et al.

Current Status in Development and Qualification of Beryllium Materials for the EU HCPB Test Blanket Module

M. Zmitko[1], P. Vladimirov[2], V. Chakin[2], H-C. Schneider[2], R. Rolli[2], A. Goraieb[3], L. Magielsen[4]

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[4]NRG Petten, Petten, The Netherlands

One of the reference tritium Breeder Blanket concepts developed in the Europe that will be tested in ITER machine under the form of Test Blanket Module (TBM) is the Helium-Cooled Pebble-Bed (HCPB) TBM concept in which lithiated ceramic pebbles are used as a tritium breeder and beryllium/beryllides pebbles as a neutron multiplier material. This concept uses the EUROFER97 reduced activation ferritic-martensitic (RAFM) steel as a structural material and pressurized helium for heat extraction (8 MPa, 300-500ºC).

The paper gives a brief general description of the European HCPB TBM design and the main design requirements including the requirements to beryllium multiplier material. The EU TBMs development and qualification plan with identification of the main milestones will be also presented.

The main part of the paper will be devoted to the presentation of beryllium materials development strategy, qualification plan and overview of the current status of research, development and characterization.

The paper will present the main results and achievements obtained in the following areas: (i) post-irradiation examination (PIE) of Be/beryllides materials irradiated in HIDOBE-01 and HIDOBE-02 irradiation campaigns aiming at determination of the materials' performance, properties and characteristics under neutron irradiation (e.g. dimensional stability/changes, porosity, morphology and microstructure by OM, SEM and TEM, tritium retention/release characteristics and mechanical properties; (ii) characterization of the as-received reference 1mm Be pebbles produced by the rotating electrode process (REP) in terms of their size distribution, chemical composition including level of impurities, characteristics of tritium release from Be pebbles, interaction of Be pebbles with air/steam at various temperatures.

A new irradiation experiment LIBERTI, foreseen for the functional materials (i.e. beryllium materials and ceramic breeder pebbles), will be introduced as well as a study performed on definition of the main objectives, test matrix, functional materials to be irradiated and irradiation parameters. A conceptual design of this new LIBERTI irradiation experiment will be briefly described.

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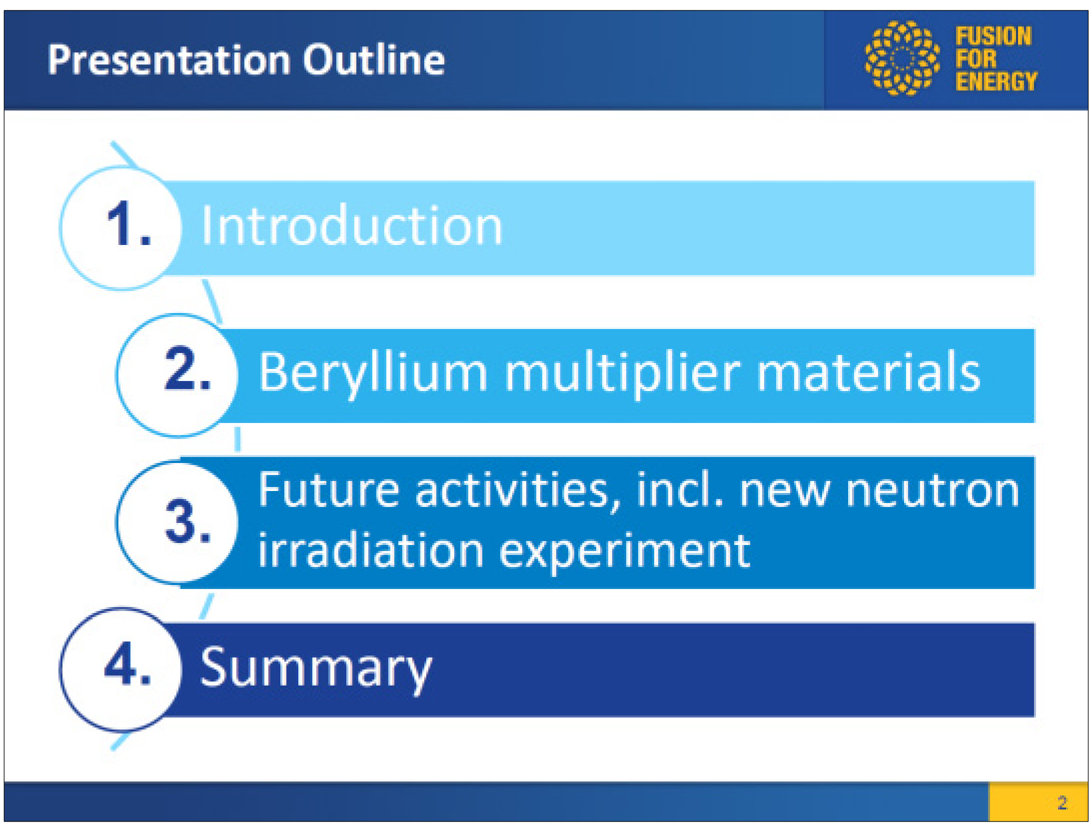
FUSION
FOR
ENERGY
BRINGING
THE POWER
OF THE SUN
TO EARTH
Current Status in Development and Qualification of Beryllium Materials for the EU HCPB Test Blanket Module
M. Zmitko – presenting author
P. Vladimirov, V. Chakin, H-C. Schneider, R. Rolli
KIT
A. Goraieb
KBHF
L. Magielsen
NRG
14th International Workshop on Beryllium Technology
Hotel Queen Mary, Long Beach, CA, USA
24-25 October, 2019

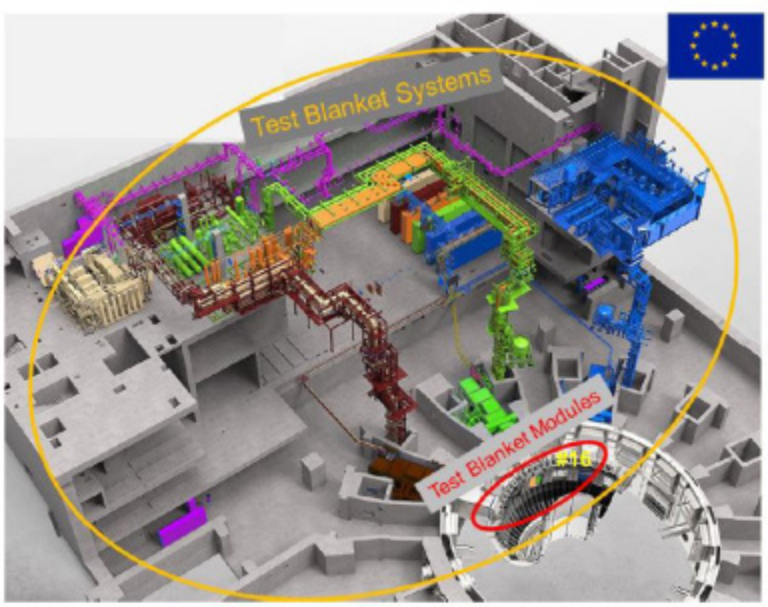

Presentation Outline
FUSION
FOR
ENERGY
1. Introduction
2. Beryllium multiplier materials
3. Future activities, incl. new neutron irradiation experiment
4. Summary
2

FUSION FOR ENERGY

1. Introduction

Test Blanket Systems testing at ITER

FUSION FOR ENERGY



"The TBM project provides test blankets to test and validate design concepts of tritium breeding blankets relevant to a power-producing reactor."
(from ITER Project Requirements)

Europe proposes **two Test Blanket Systems**, aiming to test **two types of tritium breeding materials and two coolants:**

- Liquid metal: Pb-16Li & pressurized water coolant → **WCLL TBM**
- Pebbles bed: Li ceramic & high-pressure helium coolant → **HCPB TBM**
- **EUROFER97** structural material (RAFM steel)

EU HCPB: Helium Cooled Pebble Bed TBM

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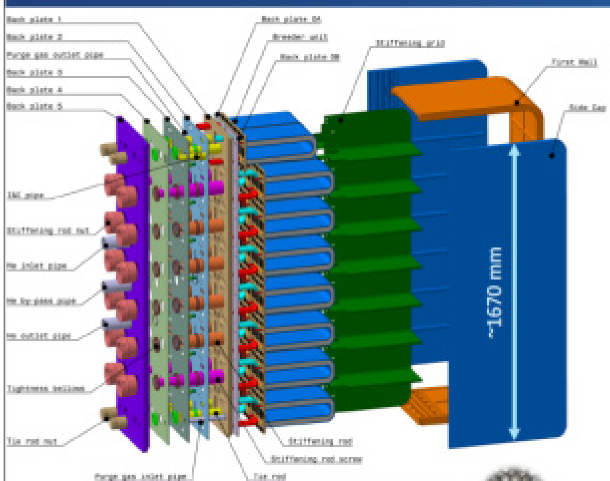
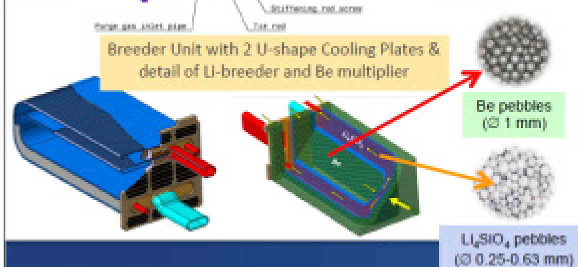


Breeder Unit with 2 U-shape Cooling Plates & detail of Li-breeder and Be multiplier



EUROFER97 structural material (1444 kg)

Li-ceramic breeder in the form of **pebble beds** (Li enriched at 90% in ^{6}Li for the optimization of the tritium breeding ratio)

Reference CB material: **Advanced Li_4SiO_4 (LOS)** with Li_2TiO_3 (LMT) as a secondary phase (improved mechanical properties) produced by melt-spraying process; total weight **~75-80 kg**

Beryllium neutron multiplier in the form of **pebble beds**

Reference material for 1st TBM: **1mm Be pebbles** produced by REP; total weight **~180-200 kg**

Subsequent TBMs: Advanced Be material - **beryllium alloys/beryllides** (e.g. $Be_{12}Ti$) → lower swelling, better T release, better oxidation resistance, smaller reactivity with structural material (production maturity & characterization)

Pressurized Helium coolant (8 MPa) for heat extraction (300-500°C)

Low-pressure (0.4 MPa) **Helium purge gas** He+0.1vol%H_2 for tritium extraction

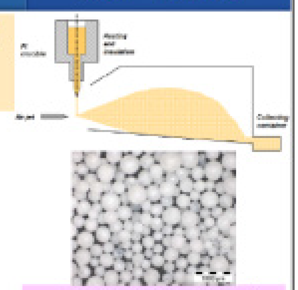
5

Functional Requirements for Functional Materials

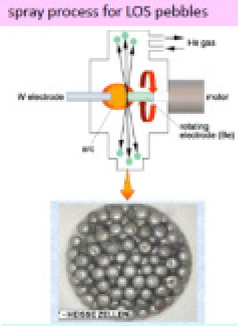
FUSION FOR ENERGY

General requirements come from DEMO objectives with a short-term objective to characterize & qualify Functional Materials (Li-ceramics breeder and Be materials) for a use in the ITER TBM

- Neutronic performances for T self-sufficiency (TBR ≥ 1.1)
- Temperature control of the pebble beds during operation in the temperature window of ~400-920ºC for the CB and ~300-650ºC for Be (PBTM issues/aspects)
- Sufficient long lifetime → (i) neutron irradiation resistance without significant changes of thermo-physical and mechanical properties; (ii) withstanding stresses induced under DEMO-relevant operating conditions without excessive fragmentation and flow resistance of the purge gas (CB and Be pebble beds do not have a structural function)
- Material compatibility between the FMs and EUROFER (up to max. T~ 550ºC), in the reference purge gas He+0.1vol%H_2)
- Low tritium residence time/tritium retention in the Li-ceramics pebbles; efficient tritium extraction → purge gas chemistry optimization
- Low tritium retention in the Be pebbles to minimize tritium inventory → safety aspect
- As low as possible activation under neutron irradiation (impurities level control; e.g. U, Co, Ni, Al, Fe,...) → for recycling / reprocessing (in the view of DEMO/future FPR) and waste management
- Limit/predict hydrogen and passive heat release during accidental conditions below safety limits (Be/steam/water interaction)

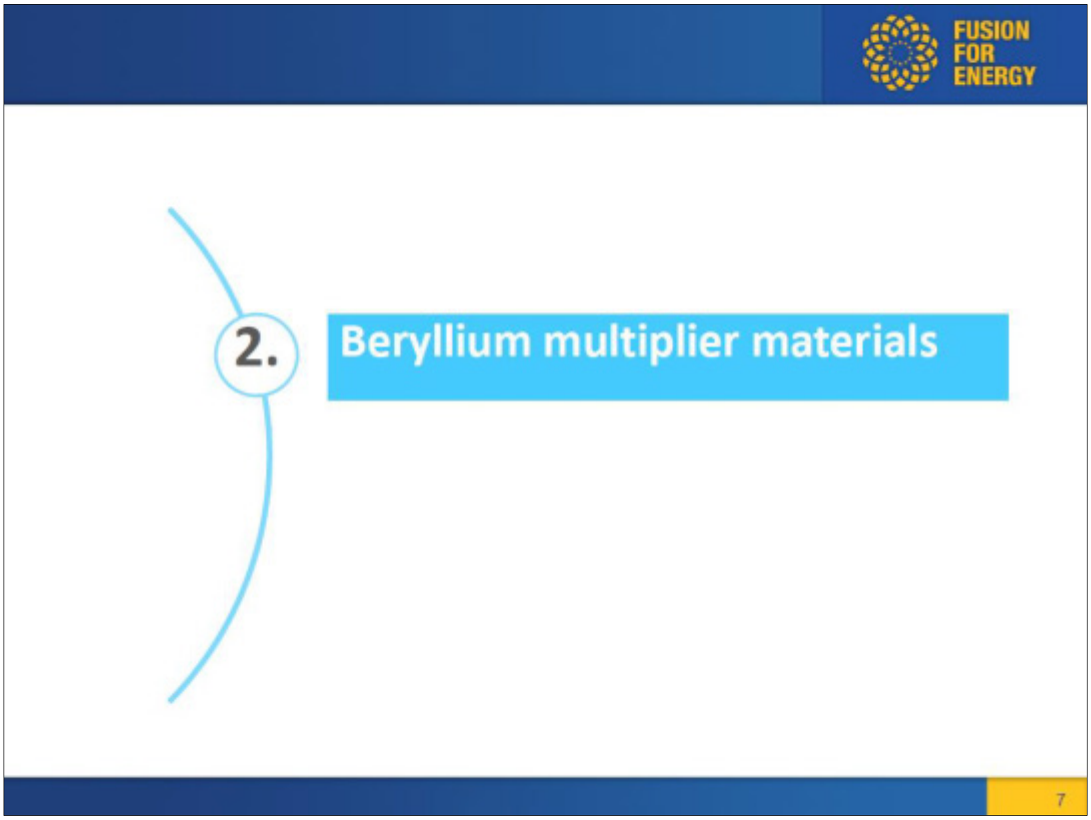


Schematic representation of melt-spray process for LOS pebbles



1 mm Be pebbles NGK (JA) Rotation Electrode Process (REP)

6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Beryllium multiplier materials
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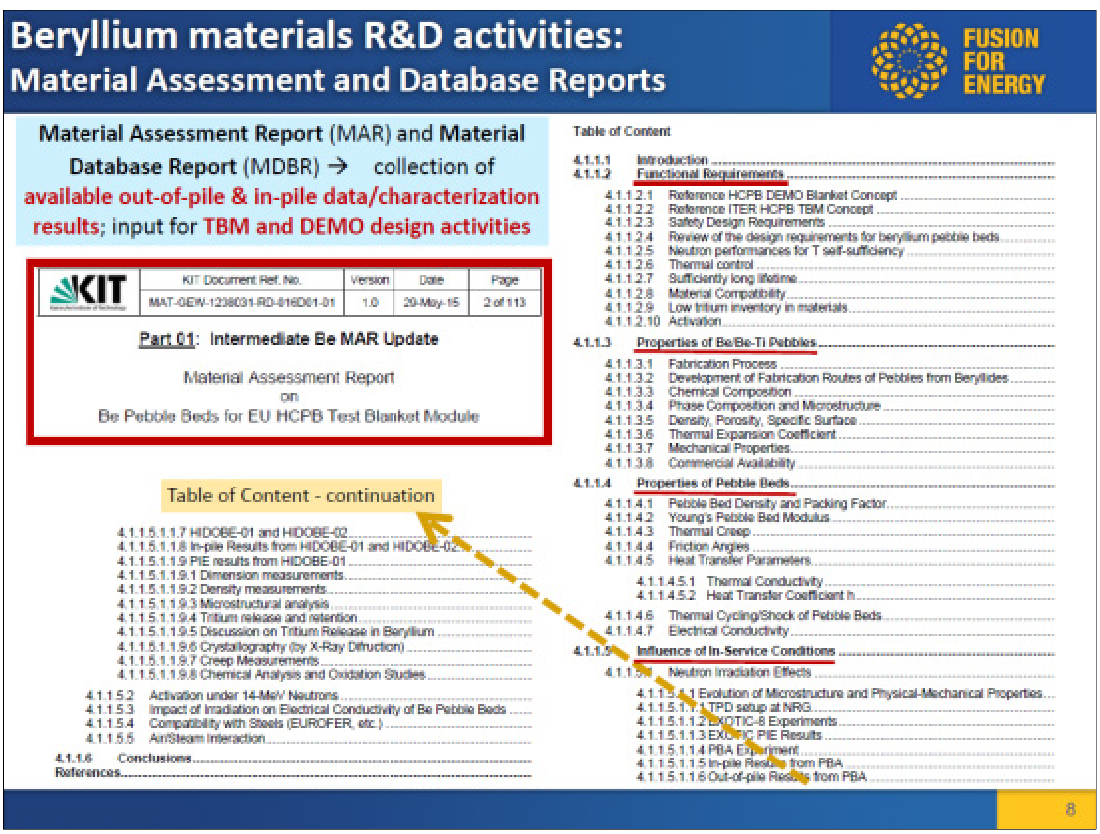
Beryllium materials R&D activities:
Material Assessment and Database Reports
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Material Assessment Report (MAR) and Material Database Report (MDBR) → collection of available out-of-pile & in-pile data/characterization results; input for TBM and DEMO design activities
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Date
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Material Assessment Report
on
Be Pebble Beds for EU HCPB Test Blanket Module
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4.1.1.5.1.1.9 PIE results from HIDOBE-01
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from PBA
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HIDOBE irradiation campaign & PIE

FUSION FOR ENERGY

High Dose Beryllium fission reactor irradiation program (**HIDOBE**)

- blanket relevant temperatures (425-750°C)
- pebbles of 0.5, 1 and 2 mm produced by REP (NGK, Japan); Be and $Be_{12}Ti$ (Be-5at%Ti and Be-7at%Ti) pellets
- Constrained & unconstrained Be pebbles

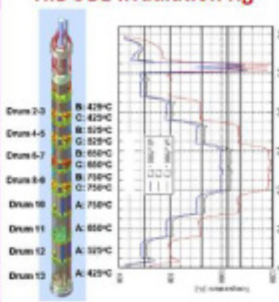
Objectives :

(i) Beryllium behaviour under DEMO relevant He/dpa ratios and temperatures
(ii) Tritium inventory as affected by microstructure, swelling, creep
(iii) Pebble beds thermo-mechanical behaviour under neutron irradiation

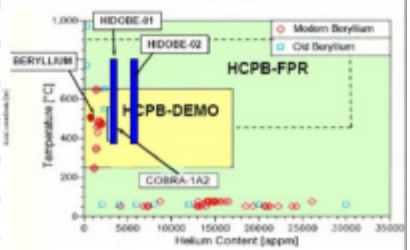
HIDOBE-01 irradiation rig:
3000 appm He (DEMO: ~18,000 appm); 18 dpa in Be
Irradiation completed - 665 FPD
PIE completed in 2012

HIDOBE-02 irradiation rig:
6000 appm He; 36 dpa in Be
Irradiation completed - 1274 FPD
PIE completed in 2017

HIDOBE Irradiation rig



HIDOBE Irradiation program vs. DEMO



NRG

Post Irradiation Examination (2012-2017):

- Dimensional stability, size distribution, porosity [**performed by NRG**]
- Microstructure and morphology (OM, SEM, TEM, XRD) [**KIT & NRG**]
- Tritium and He release (TPD) [**NRG & KIT**]
- Mechanical properties – Vickers hardness, creep [**KIT**]
- Thermal properties (LFA) [**NRG**]

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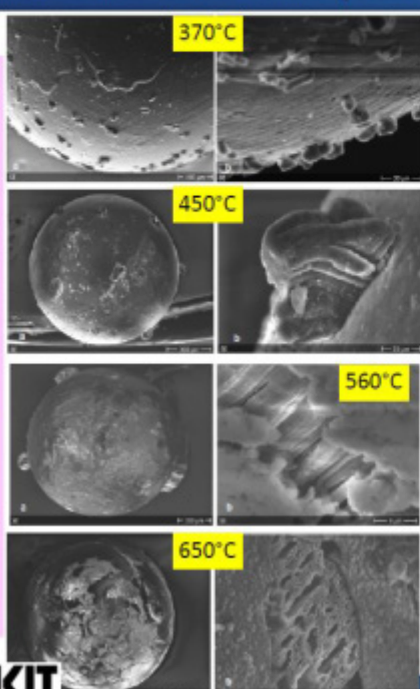
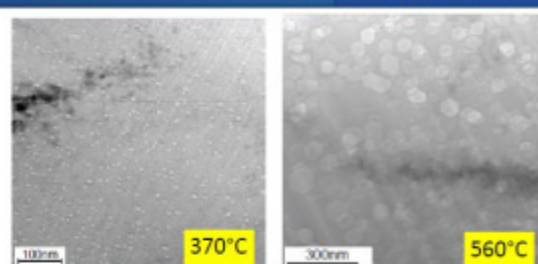
HIDOBE Post-Irradiation Examination Microstructure of Be pebbles

FUSION FOR ENERGY

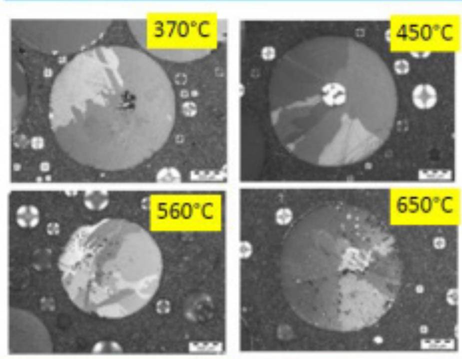
Effect of irradiation temperature on surface oxidation layer



KIT



TEM images of He bubbles in 1 mm Be pebbles (hexagonal shape)



Cross sections (OM) of irradiated unconstrained Be pebbles of 1 mm
→ formation of bubbles and pores

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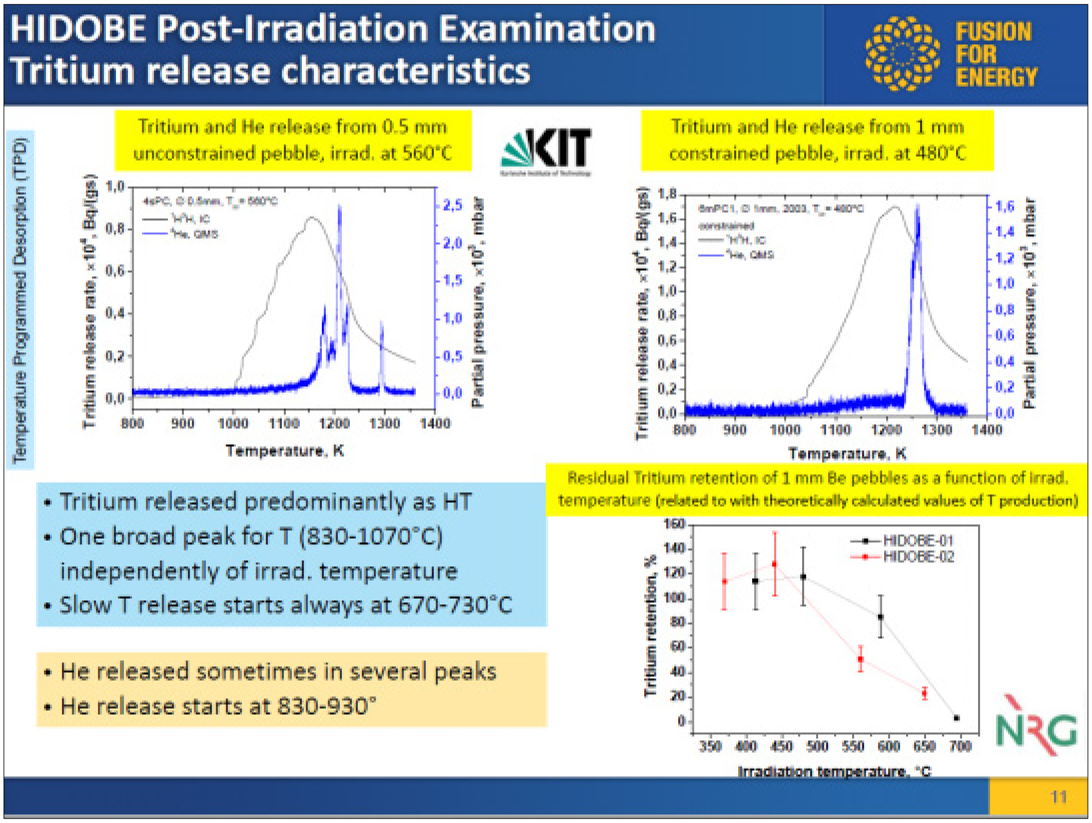
HIDOBE Post-Irradiation Examination
Tritium release characteristics
FUSION FOR ENERGY
Temperature Programmed Desorption (TPD)
Tritium and He release from 0.5 mm unconstrained pebble, irrad. at 560°C
KIT
Tritium and He release from 1 mm constrained pebble, irrad. at 480°C
Tritium release rate, ×10^4, Bq/(gs)
Partial pressure, ×10^3, mbar
Temperature, K
Residual Tritium retention of 1 mm Be pebbles as a function of irrad. temperature (related to with theoretically calculated values of T production)
• Tritium released predominantly as HT
• One broad peak for T (830-1070°C) independently of irrad. temperature
• Slow T release starts always at 670-730°C
• He released sometimes in several peaks
• He release starts at 830-930°
HIDOBE-01
HIDOBE-02
Tritium retention, %
Irradiation temperature, °C
NRG
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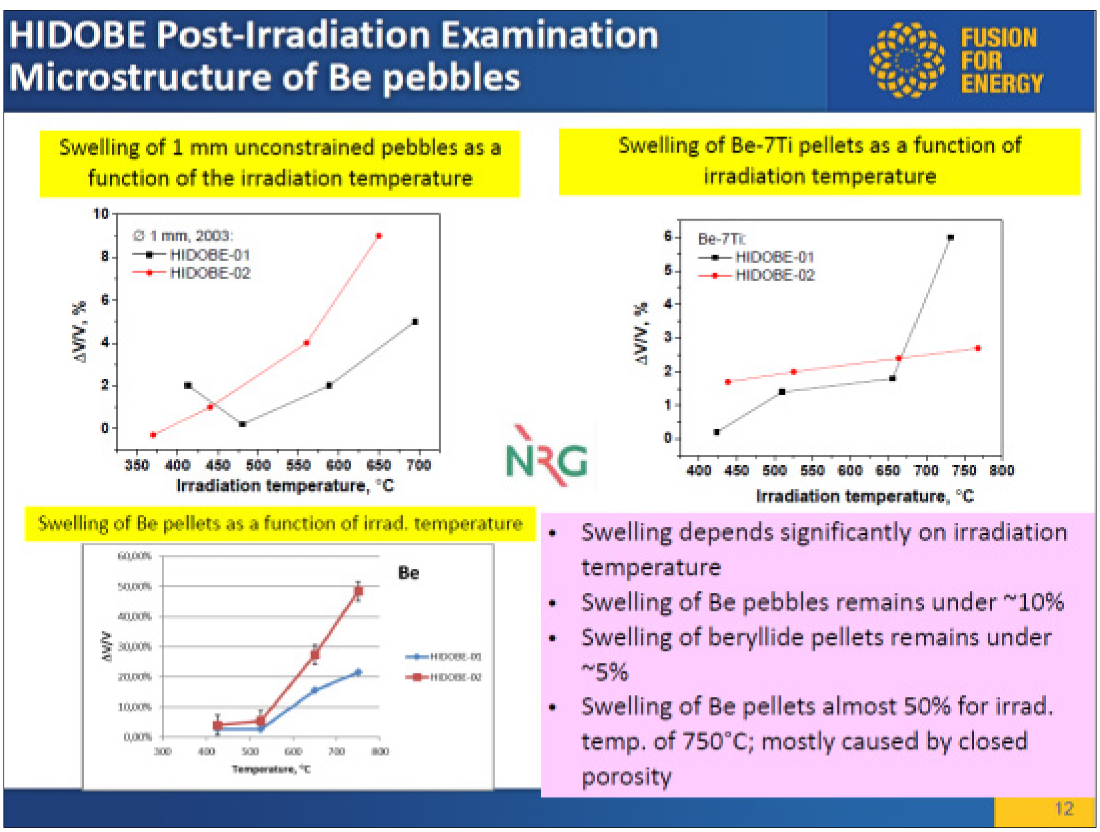
HIDOBE Post-Irradiation Examination
Microstructure of Be pebbles
FUSION FOR ENERGY
Swelling of 1 mm unconstrained pebbles as a function of the irradiation temperature
Swelling of Be-7Ti pellets as a function of irradiation temperature
∅ 1 mm, 2003:
HIDOBE-01
HIDOBE-02
ΔV/V, %
Irradiation temperature, °C
NRG
Be-7Ti:
HIDOBE-01
HIDOBE-02
ΔV/V, %
Irradiation temperature, °C
Swelling of Be pellets as a function of irrad. temperature
Be
• Swelling depends significantly on irradiation temperature
• Swelling of Be pebbles remains under ~10%
• Swelling of beryllide pellets remains under ~5%
• Swelling of Be pellets almost 50% for irrad. temp. of 750°C; mostly caused by closed porosity
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Beryllium materials R&D activities: HIDOBE-01/02 PIE results

FUSION FOR ENERGY

Summary of the main PIE results:

Microstructure:

- Strongly depends on irradiation temperatures independently for irradiated constrained/ unconstrained Be pebbles. No significant pore or bubble formations occur at 425 and 525ºC. Irradiations at 650 and 750ºC lead to intensive pore formation resulting swelling up to the highest value of 7 %.
- No compact oxidation layer is observed on the surface regions at 425 and 525 ºC. However, strong oxidation occurs after irradiation at 650 and 750ºC where the thickness of BeO layers on the pebble surfaces can reach 10-25 µm.

Tritium release:

- One peak is observed (at TPD) in the temperature range ~900-1100ºC independently to the irradiation temperature or the pebbles bed state (constrained/unconstrained).
- Tritium is released mainly in the form of HT.
- Irradiation temperature influences the amount of Tritium released/retained throughout irradiation: it increases significantly above 650ºC; at 750ºC, the residual Tritium measured in irradiated samples is 6 to 8 times lower than in samples irradiated at lower temperature.

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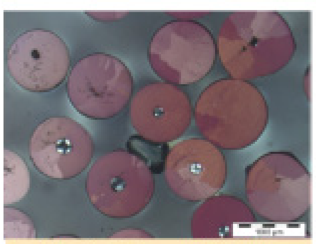
Characterization of reference Be pebbles

FUSION FOR ENERGY

Characterization of as-received Be pebbles supplied by NGK Insulators Ltd., Japan

KIT

- 1 mm Be pebbles produced by Rotation Electrode Process (REP)
- Over 90% of pebbles in the size range of 890-1220 µm



Microstructure of Be pebbles

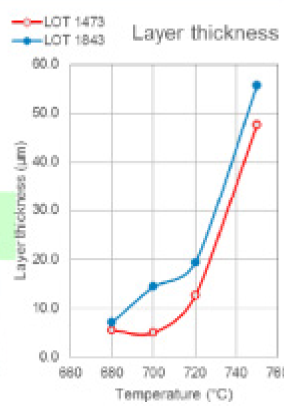
Chemical analyses obtained by Ion Beam (in ppm)

Element	Content	Element	Content
Mg	56±12	Cr	123±35
Al	780±87	Mn	81±28
Si	529±95	Fe	1082±242
P	31±24	Co	<4
S	13±4	Ni	188±22
Cl	7±1	Cu	75±23
Ca	67±63	W	27
Sc	18±9	U	<7
Ti	204±39		

Interaction of Be pebbles with air and steam → reaction kinetic

Be specimens after reaction with steam at different temperatures

Be oxidation in steam



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FUSION FOR ENERGY

3. Future activities, incl. new neutron irradiation experiment

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FUSION FOR ENERGY

Note: Following a new organization of the **European TBM Project** → R&D support activities on Functional Materials (incl. Be materials for TBM) now under **EURO*fusion* responsibility;**
Role of F4E/TBM – scope & objectives specification for ITER TBM activities

Activities planned within the Preliminary Design (PD) phase (till 2022):

- **Development & Qualification**
  - Complementary **oxidation studies** of reference Be pebbles in air and water vapor (experiments at temperatures not covered by the previous activity)
  - **Update** of the existing Functional Materials properties database (Version for HCPB PDR in 2022)
- **Supply Capacity Development**
  - Determination of **supply capacities** for a (semi-industrial) production of Be pebbles (for HCPB TBM) - reference & alternative production routes; elaboration of a **roadmap for delivery** of Be materials for HCPB TBMs
  - Characterization of Be pebbles produced with **an alternative production route** based on fluoride reduction method (FRM) (e.g. chemical composition, microstructure, porosity, thermo-mechanical properties, oxidation characteristics, tritium release characteristics,...)
- **Functional Materials Neutron Irradiation**
  - Design of irradiation rig for Functional Materials new irradiation test (conceptual & detailed design) and the rig manufacturing
  - Characterization of Be pebbles for new irradiation test (Be pebbles produced by both reference REM and alternative FRM)

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FUSION FOR ENERGY

Activities planned after the PD phase:

- **Development & Qualification**
  - Experimental study on development of a **filling procedure for CB and Be pebbles** into the HCPB Breeder Units
  - **Update** of the existing Functional Materials properties database (Version for HCPB FDR in 2025)
- **Supply Capacity Development**
  - Characterization of the reference Be pebbles produced with an **upgraded rotation electrode method**
  - Characterization of Be pebbles supplied for 1st HCPB TBM
- **Functional Materials Neutron Irradiation**
  - New irradiation experiment realization (irradiation for ~1.5 years; equivalent ~2-3 dpa in steel - still TBD)
  - Post-irradiation activities (dismantling, retrieval of irradiation drums, transport, waste management and permanent storage)
  - Post-irradiation examination of Be *(& CB)* materials
- **Reviews and Gates**
  - Functional Materials Development, Qualification, Supply - RoX for DEMO - Be *(& CB)*
  - Technical Specifications for Be *(& CB)* pebbles procurement

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A new irradiation experiment LIBERTI *(preliminary considerations)*

FUSION FOR ENERGY

Motivation for a new irradiation experiment:

- Characterization of advanced CB material
- Characterization of Be pebbles fabricated by an alternative process (e.g. FRM)
- Characterization of developed beryllides
- **International collaboration envisaged**

Li Ceramic Breeder irradiation

Experimental Objective
In pile T-release/ purge gas experiments
Out of pile tritium release
Compatibility studies
Compressed pebble beds

Beryllium/beryllides irradiation

Experimental Objective
Compressed pebble beds
Out of pile tritium release
Compatibility studies

MATERIALS TEST MATRIX AND IRRADIATION CONDITIONS

Lithium ceramics:

Pebble a	65 mol% Li_4SiO_4 + 35 mol% Li_2TiO_3
Pebble b	70 mol% Li_4SiO_4 + 30 mol% Li_2TiO_3
Pebble c	80 mol% Li_4SiO_4 + 20 mol% Li_2TiO_3

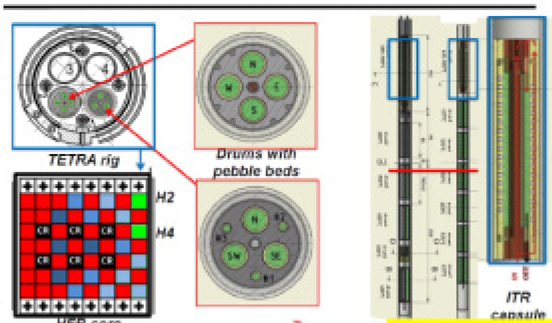
Pebble b and c→ in pile tritium release 400- 900 C
Pebble a,b,c → Target temperatures 400, 600 and 850 C

Beryllium :
REM production route Ø 1mm
Fluoride reduction method Ø 1 mm
Fluoride reduction method Ø 0.5-2 mm

Beryllide:
BeTi (KIT)

All pebble beds → Target temperatures 425, 525 and 600 C
three different thermomechanical loads (to be determined)

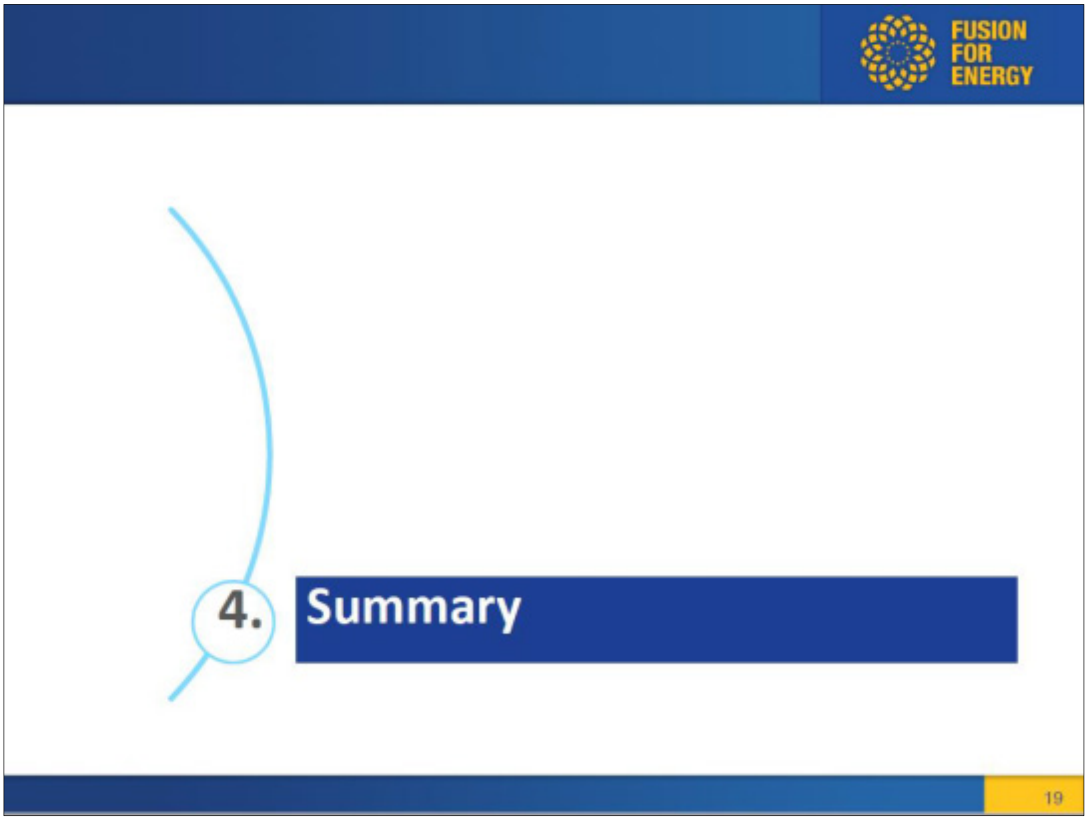
DESIGN OF IN PILE TEST FACILITY

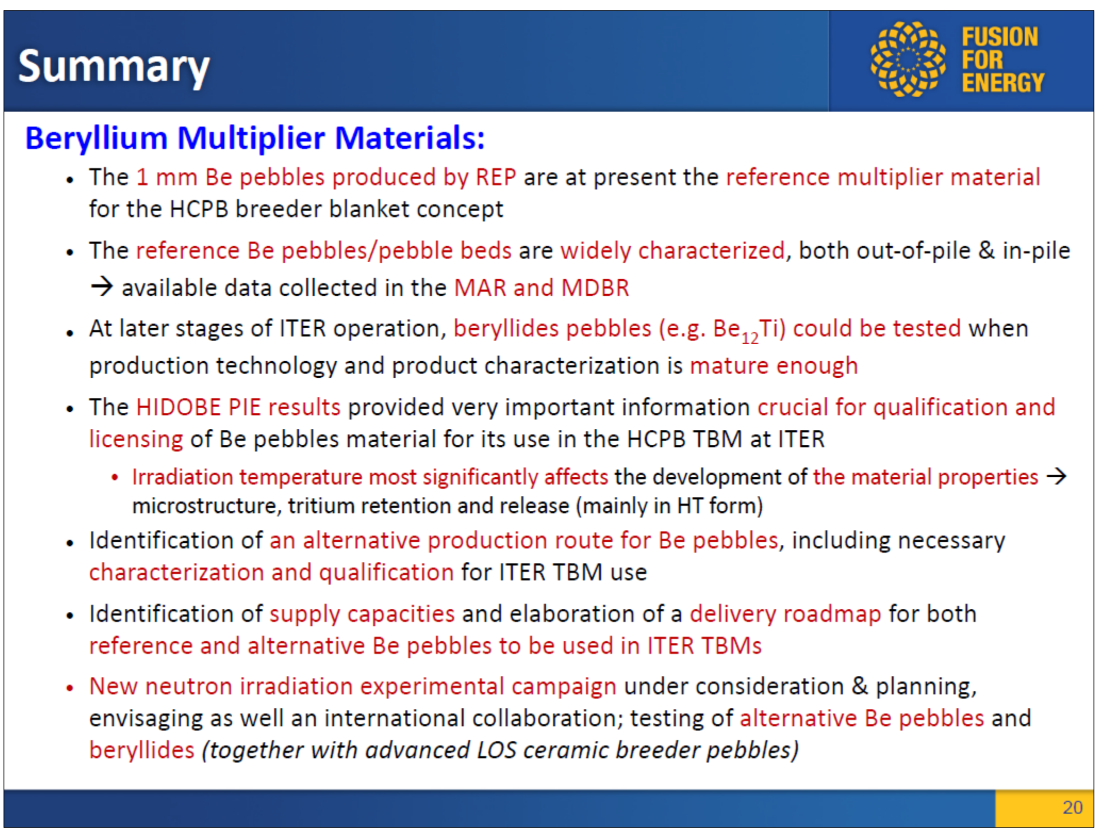


ITR = In-pile T release

NRG

18


FUSION FOR ENERGY
4.
Summary
19


Summary
FUSION FOR ENERGY
Beryllium Multiplier Materials:
• The 1 mm Be pebbles produced by REP are at present the reference multiplier material for the HCPB breeder blanket concept
• The reference Be pebbles/pebble beds are widely characterized, both out-of-pile & in-pile → available data collected in the MAR and MDBR
• At later stages of ITER operation, beryllides pebbles (e.g. $Be_{12}Ti$) could be tested when production technology and product characterization is mature enough
• The HIDOBE PIE results provided very important information crucial for qualification and licensing of Be pebbles material for its use in the HCPB TBM at ITER
• Irradiation temperature most significantly affects the development of the material properties → microstructure, tritium retention and release (mainly in HT form)
• Identification of an alternative production route for Be pebbles, including necessary characterization and qualification for ITER TBM use
• Identification of supply capacities and elaboration of a delivery roadmap for both reference and alternative Be pebbles to be used in ITER TBMs
• New neutron irradiation experimental campaign under consideration & planning, envisaging as well an international collaboration; testing of alternative Be pebbles and beryllides *(together with advanced LOS ceramic breeder pebbles)*
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Recycle Business Challenge of Activated Beryllium Neutron Reflectors used in Research Reactors

H. Kawamura[1], S. Daulbayev[2], Y.A. Kenzhin[3], I.L. Tazhibaeva[4], V. Shestakov[5], K. Nojiri[6], H. Kanazawa[1], H. Hokari[7], K. Tsuchiya[8], and T. Hosoda[1]

[1]Chiyoda Technol Co., [2]Ulba Metallurgical Plant JSC, [3]Institute of Nuclear Physics, [4]National Technology and Safety Center, [5]Al-Farabi National Kazakhstan University, [6]NGK Insulators Ltd., [7]Sumitomo Corp., [8]Japan Atomic Energy Agency

Chiyoda Technol Co. (CTC) was starting the feasibility study concerning beryllium recycle with Kazakhstan team (Ulba, INP, NTSC and so on) from last January. Japanese team consists of CTC, NGK, Sumitomo Corp. and JAEA.

Concerning the decommissioning of the research reactors all over the world, only problem is "treatment of activated beryllium neutron reflector (ANBR)" and a common issue facing the world's research reactors. In addition, by recycling the beryllium recovered from "activated beryllium neutron reflector" which is like urban mineral resource, it can also contribute to the production and supply of beryllium neutron multipliers of International Thermonuclear Engineering Reactor (ITER), which is jointly developed worldwide with Kazakhstan, and beryllium material for advanced neutron reflectors of research reactors. Finally, we will start our business for beryllium pebble supply from 2025 year.

Our concept is shown as follows. First of all, research reactor owners cut beryllium parts (valuables, not waste) beryllium neutron reflector, remove tritium from beryllium parts and export to Kazakhstan. Because Kazakhstan cannot receive waste from foreign country by Kazakhstan's law.

The recycle process consist of two, i.e. treatment process and Be pebble fabrication process. The treatment technology (Patent) of activated beryllium neutron reflector designed before 15 years is shown as follows. First, ANBR of research reactors is reacted at about 500°C with halogen gas flowing, and to form Beryllium halide. As the beryllium is changed from solid to gas by sublimation reaction, beryllium is flying out. However, the impurities of activated beryllium neutron reflector such as activated ^{60}Co remain in place and can be separated from beryllium. Then, lower activated beryllium is obtained. The problem is solved by this treatment.

We will present the outline of beryllium recycle, business model and so on.

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14th IEA International Workshop on Beryllium Technology, October 24-25, 2019 (Long Beach, California, USA)

Recycle Business Challenge of activated Beryllium Neutron Reflectors used at the Research Reactors

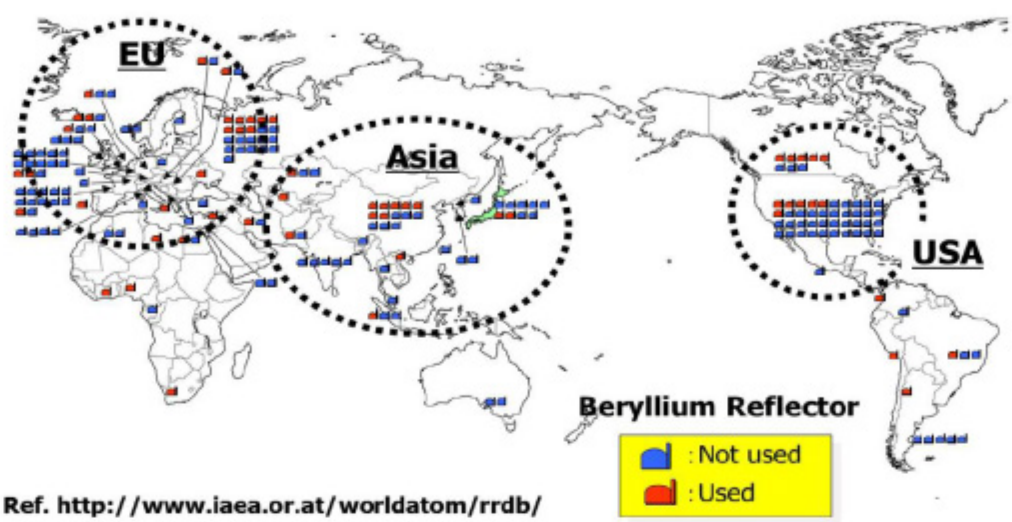
24th Oct., 2019

H.Kawamura[*1], S.Daulbayev[*2], Y.A.Kenzhin[*3], I.L.Tazhibaeva[*4], V.Shestakov[*5], K.Nojiri[*6], H.Kanazawa[*1], H.Hokari[*7], K.Tsuchiya[*8] M.Hiyoshi[*9] and T.Hosoda[*1]

*1;Chiyoda Technol Co., *2;JSC ULBA Metallurgical Plant, *3; Institute of Nuclear Physics, *4;National Technology and Safety Center, *5; National Kazakhstan University, *6;NGK Insulators LTD., *7;Sumitomo Co., *8;JAEA , *9:Nippon Express

Background

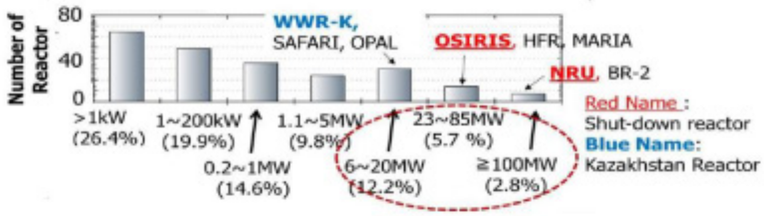
Reactors for Research in the World



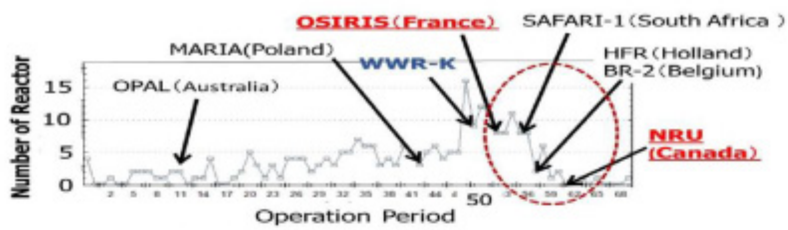
Ref. http://www.iaea.or.at/worldatom/rrdb/

1

Status of Research Reactor



(a) Classification by Thermal Power



(b) Classification by operation period

- ✓ It is indispensable to establish the treatment technology of Beryllium Neutron Reflector.
- ✓ This subject is universally common all over the world.
- ✓ Therefore, the ripple effect is very large.

2

Handling of Beryllium used at the Research Reactor

Delicate Characteristics

- ✓ International Regulated Material
- ✓ Specific Chemical Material (BeO)
- ✓ Tritium included Material
- ✓ Activated Material

➡ **The handling of used beryllium is difficult and "used beryllium" is produced on and on.**

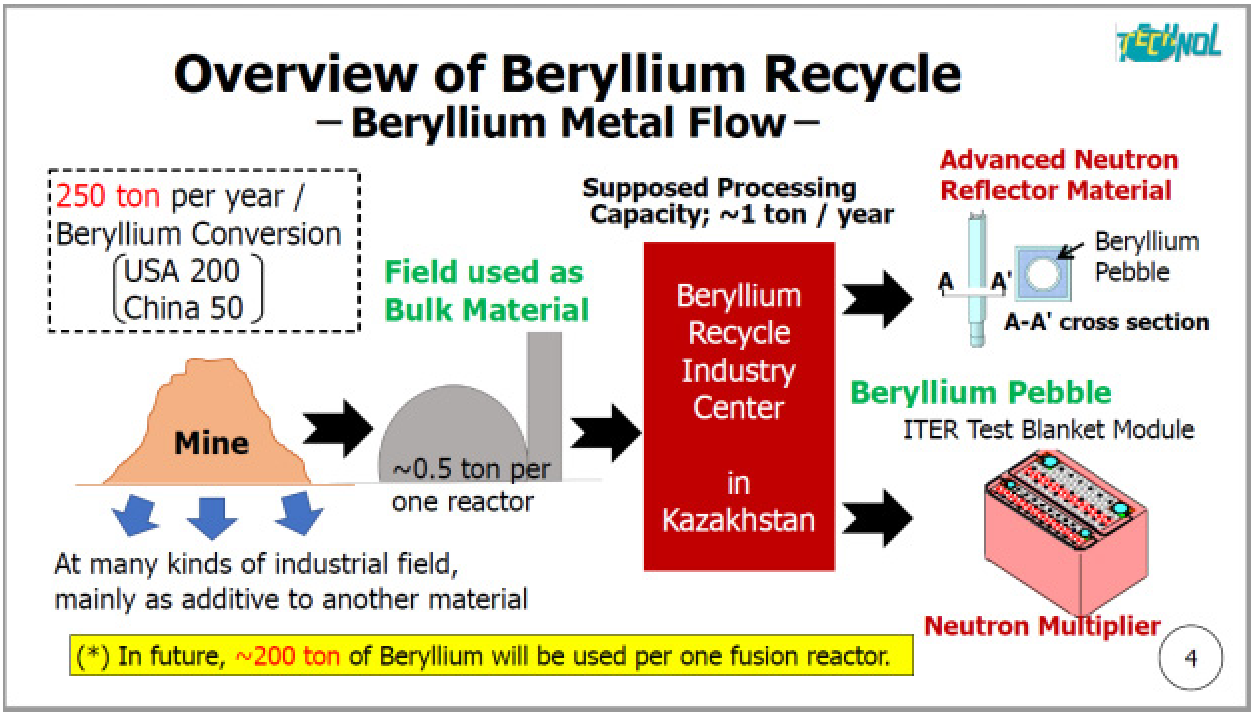
Expected Amount of used Beryllium Storage

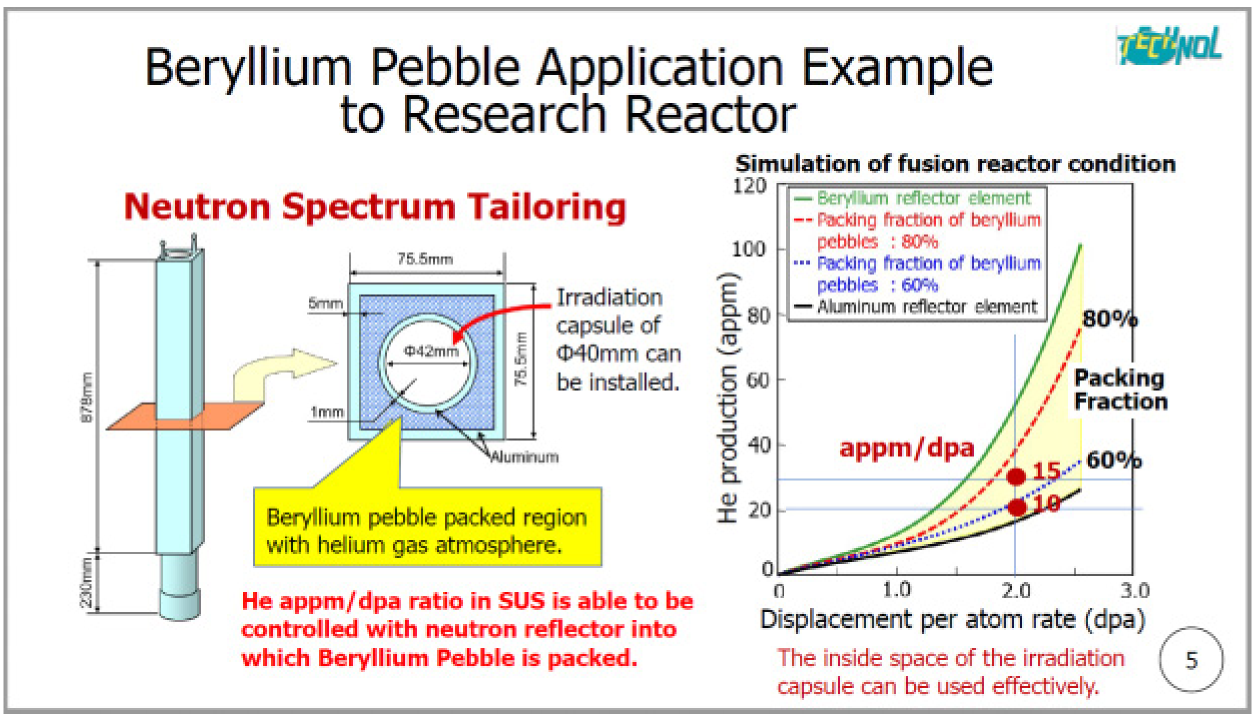
Japan : ~ 5 ton
World : 30~40 ton

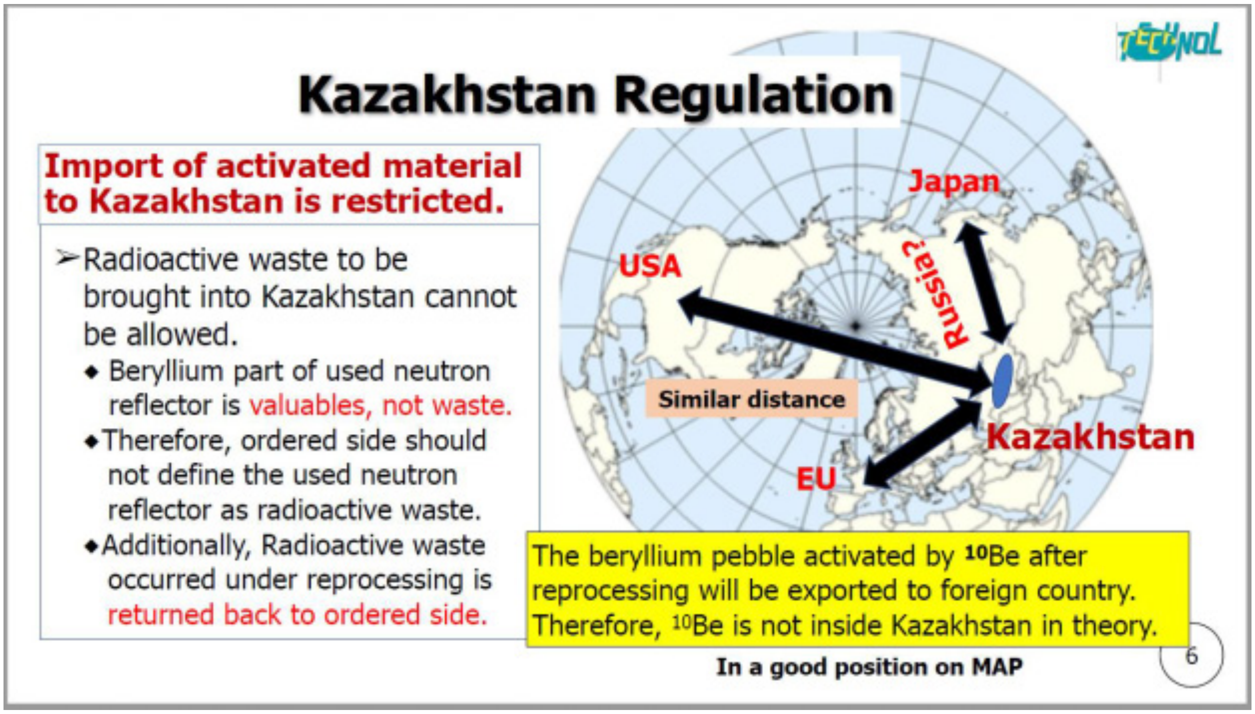
Area	Deal of used Beryllium
USA	The beryllium used at ATR was buried in the desert before about 15 years. However, as the water pollution occurred by ^{14}C, buried beryllium was dug up and has been kept at surface place briefly.
EU	Used beryllium has been kept in the pool generally.
Russia	There is the situation similar to EU.

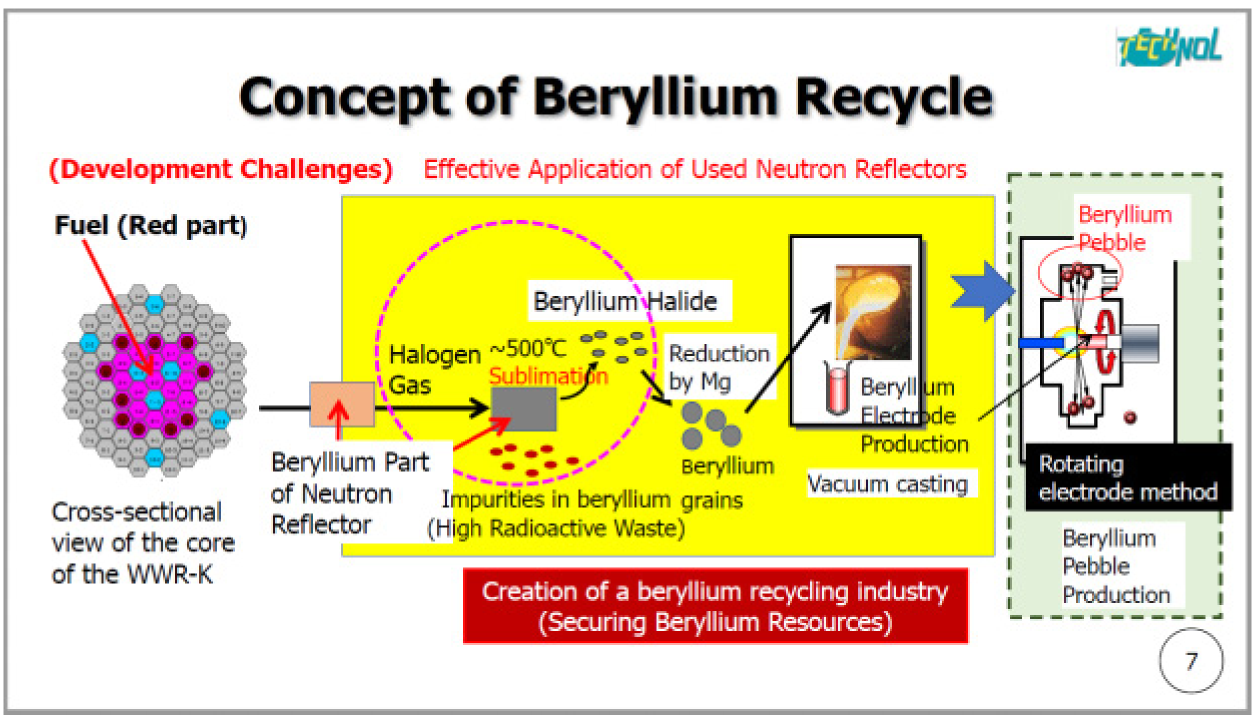
How should we deal with common issue together ?

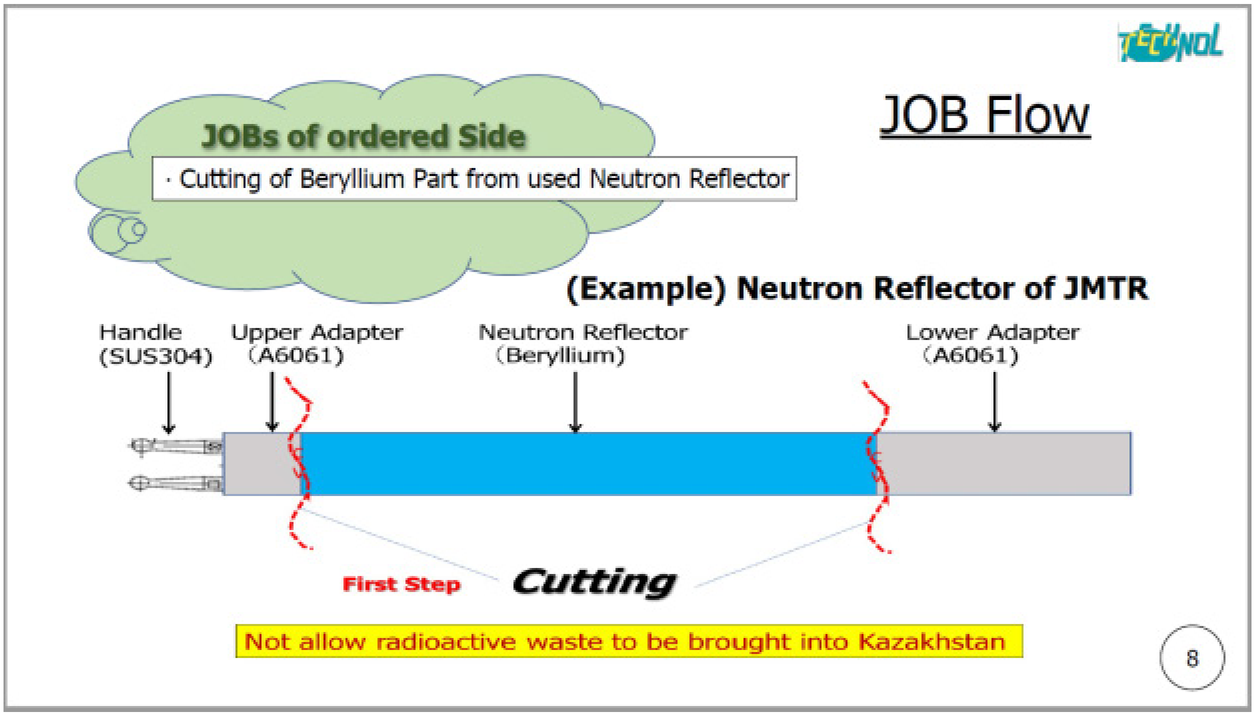
3

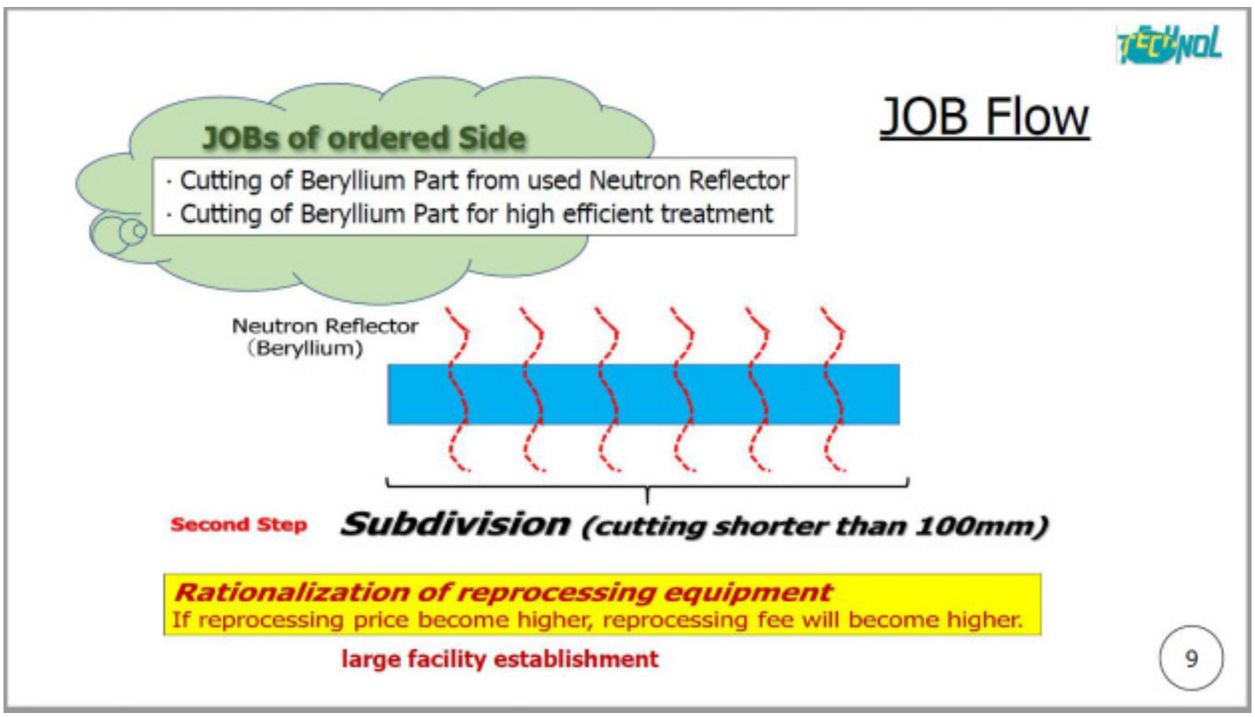

Overview of Beryllium Recycle
– Beryllium Metal Flow –
250 ton per year / Beryllium Conversion
USA 200
China 50
Field used as Bulk Material
Supposed Processing Capacity; ~1 ton / year
Advanced Neutron Reflector Material
Beryllium Pebble
A
A'
A-A' cross section
Mine
~0.5 ton per one reactor
Beryllium Recycle Industry Center in Kazakhstan
Beryllium Pebble
ITER Test Blanket Module
At many kinds of industrial field, mainly as additive to another material
Neutron Multiplier
(*) In future, ~200 ton of Beryllium will be used per one fusion reactor.
4

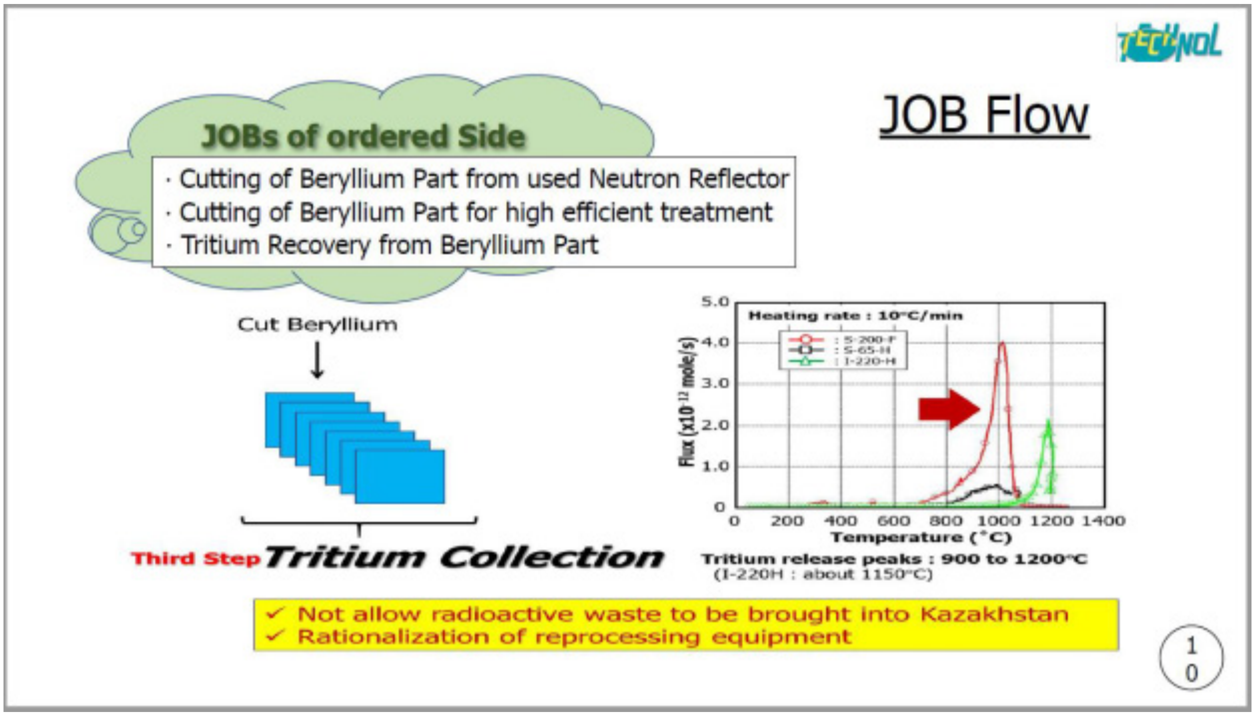

Beryllium Pebble Application Example to Research Reactor
Neutron Spectrum Tailoring
75.5mm
5mm
Φ42mm
75.5mm
1mm
878mm
230mm
Aluminum
Irradiation capsule of Φ40mm can be installed.
Beryllium pebble packed region with helium gas atmosphere.
He appm/dpa ratio in SUS is able to be controlled with neutron reflector into which Beryllium Pebble is packed.
Simulation of fusion reactor condition
Beryllium reflector element
Packing fraction of beryllium pebbles : 80%
Packing fraction of beryllium pebbles : 60%
Aluminum reflector element
He production (appm)
120
100
80
60
40
20
0
80%
Packing Fraction
60%
appm/dpa
15
10
0
1.0
2.0
3.0
Displacement per atom rate (dpa)
The inside space of the irradiation capsule can be used effectively.
5

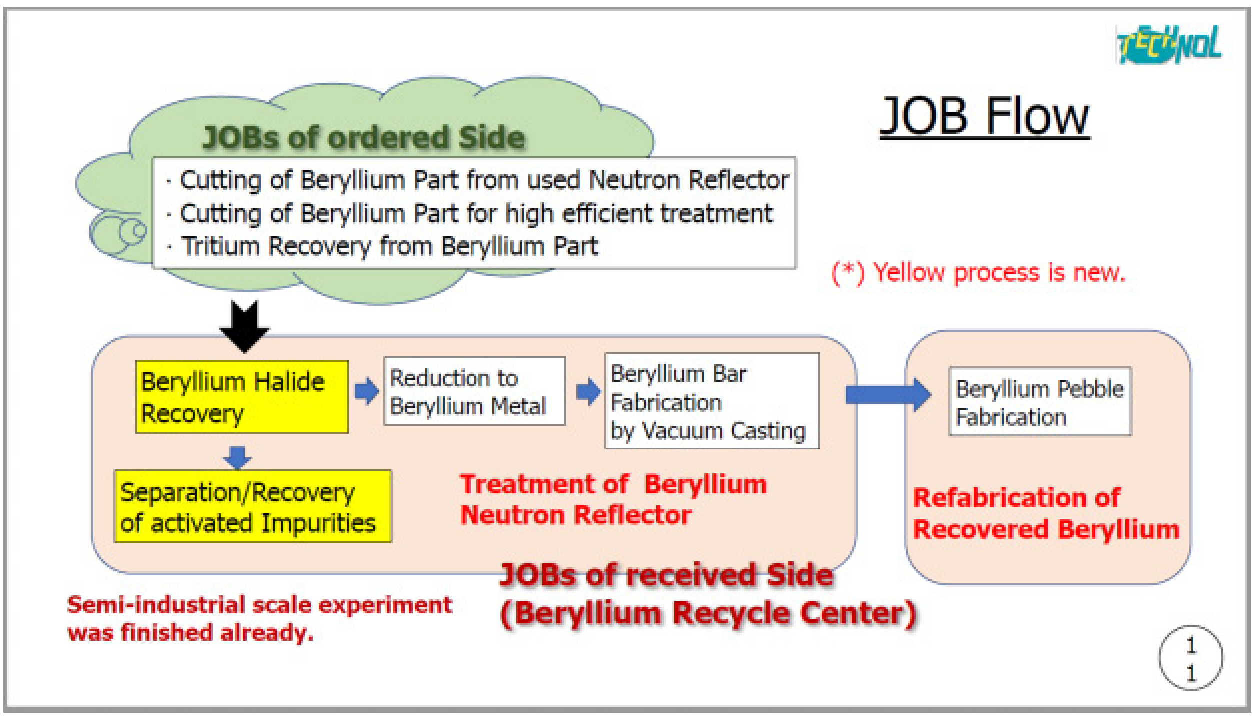

Kazakhstan Regulation
Import of activated material to Kazakhstan is restricted.
➢Radioactive waste to be brought into Kazakhstan cannot be allowed.
◆ Beryllium part of used neutron reflector is valuables, not waste.
◆Therefore, ordered side should not define the used neutron reflector as radioactive waste.
◆Additionally, Radioactive waste occurred under reprocessing is returned back to ordered side.
Japan
USA
Russia?
Similar distance
Kazakhstan
EU
The beryllium pebble activated by 10Be after reprocessing will be exported to foreign country. Therefore, 10Be is not inside Kazakhstan in theory.
In a good position on MAP
6

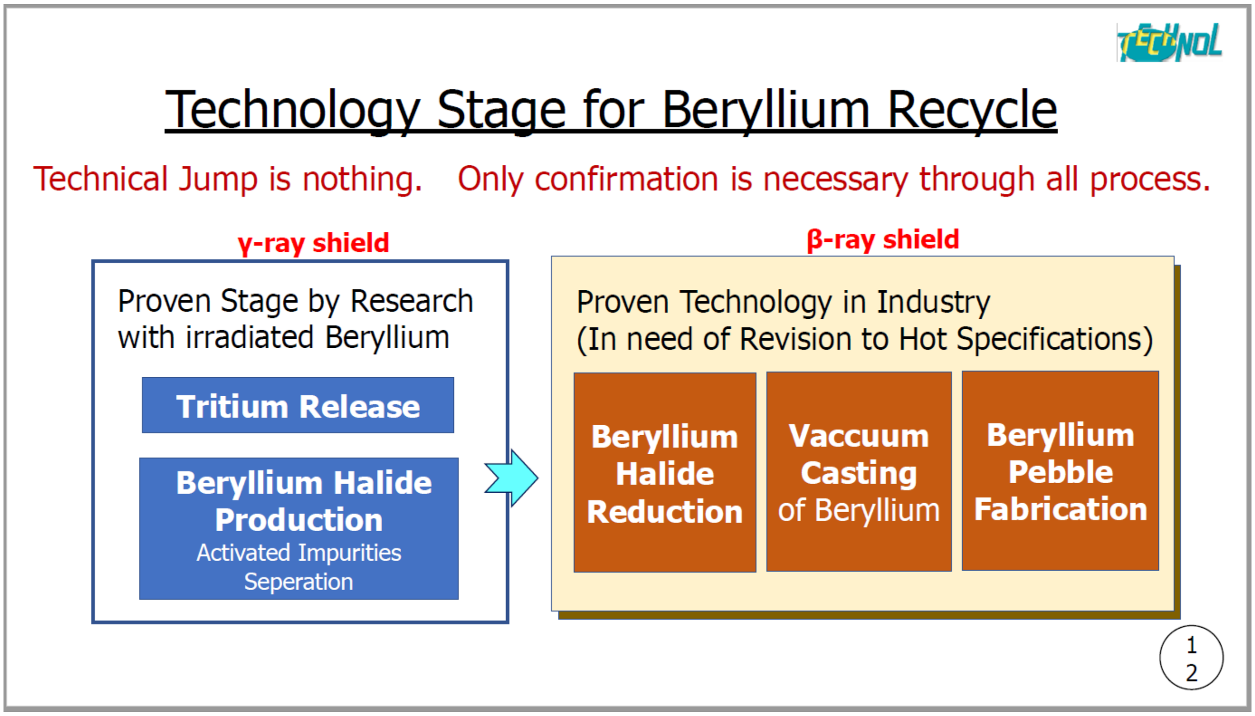

Concept of Beryllium Recycle
(Development Challenges) Effective Application of Used Neutron Reflectors
Fuel (Red part)
Cross-sectional view of the core of the WWR-K
Beryllium Part of Neutron Reflector
Halogen Gas
~500℃
Sublimation
Beryllium Halide
Reduction by Mg
Beryllium
Impurities in beryllium grains
(High Radioactive Waste)
Beryllium Electrode Production
Vacuum casting
Beryllium Pebble
Rotating electrode method
Beryllium Pebble Production
Creation of a beryllium recycling industry
(Securing Beryllium Resources)
7

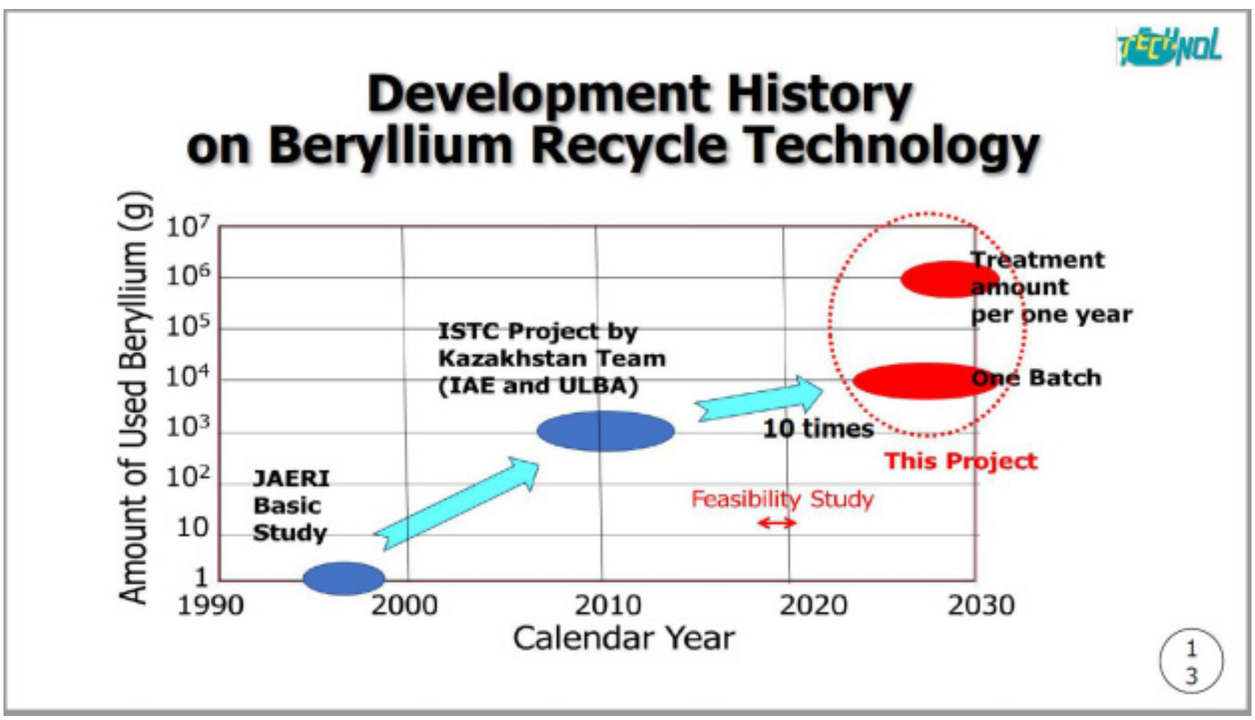

TECHNOL
JOB Flow
JOBs of ordered Side
· Cutting of Beryllium Part from used Neutron Reflector
(Example) Neutron Reflector of JMTR
Handle (SUS304)
Upper Adapter (A6061)
Neutron Reflector (Beryllium)
Lower Adapter (A6061)
First Step
Cutting
Not allow radioactive waste to be brought into Kazakhstan
8

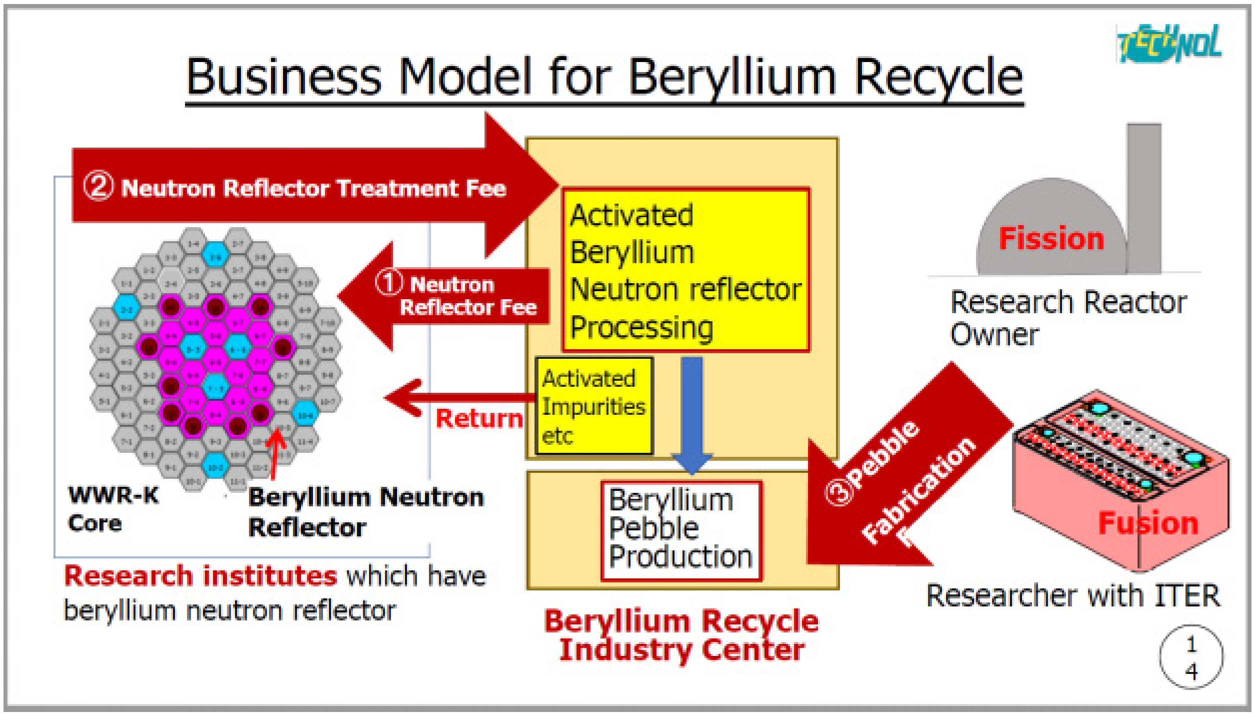

TECHNOL
JOB Flow
JOBs of ordered Side
· Cutting of Beryllium Part from used Neutron Reflector
· Cutting of Beryllium Part for high efficient treatment
Neutron Reflector (Beryllium)
Second Step
Subdivision (cutting shorter than 100mm)
Rationalization of reprocessing equipment
If reprocessing price become higher, reprocessing fee will become higher.
large facility establishment
9


JOB Flow
JOBs of ordered Side
· Cutting of Beryllium Part from used Neutron Reflector
· Cutting of Beryllium Part for high efficient treatment
· Tritium Recovery from Beryllium Part
Cut Beryllium
Third Step Tritium Collection
Heating rate : 10°C/min
Flux (x10⁻¹² mole/s)
Temperature (°C)
Tritium release peaks : 900 to 1200°C
(I-220H : about 1150°C)
✓ Not allow radioactive waste to be brought into Kazakhstan
✓ Rationalization of reprocessing equipment
10


JOB Flow
JOBs of ordered Side
· Cutting of Beryllium Part from used Neutron Reflector
· Cutting of Beryllium Part for high efficient treatment
· Tritium Recovery from Beryllium Part
(*) Yellow process is new.
Beryllium Halide Recovery
Reduction to Beryllium Metal
Beryllium Bar Fabrication by Vacuum Casting
Beryllium Pebble Fabrication
Separation/Recovery of activated Impurities
Treatment of Beryllium Neutron Reflector
Refabrication of Recovered Beryllium
JOBs of received Side (Beryllium Recycle Center)
Semi-industrial scale experiment was finished already.
11


Technology Stage for Beryllium Recycle
Technical Jump is nothing. Only confirmation is necessary through all process.
γ-ray shield
Proven Stage by Research with irradiated Beryllium
Tritium Release
Beryllium Halide Production
Activated Impurities Seperation
β-ray shield
Proven Technology in Industry
(In need of Revision to Hot Specifications)
Beryllium Halide Reduction
Vaccuum Casting of Beryllium
Beryllium Pebble Fabrication
12


Development History on Beryllium Recycle Technology
Amount of Used Beryllium (g)
10⁷
10⁶
10⁵
10⁴
10³
10²
10
1
1990
2000
2010
2020
2030
Calendar Year
JAERI Basic Study
ISTC Project by Kazakhstan Team (IAE and ULBA)
10 times
Treatment amount per one year
One Batch
This Project
Feasibility Study
13



Provisional Overall Schedule

Theme	2019	2020	2021	2022	2023	2024	2025	2026
Feasibility Study ✓IAEA ✓Reactor Operation Side (USA,EU, etc) ✓ITER User Side, etc	←→	C&R						
Commercialization Study			←→	←→	C&R			
Business Development					C&R ←→	←→	←→	START

(＊) C&D : Check and Review

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Feasibility Study Meeting on Beryllium Recycle

1. Meeting Name: Japan-Kazakhstan Joint Committee on Beryllium Recycle Project
2. Objectives: Search for the possibility of beryllium recycle project
3. Structure Draft :

	Kazakhstan Side	Japanese Side
Member	Investment Committee	Chiyoda Technol Co.
	Industrial Development and Industrial Safety Committee	Sumitomo Co.
	Kazatomprom	NGK Insulators, LTD
	ULBA	Nippon Express
	Institute of Nuclear Physics	Japan Atomic Energy Agency
	National Technology Safety Center	Embassy of Japan
Secretariat	Kazakh Invest	Chiyoda Technol Co.

(Remarks) Kick-off Meeting will be held on 11th Nov. of 2019.

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Items of Feasibility Study on Beryllium Recycle

1. Overview
2. Business description
3. Business organization
4. Business location
5. Commercialization schedule
   ① Market research ② Contents and schedule of Confirmation and verification test
   ③ License acquisition (exporting to each country and importing into Kazakhstan)
   ④ Financing ⑤ Site securing (including examination inside and outside ULBA)
   ⑥ Material equipment contract / delivery (including domestic procurement in Kazakhstan and import from Japan)
   ⑦ Equipment installation ⑧ Start of business ⑩ Sustainability
6. Production plan (including annual processing business scale)
7. Sales plan
8. Personnel plan
9. Logistics
10. Authorization related
11. Investment relationship
12. Company formation
13. Summary

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Summary

- In the first stage (feasibility study), we investigate various data for business plans and the determination of business forms shown as follows,
  - ✓ Requirements from owner of research reactor
  - ✓ Laws and regulations concerning work-line management, Methods for treating residual radioactive impurities
  - ✓ Beryllium handling technology in Kazakhstan
- In the second stage (commercialization study), we will consult reasonably with the following partner organizations and related ministries and agencies. Finally, we will start our business for beryllium pebble supply from 2026 year.
- Partners are shown as follows
  - ✓ Kazatomprom and ULBA that is one of only two companies which have systematic beryllium treatment technology all over the world.
  - ✓ Institute of Nuclear Physics that owns research reactor WWR-K with PIE Technology.
  - ✓ NuclearTechnology Safety Center that is familiar with domestic regulations,etc.
  - ✓ NGK that is the company with Beryllium Pebble Fabrication Know-how.
  - ✓ Nippon Express that is one of representatives on worldwide logistics.
  - ✓ Sumitomo Corporation that is the representative trading company on worldwide business.

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Related Ripple Effect

- 191 years has passed since the name " BERYLLIUM" was born. CTC would like to realize Beryllium Recycle in an indomitable spirit up to 200 years anniversary from name birth.
- The beryllium recycle is indispensable for Fusion Demonstration Reactor which uses a lot of beryllium pebbles. If we were successful, effective application technology of neutron multiplier may be demonstrated by using recycled beryllium pebbles for the blanket module test with ITER.

I proposed the beryllium recycle before ~20 years, first.
Today, I propose the beryllium recycle, again.
The beryllium recycle is a common issue for Fission and Fusion Field.
Let us promote the beryllium recycle together !!

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Session 2: Alternative Blanket Concepts & Beryllide Intermetallic Compounds

On Beryllium for Tritium Breeding Blankets: Resource Availability, Alternatives and Determining the Optimum Route to Commercial Fusion

R. Pearson (The Open University, UK) et al.

On beryllium for tritium breeding blankets: resource availability, alternatives and determining the optimum route to commercial fusion

Richard J. Pearson and William J. Nuttall

School of Engineering & Innovation, The Open University, United Kingdom

Beryllium is a critical material for DT fusion reactors, particularly for its use as a neutron multiplier in tritium breeding blankets. However, the requirement for just one GW-e scale fusion DEMO reactor is around 400 tons and the global beryllium industry produces just 300 to 400 tons per year.

Beryllium production would have to increase significantly to support the demands of an emerging fusion industry. Furthermore, terrestrial reserves of beryllium are scarce; estimated at around 100,000 tons, it is enough to support a maximum of only ~250GW-e of installed fusion power plants. This is indicative of a broader problem that seemingly long-term challenges that may present a showstopper for the development of commercial fusion, such as the availability of beryllium resource, are affected by near-term decisions.

Here, we provide a strategic analysis concerning the choice of breeding blanket for future DT fusion reactors. We take a commercial perspective on a problem which has, up until now, been fundamentally considered to be a technical challenge. Alternative breeding blanket materials and designs, specifically those using lead or uranium, are considered against beryllium from the perspective of availability, supply chain performance, cost and technological viability.

The strategic differences between these breeding blanket options, as well as the knock-on effects on lithium-6 enrichment and the availability of blanket coolants (specifically helium) are also considered. Through this, we seek to ensure that a systems' view of all possible challenges that may affect commercial viability.

Finally, in the event that smaller, cheaper fusion devices can be developed on rapid timescales by private fusion organizations, we detail the challenges to developing breeding blankets on such accelerated timescales. From this, we aim to understand whether a faster route to commercial fusion is feasible, and indeed whether such a faster route is preferable to current public programs using large devices on slower timescales, from the perspective of breeding blanket development.

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The Open University, School of Engineering & Innovation
Milton Keynes, MK7 6AA
UNITED KINGDOM


BeWS-14 || 24 October 2019 || Long Beach, CA, USA
EPSRC
The Open University
centre for doctoral training
On beryllium for tritium breeding blankets:
Resource availability and determining the optimal route to commercial fusion
Richard Pearson || PhD student – fusion innovation, strategy and policy || The Open University


Contents
1. Fuels for fusion and the need for tritium breeding blankets
2. Fusion start-ups: an innovation paradigm shift
3. Analysis of consumable resources for fusion from commercial, strategic and policy perspective
4. An optimal blanket for fusion reactors (from commercial perspective)
5. A way forward? Going "Beyond"


Fuels for fusion
Deuterium
Policy analysis of deuterium for fusion
Proliferation concerns: supply and use of heavy water for deuterium
Cost is low, but not as low as suggested in previous fusion literature
Deuterium still not a major problem, but it is also not without issue as claimed
Tritium
Tritium supply issue subject of analysis, now well characterised:
Romanian tritium case study (Pearson et al. 2017)
Considered global tritium supply and use, accounting for delays to ITER and impact of other fusion efforts (Pearson et al., 2018)
Considered issues affecting commercial viability:
Cost; helium-3 production; international regulations and trade; proliferation.
Does not affect the serious need for tritium breeding blankets for a commercial DT fusion programme.
3


Types of breeding blanket
Blankets based on several key resources:
Lead or beryllium as a multiplier?
What level of Lithium-6 enrichment?
Helium, water or molten salt as a coolant?
Blanket configuration and specific designs vary – and – the field is open.
All the technical issues are in-hand. However, “commercial” issues have been – largely – “left for later”.
Overt technical focus is typical (and OK) for public science programmes, including fusion. However, commercial focus is crucial if fusion is to be successful.

Commercial drivers for innovation

- We must therefore consider innovation in fusion, and specifically the commercial drivers
- **Innovation = invention + exploitation**
- Scientists are au fait with invention. But inventions need applications to be innovative.
- Inherently, therefore, we must have an invention *and* a market for that invention. Ideas that are *both* technically and commercially feasible **win**.
- This study will explore the commercial issues.

5

Fusion start-ups: An innovation paradigm shift

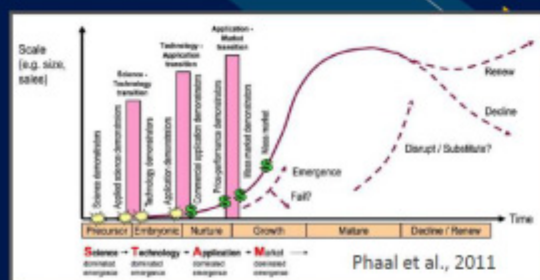
Public fusion programme operates on linear innovation model: *science-product-market*.

True innovation is **dynamic**. Market drivers affect the science, and science finds new discoveries that opens new markets.

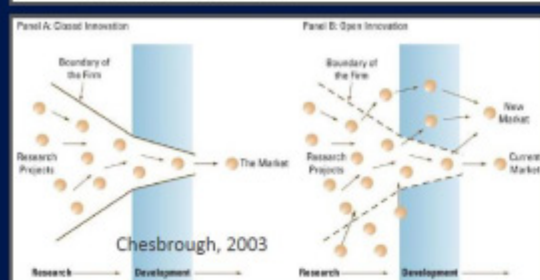
The **public fusion programme** inherently follows a linear model. Low risk approach causes high costs and delays.

Fusion start-ups operate on an *agile* innovation model: not only developing new concepts, but taking risks to **fail and learn fast** (and often) through **rapid** cycles of *build-test-learn*.

Simultaneously, they are aiming at a specific commercial goal, which provides a constraint on the performance (*"good enough"*), cost, and timescales.



Phaal et al., 2011



Chesbrough, 2003

Publication (currently in peer review): **A Paradigm Shift in Fusion: Start-ups, Innovation and Technology Roadmapping for Commercial Fusion Energy**

6

What are the commercial drivers for blankets?

Most studies are necessarily focused on the technical problems, *<u>not</u>* commercial problems. Start-ups are changing this. However, they are focused on plasma problems…

Through policy and strategic analysis, the aims of this research were to drive a new wave of fusion innovation from a similar perspective, but in **breeding blanket innovation.**

Blankets can broadly be divided into the "*consumable*" resources make-up:

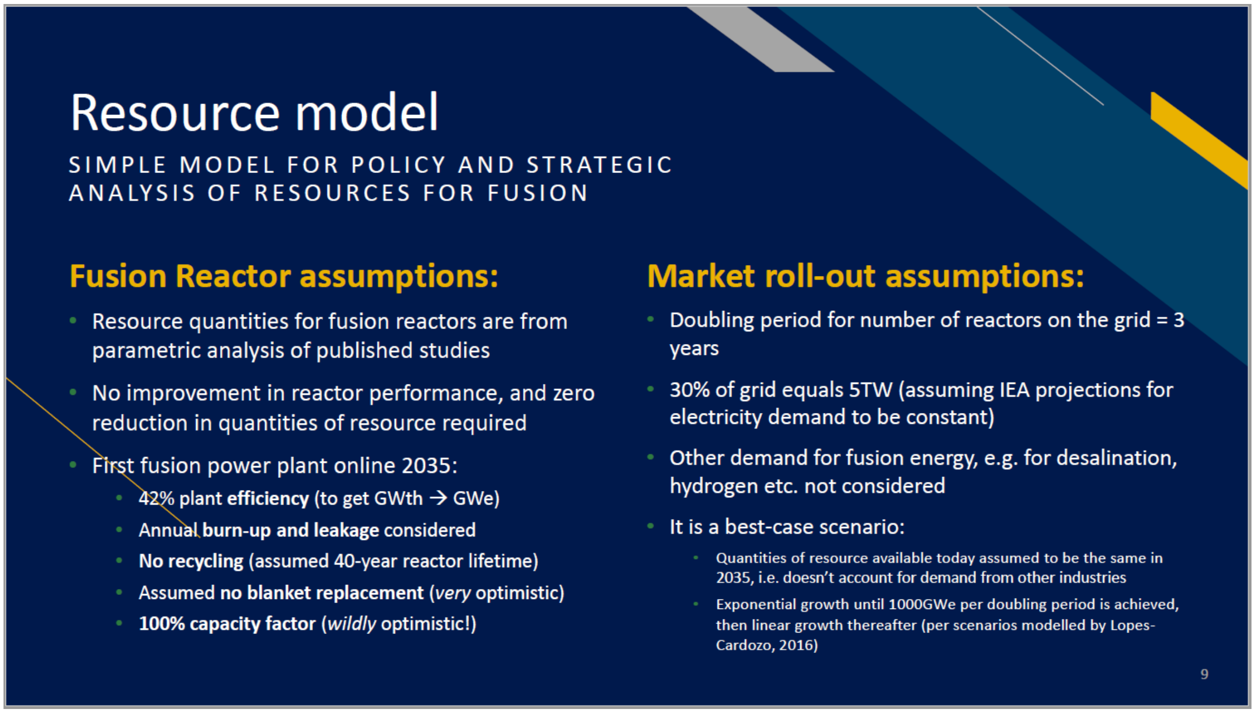
	BREEDER	NEUTRON MULTIPLIER	COOLANT
LEAD-BASED BREEDING BLANKET	Lithium-6 (~90% enrichment)	Lead	Helium (HCLL/DCLL); Water (WCLL)
BERYLLIUM-BASED BREEDING BLANKET	Lithium-6 (~30% enrichment)	Beryllium	Helium (HCPB); Water (WCCB); Molten salt (FLiBe/FLiNaBe))

"Consumable" resources (Li, Be, Pb, He) are directly related to the commercial viability as they drive cost.

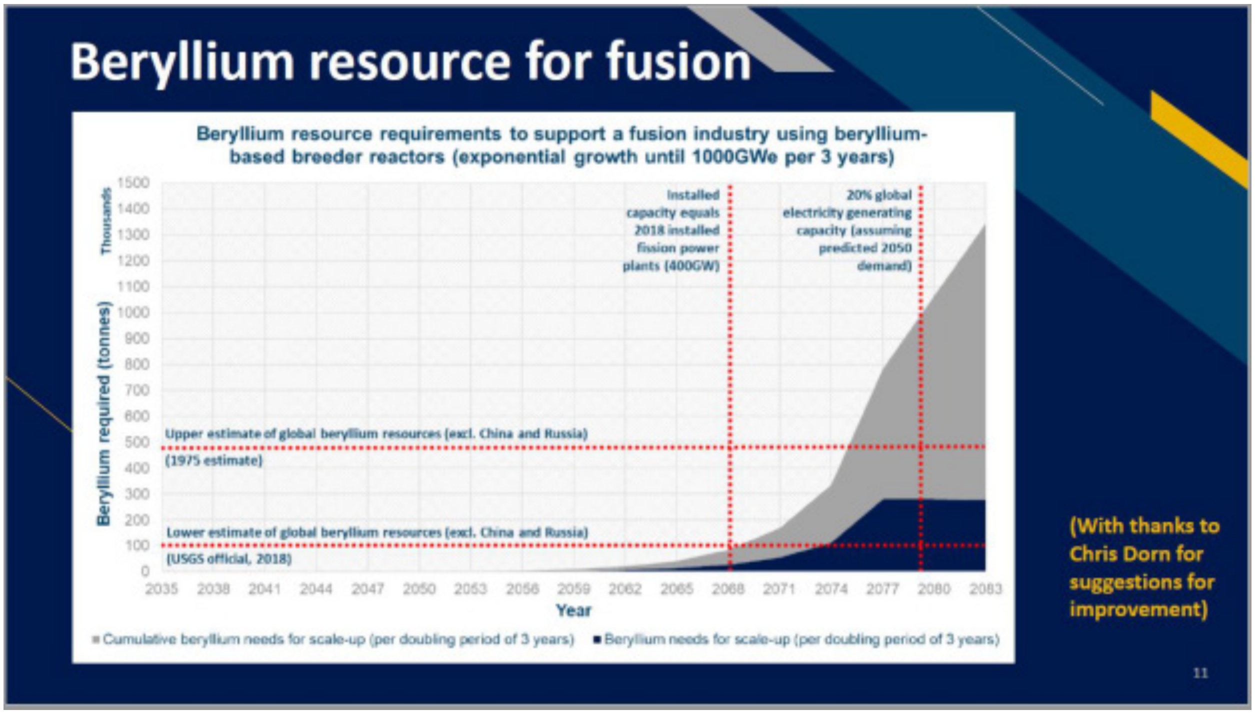
Each of the resources has a unique set of issues. Requires *multi-disciplinary* analysis.

7

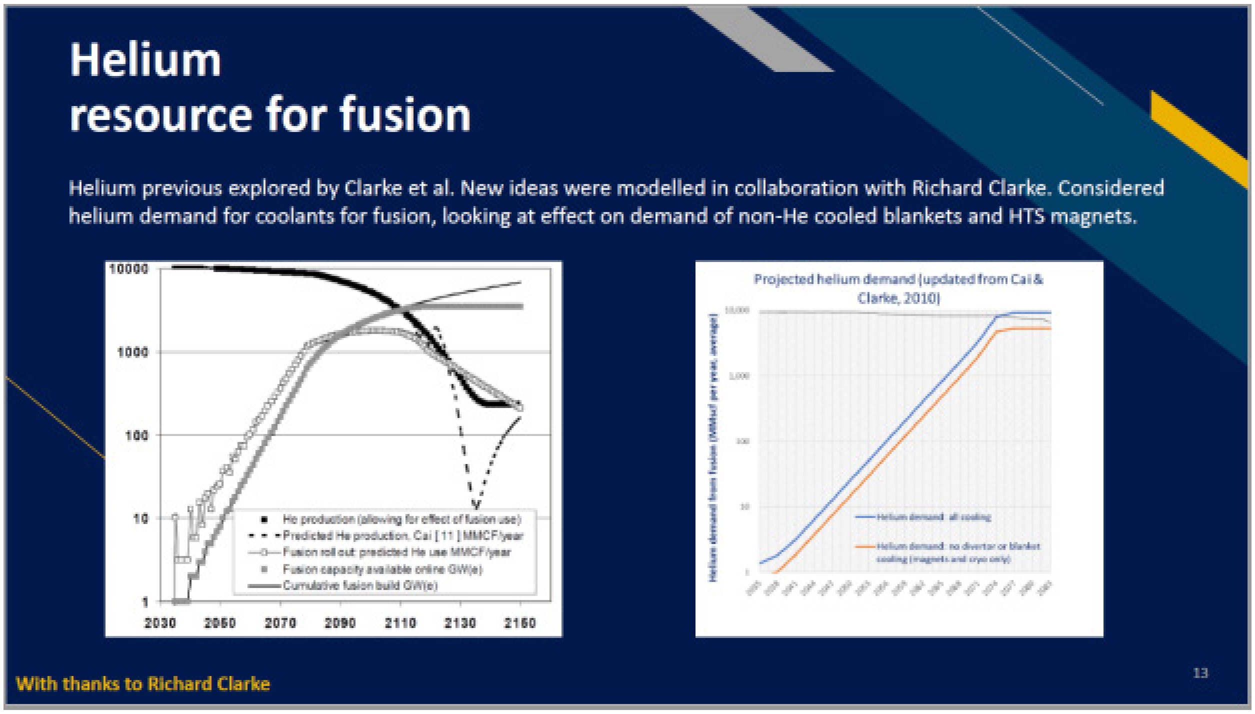
Assessment of resource availability


Resource model
SIMPLE MODEL FOR POLICY AND STRATEGIC ANALYSIS OF RESOURCES FOR FUSION
Fusion Reactor assumptions:
Resource quantities for fusion reactors are from parametric analysis of published studies
No improvement in reactor performance, and zero reduction in quantities of resource required
First fusion power plant online 2035:
42% plant efficiency (to get GWth → GWe)
Annual burn-up and leakage considered
No recycling (assumed 40-year reactor lifetime)
Assumed no blanket replacement (very optimistic)
100% capacity factor (wildly optimistic!)
Market roll-out assumptions:
Doubling period for number of reactors on the grid = 3 years
30% of grid equals 5TW (assuming IEA projections for electricity demand to be constant)
Other demand for fusion energy, e.g. for desalination, hydrogen etc. not considered
It is a best-case scenario:
Quantities of resource available today assumed to be the same in 2035, i.e. doesn't account for demand from other industries
Exponential growth until 1000GWe per doubling period is achieved, then linear growth thereafter (per scenarios modelled by Lopes-Cardozo, 2016)
9


Lithium
resource for fusion
Lithium is very abundant.
Future cost may fluctuate due to demand (batteries!) but it is available in large quantities and there is a significant amount present in seawater
Currently – in effect – global production of lithium-6 is zero
Production is heavily controlled. Other than for use in minute quantities for scientific research – it has only been produced for one purpose.
The United States produced a total of 442 tonnes of lithium-6 from 1954 to 1963 via the COLEX process; no stockpile exists
For 30% electricity grid by fusion, lithium-6 requirement (Bradshaw et al., 2011):
Be-based: 22,000 tonnes
Pb-based: 130,000 tonnes
See: https://www.osti.gov/opennet/forms?formurl=document/press/pc23.html#ZZ7
10


Beryllium resource for fusion
Beryllium resource requirements to support a fusion industry using beryllium-based breeder reactors (exponential growth until 1000GWe per 3 years)
Thousands
Beryllium required (tonnes)
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
Installed capacity equals 2018 installed fission power plants (400GW)
20% global electricity generating capacity (assuming predicted 2050 demand)
Upper estimate of global beryllium resources (excl. China and Russia) (1975 estimate)
Lower estimate of global beryllium resources (excl. China and Russia) (USGS official, 2018)
2035 2038 2041 2044 2047 2050 2053 2056 2059 2062 2065 2068 2071 2074 2077 2080 2083
Year
Cumulative beryllium needs for scale-up (per doubling period of 3 years)
Beryllium needs for scale-up (per doubling period of 3 years)
(With thanks to Chris Dorn for suggestions for improvement)
11


Lead resource for fusion
From the perspective of resource availability, actually… there are no issues!
Lead resource:
Current production* is 4.9MT per year.
Total resource base is over 2 BT.
About 5 years' current production of lead globally is the amount required to power 30% of the electricity grid with fusion (21 MT).
Resource availability, nor its supply chain, an issue for fusion demand for lead
Not to say that lead doesn't have problems, though…
*2016 production value, USGS


Helium
resource for fusion
Helium previous explored by Clarke et al. New ideas were modelled in collaboration with Richard Clarke. Considered helium demand for coolants for fusion, looking at effect on demand of non-He cooled blankets and HTS magnets.
10000
1000
100
10
1
2030
2050
2070
2090
2110
2130
2150
He production (allowing for effect of fusion use)
Predicted He production, Cai [11] MMCF/year
Fusion roll out predicted He use MMCF/year
Fusion capacity available online GW(e)
Cumulative fusion build GW(e)
Projected helium demand (updated from Cai & Clarke, 2010)
Helium demand: all cooling
Helium demand: no divertor or blanket cooling (magnets and cryo only)
With thanks to Richard Clarke
13


61.6 %: 99.19
104.19
86.72
Analysis
from commercial, strategic
and policy perspective


Lithium-6
COLEX process is the most effective way to produce lithium-6. It is complex, energy intensive and environmentally damaging.
Other processes being explored, but are unproven (Ault, 2012).
Lithium-6 est. ~$4000/kg (see Goldston and von Hippel, 2016).
Using current lithium-7 as an analogue, the real cost could be as high as $15,000/kg (Ref: Pederson, UC Berkeley).
Capital cost must also be considered (CapEX and OpEX)
Estimated cost for lithium-6 for a fusion reactor in the order of hundreds of millions. Who pays for this?
15


Lithium-6 (cont.)
Lithium-6 is safeguarded by IAEA for Nuclear Non-proliferation Import and Export Control Regulations (see Canadian Nuclear Safety and Control Act SOR/2000-210)
Actual production capability would need to be in the order of 5000-30,000 tonnes per year* to support a fusion industry wishing to excel beyond 30% grid capacity
Therefore, the issue is not necessarily quantity (and cost), but actually production of lithium-6; full stop.
What is the alternative? Use natural lithium…
*depending on blanket type
16

Beryllium or lead?

Beryllium

- Beryllium is **scarce** and **expensive**
- Beryllium is a **strategic material**. There may be geopolitical concerns over widespread production, distribution and exhaustion.
- For fusion:
  - Recycling of beryllium in fusion reactors may help, but cannot solve the problem (*blanket replacement actually exacerbates*)
  - Supply chain would need to be ramped up for fusion. The capability to increase mine and process capacity will require investment of time, money and effort
  - The beryllium-fusion industrial relationship could become monopolistic/monopsonistic, which presents a big risk for industry

Lead

- Lead may be **abundant** and **cheap**, but there are many technical issues that actually directly affect the commercial viability
- High Lithium-6 **enrichment** affects cost.
- Production of **Po-210** and **Hg-203** (and tritium) means expensive purification systems, presents a safety issue and is a PR concern*.
- Cost of **pumping** and **tritium extraction** could increase cost, although similar costly technical issues exist for beryllium blankets

***fusion produces waste.**

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Helium

- Helium resource may be sufficient for fusion, but it will dominate demand, and it *isn't* cheap…
- Estimated **50 tonnes per GWe**, but 50% of this is for the blanket
- Initial cost **~$85M – $200M** (plus annual top-ups due to leakage).
- Other issues:
  - **Competition** from other industries, including advanced nuclear
  - Helium currently produced using **fossil fuels**, it therefore adds a carbon footprint to fusion. Possible to separate cleanly from air, but expensive and energy intensive.
- Solutions to reduce helium consumption:
  - Pursue **non-helium cooled blanket options** (and divertors)
  - Use **HTS magnets** *not* cooled by helium

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What is the optimal commercial fusion blanket?

Conclusion from last year: "Generation 1" of fusion reactors could use beryllium and helium cooled blankets, but DT fusion reactors based on this would not meet even 20% of the world's energy needs

- Optimal from **commercial perspective**:
  - Pb-based blanket
  - Low-enrichment (*or preferably zero*) lithium-6.
  - Cooled by helium, but could utilise with indirect hydrogen (or Neon) coolant loop (see Glowacki, 2010)). Could be water-cooled*
- The technical perspective may say something different. We must *combine* the two perspectives, to consider technical and commercial drivers simultaneously:
  - Blanket using both Be and Pb, with *reduced* quantities of Be (~10 tonnes?) to provide neutron boost.
  - Can we get to zero lithium-6 enrichment?
  - What about cooling?
- **Bigger picture**: innovative blanket solutions needed, but also we must eventually move away from DT fusion cycle:
  - Fission-fusion? (*uranium for (n,3n) technically attractive, potentially commercial suicide?*)
  - D-D? D-He3? P-B11?
- **Bigger picture, *still***: Even with accelerated fusion, the scale of the climate challenge is vast. We need conventional nuclear near-term!

Water as a coolant limits high performance that force us to ask another commercially-relevant question "why not use a conventional fission reactor?"


Next steps: "Beyond"
We need blanket technology, and we need to speed up the realisation of commercial fusion
21


Our Vision: to become the private industry for blanket development by focusing on commercial opportunities and challenges to accelerate the realization of fusion energy
Kyoto Fusioneering
Launched October 2019. Co-founders: Taka Nagao (Chief Executive), Satoshi Konishi (Chief Technical), Shutaro Takeda (Chief Operations), Richard Pearson (Chief Strategy)
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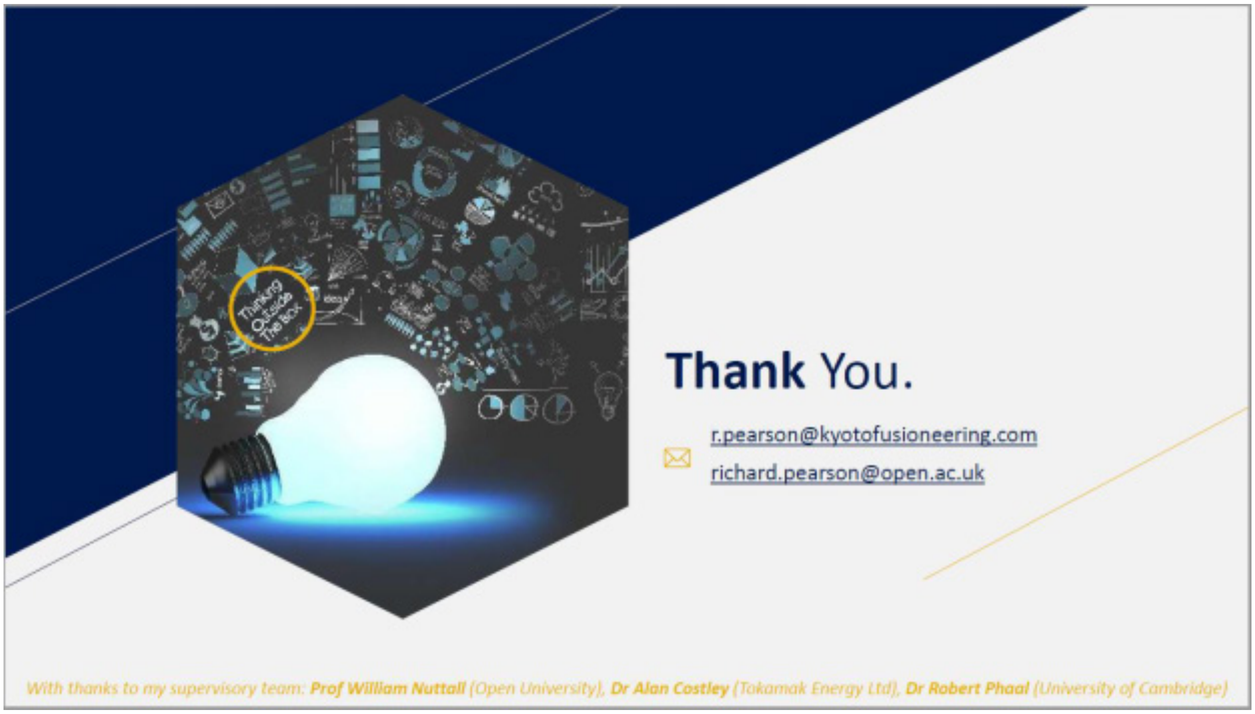
Thinking Outside The Box
Thank You.
r.pearson@kyotofusioneering.com
richard.pearson@open.ac.uk
With thanks to my supervisory team: Prof William Nuttall (Open University), Dr Alan Costley (Tokamak Energy Ltd), Dr Robert Phaal (University of Cambridge)

Overview of beryllium and beryllide R&D as neutron multipliers in QST

Masaru Nakamichi and Jae-Hwan Kim

National Institutes for Quantum and Radiological Science and Technology, Aomori, Japan

DEMO reactors require advanced neutron multipliers that have higher stability at high temperature. Beryllium intermetallic compounds (beryllides) are the most promising advanced neutron multipliers. Development of the advanced neutron multipliers has been started between Japan and the EU in the DEMO R&D of the International Fusion Energy Research Centre (IFERC) project as a part of the Broader Approach activities. In Japan, beryllides fabrication R&D has been carried out in the DEMO R&D building at IFERC, Rokkasho.

Because beryllides are too brittle to allow production of the pebbles, establishing fabrication techniques for beryllides is a key issue for development of the advanced neutron multipliers. Conventional syntheses of the beryllides involve a powder metallurgy process involving a hot isostatic pressing method, a casting method, and an arc-melting method. However, beryllides synthesized conventionally are so brittle that it was not easy to fabricate the block or rod type by these methods.

On the other hand, a plasma sintering method has been proposed as a new technique for beryllides synthesis and joining because this method results in powder surface activation that enhances powder particle sinterability and reduces high temperature exposure. From the results of beryllide synthesis experiments, it was clarified that the not only disk type but rod type of beryllide has been successfully fabricated by the plasma sintering method.

To fabricate the prototypic beryllide pebbles using the plasma-sintered beryllide rod, the rotating electrode method (REM) was selected because the experience base for its use is broad for not only Be pebbles but also metallic pebbles in industry in general. The result of beryllide granulation revealed that the prototypic beryllide pebbles with 1 mm in average diameter were successfully fabricated. In this study, the recent progress on R&D of beryllides as the advanced neutron multipliers in Japan will be presented.

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The 14th International Workshop on Beryllium Technology, BeWS-14,
The Queen Mary, Long Beach, California, USA, 24-25 October2019.

Overview of beryllium and beryllide R&D as neutron multipliers in QST

Masaru NAKAMICHI

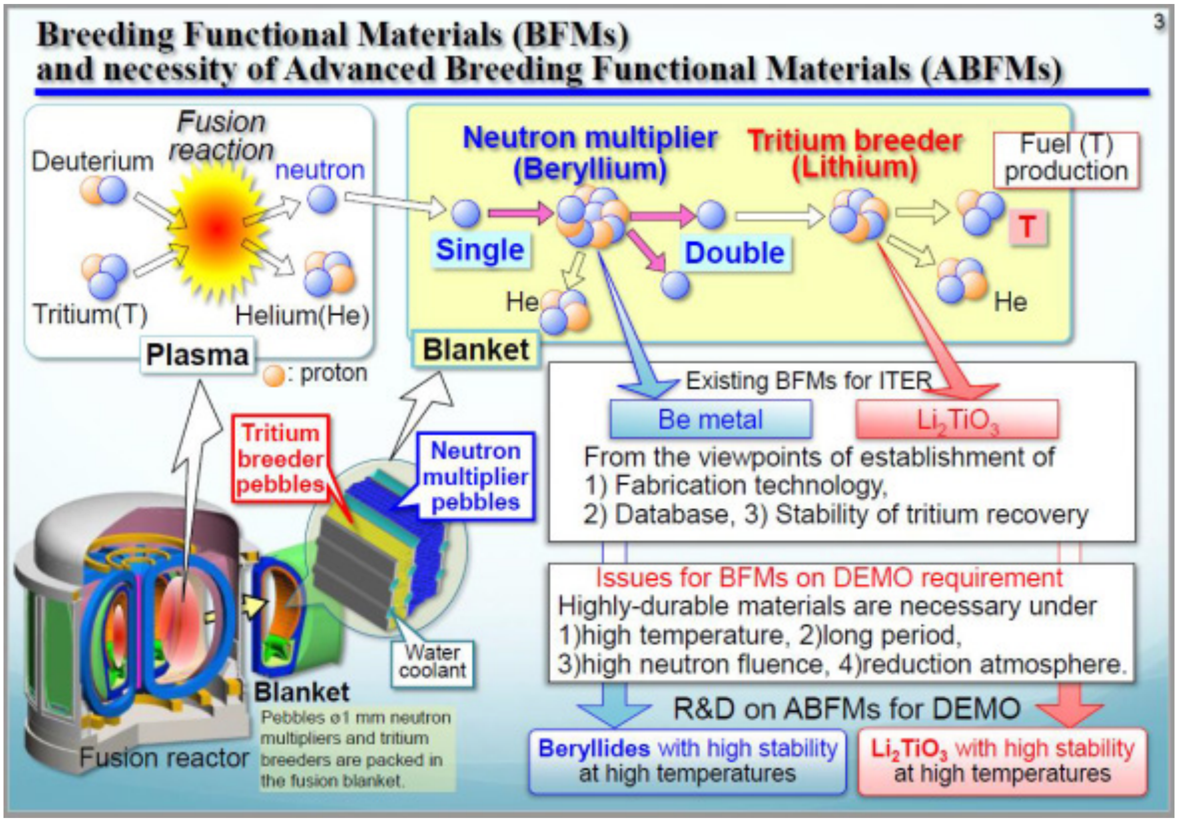
National Institutes for Quantum and Radiological Science and Technology, QST

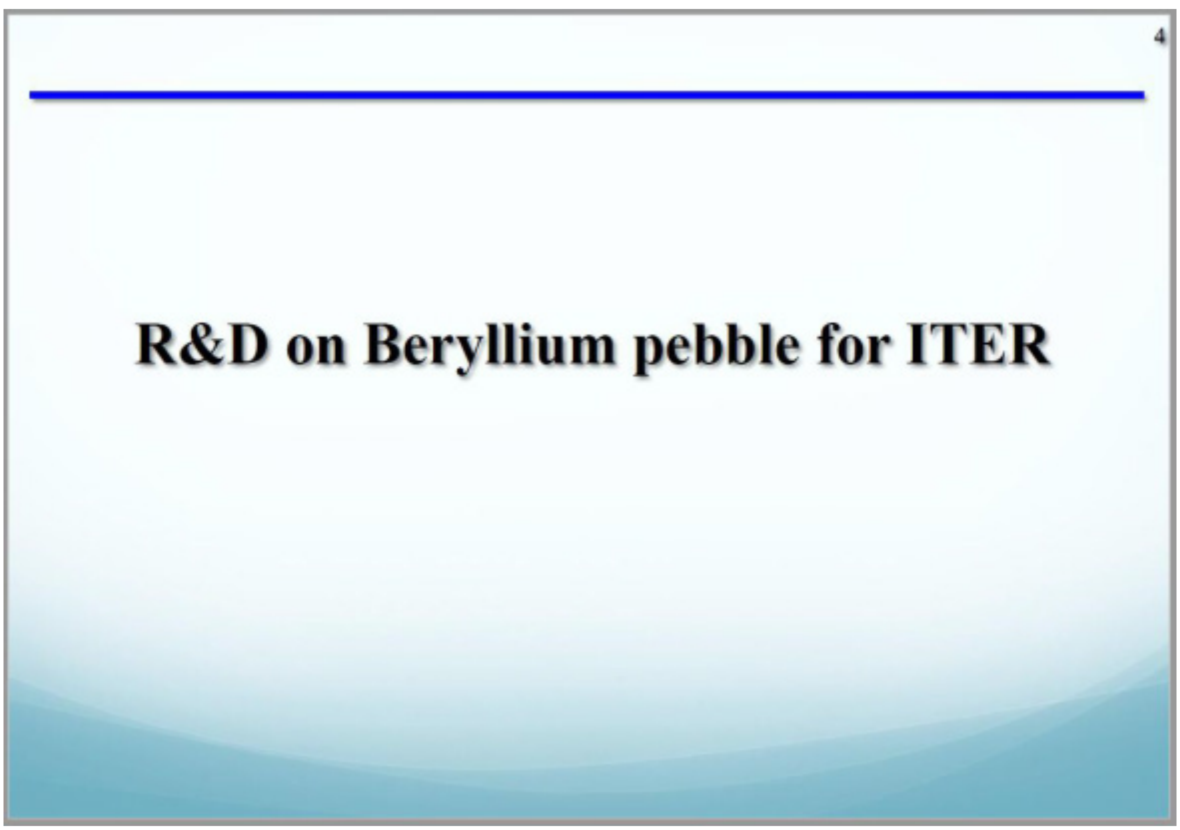
IFERC

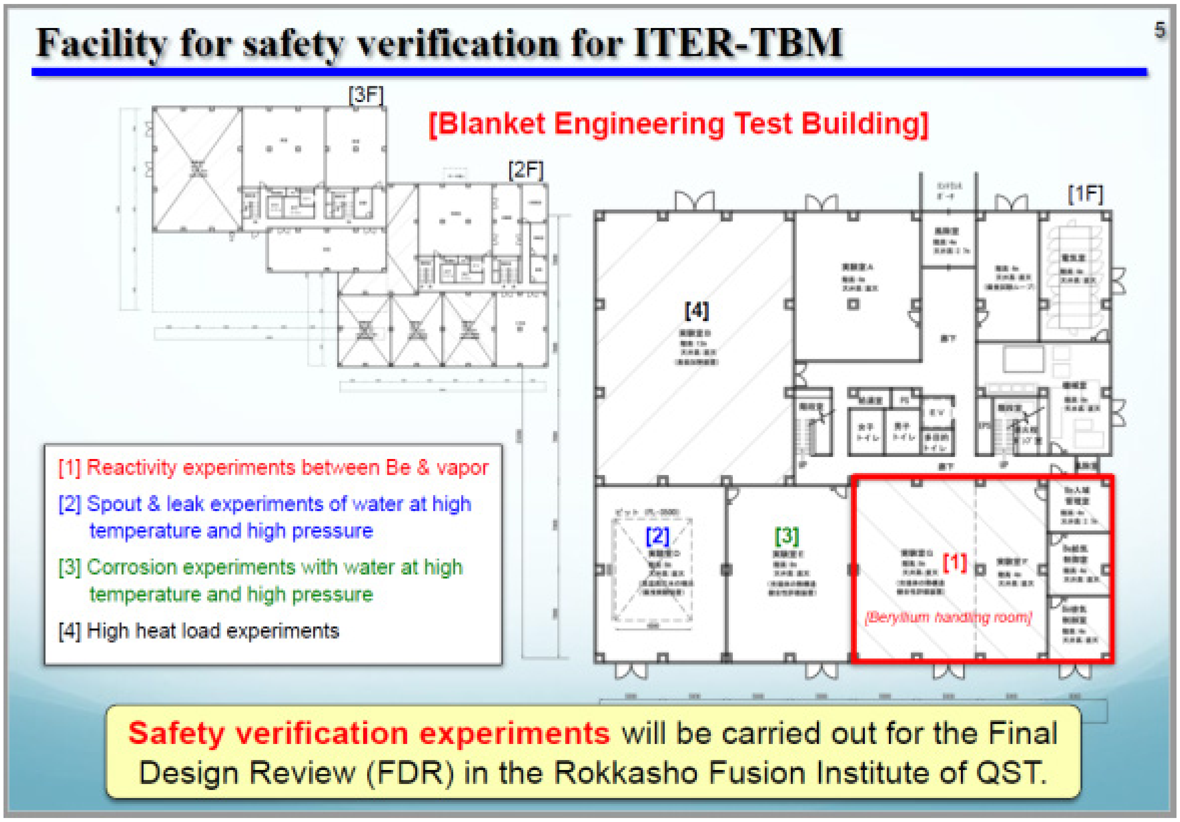
QST

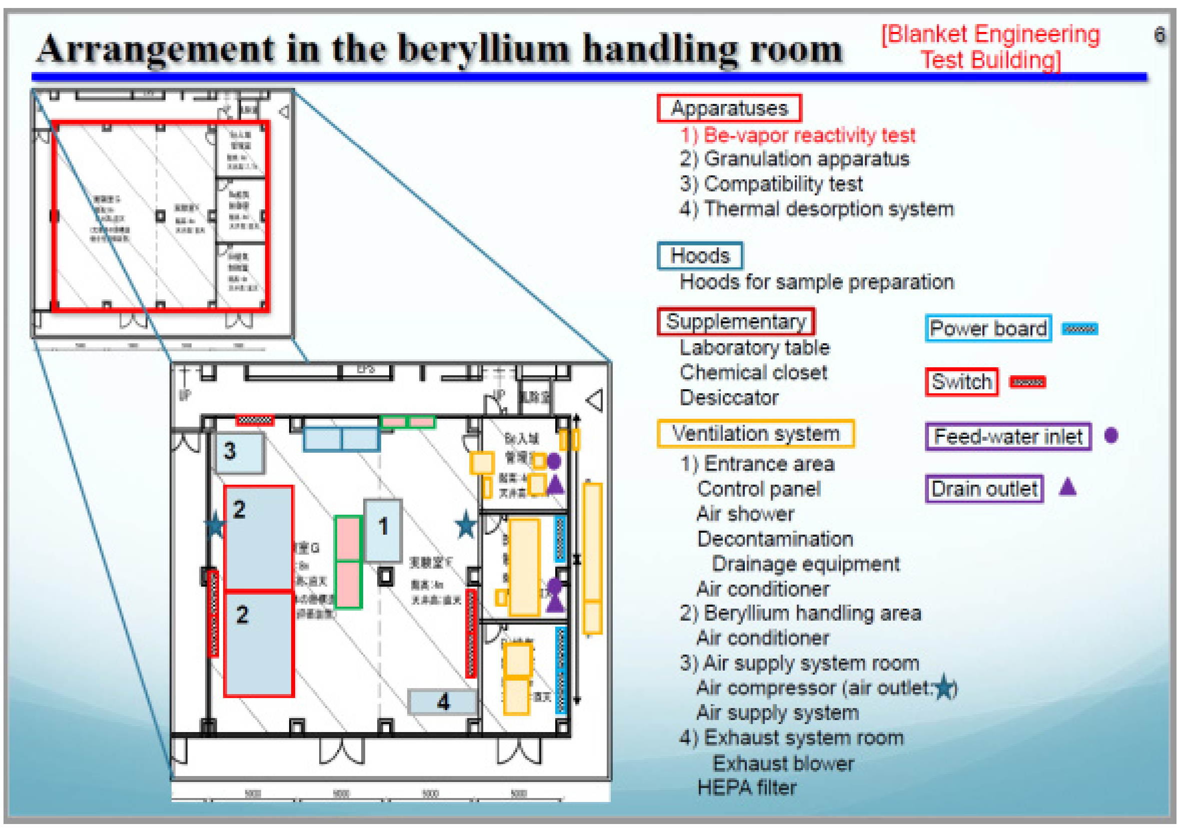
2

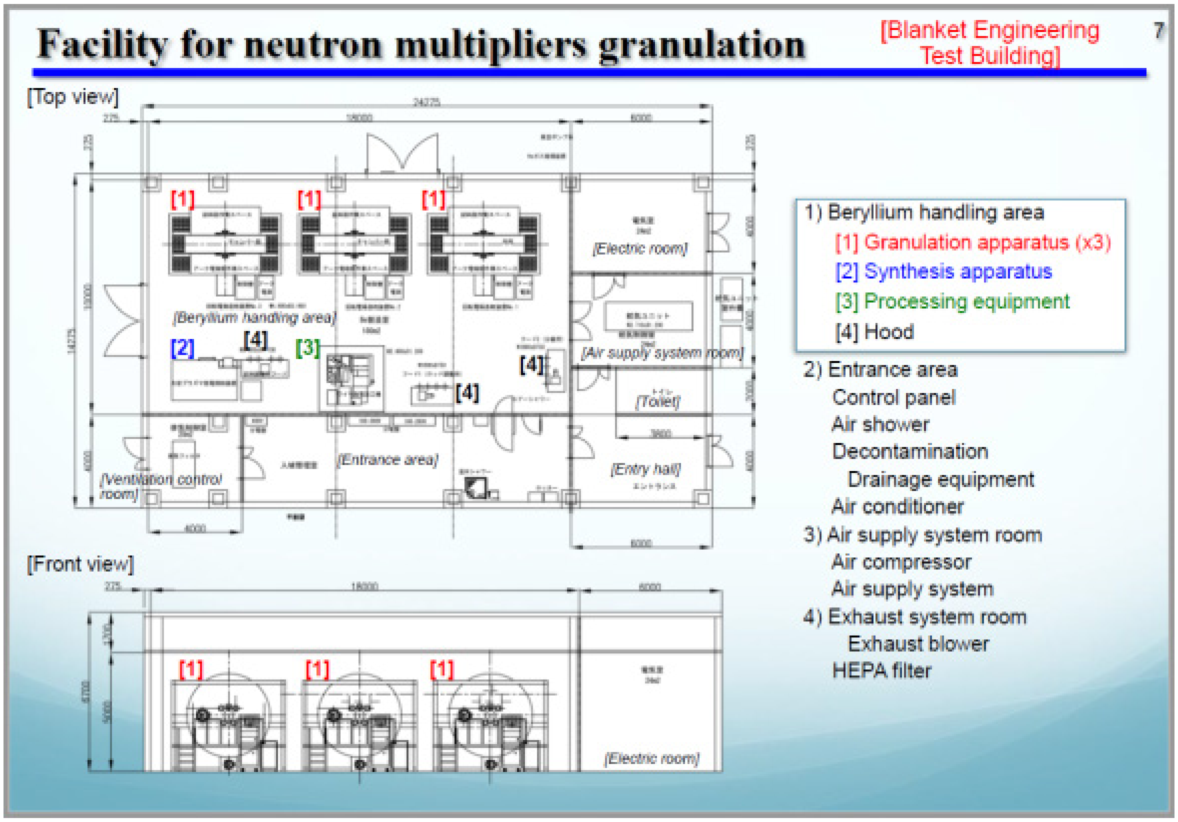
Introduction

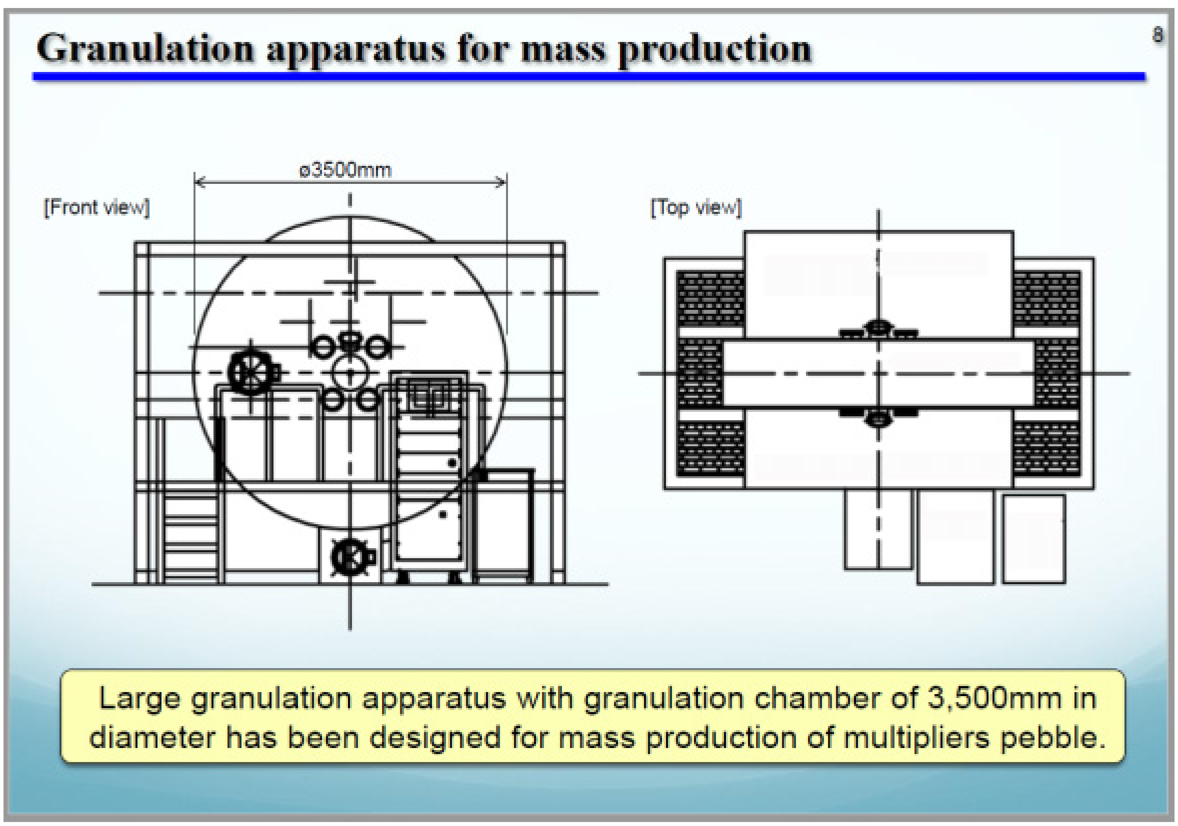

3
Breeding Functional Materials (BFMs)
and necessity of Advanced Breeding Functional Materials (ABFMs)
Fusion reaction
Deuterium
neutron
Tritium(T)
Helium(He)
Plasma
: proton
Neutron multiplier (Beryllium)
Tritium breeder (Lithium)
Fuel (T) production
Single
Double
He
T
He
Blanket
Existing BFMs for ITER
Be metal
Li2TiO3
From the viewpoints of establishment of
1) Fabrication technology,
2) Database, 3) Stability of tritium recovery
Tritium breeder pebbles
Neutron multiplier pebbles
Water coolant
Issues for BFMs on DEMO requirement
Highly-durable materials are necessary under
1)high temperature, 2)long period,
3)high neutron fluence, 4)reduction atmosphere.
Blanket
Pebbles ø1 mm neutron multipliers and tritium breeders are packed in the fusion blanket.
Fusion reactor
R&D on ABFMs for DEMO
Beryllides with high stability at high temperatures
Li2TiO3 with high stability at high temperatures

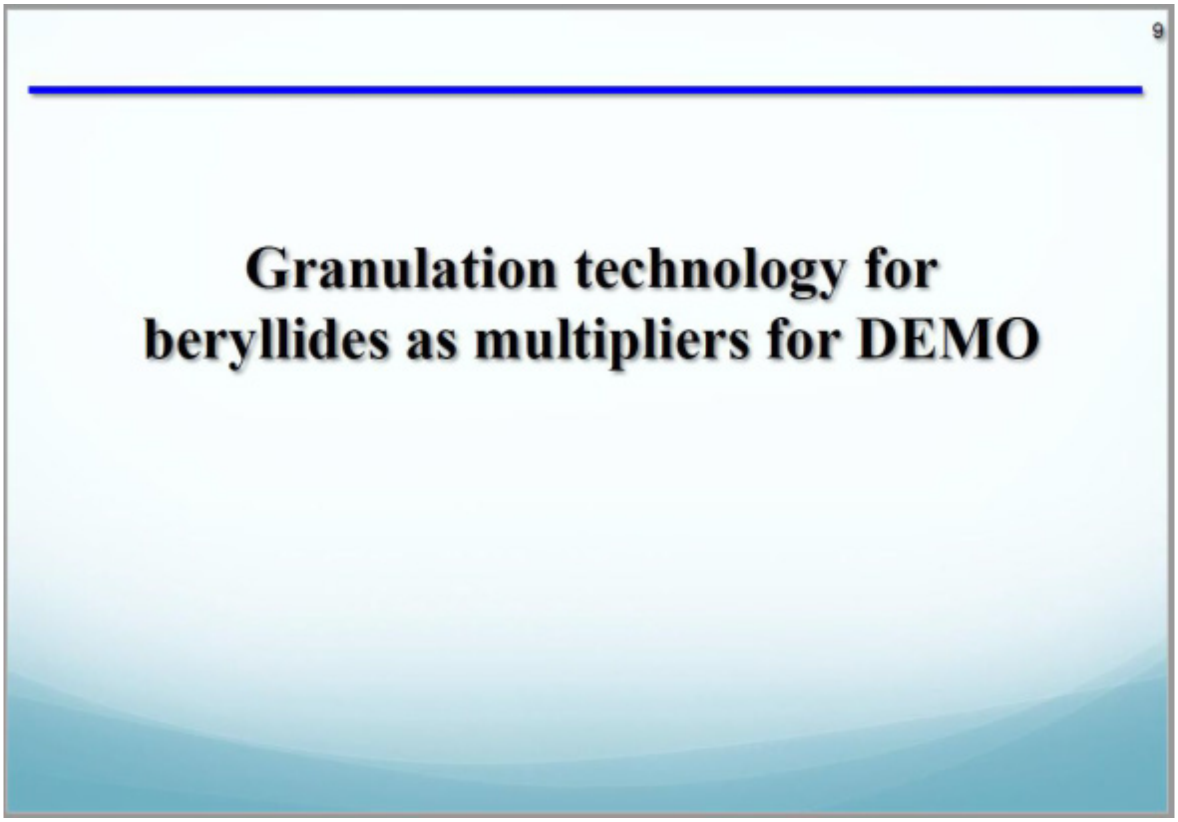

4
R&D on Beryllium pebble for ITER

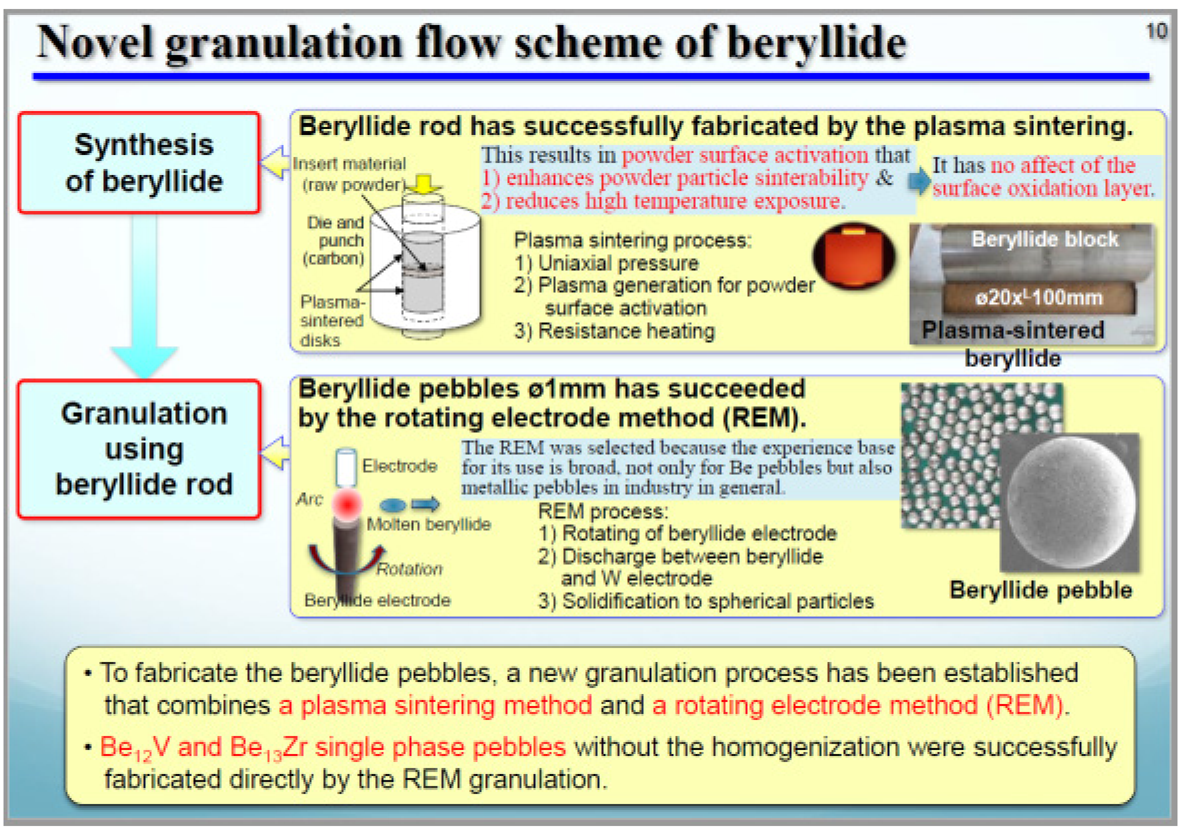

Facility for safety verification for ITER-TBM
5
[3F]
[2F]
[1F]
[Blanket Engineering Test Building]
[4]
[2]
[3]
[1]
[Beryllium handling room]
[1] Reactivity experiments between Be & vapor
[2] Spout & leak experiments of water at high temperature and high pressure
[3] Corrosion experiments with water at high temperature and high pressure
[4] High heat load experiments
Safety verification experiments will be carried out for the Final Design Review (FDR) in the Rokkasho Fusion Institute of QST.

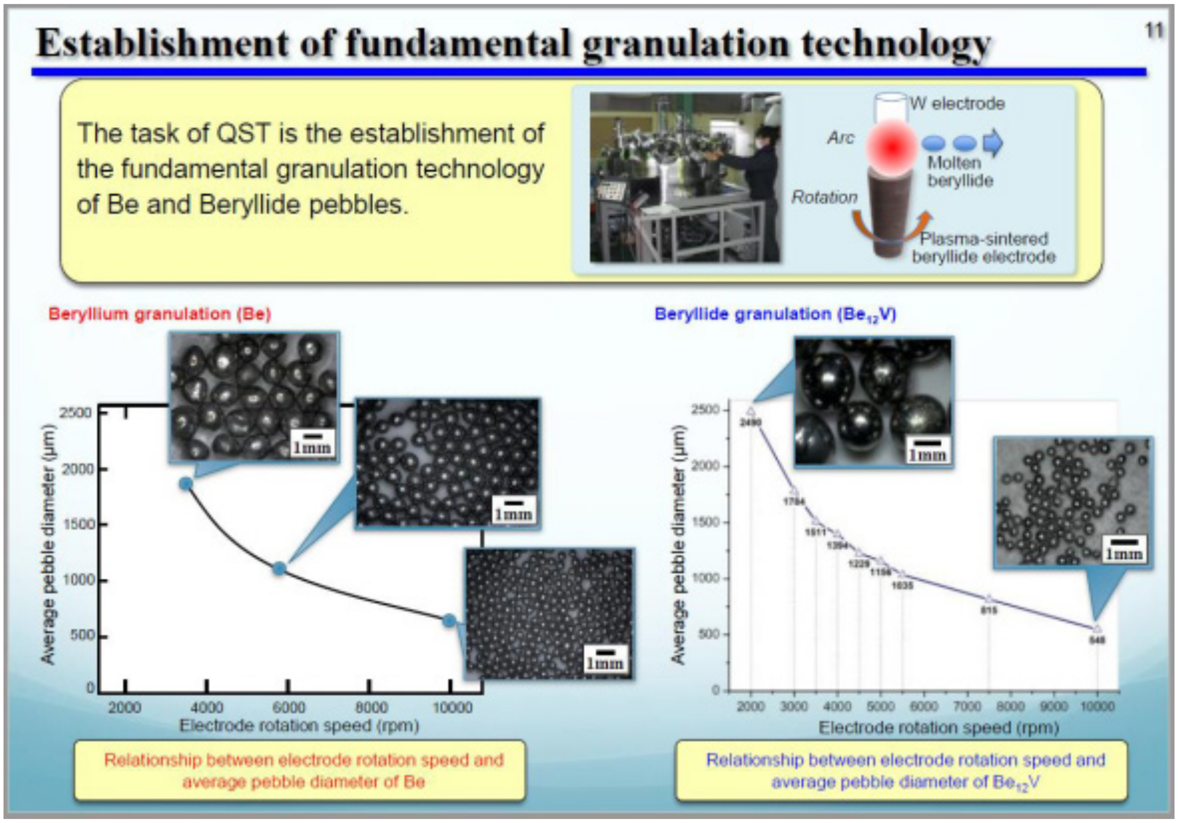

Arrangement in the beryllium handling room
[Blanket Engineering Test Building]
6
Apparatuses
1) Be-vapor reactivity test
2) Granulation apparatus
3) Compatibility test
4) Thermal desorption system
Hoods
Hoods for sample preparation
Supplementary
Laboratory table
Chemical closet
Desiccator
Ventilation system
1) Entrance area
Control panel
Air shower
Decontamination
Drainage equipment
Air conditioner
2) Beryllium handling area
Air conditioner
3) Air supply system room
Air compressor (air outlet:)
Air supply system
4) Exhaust system room
Exhaust blower
HEPA filter
Power board
Switch
Feed-water inlet
Drain outlet
1
2
2
3
4

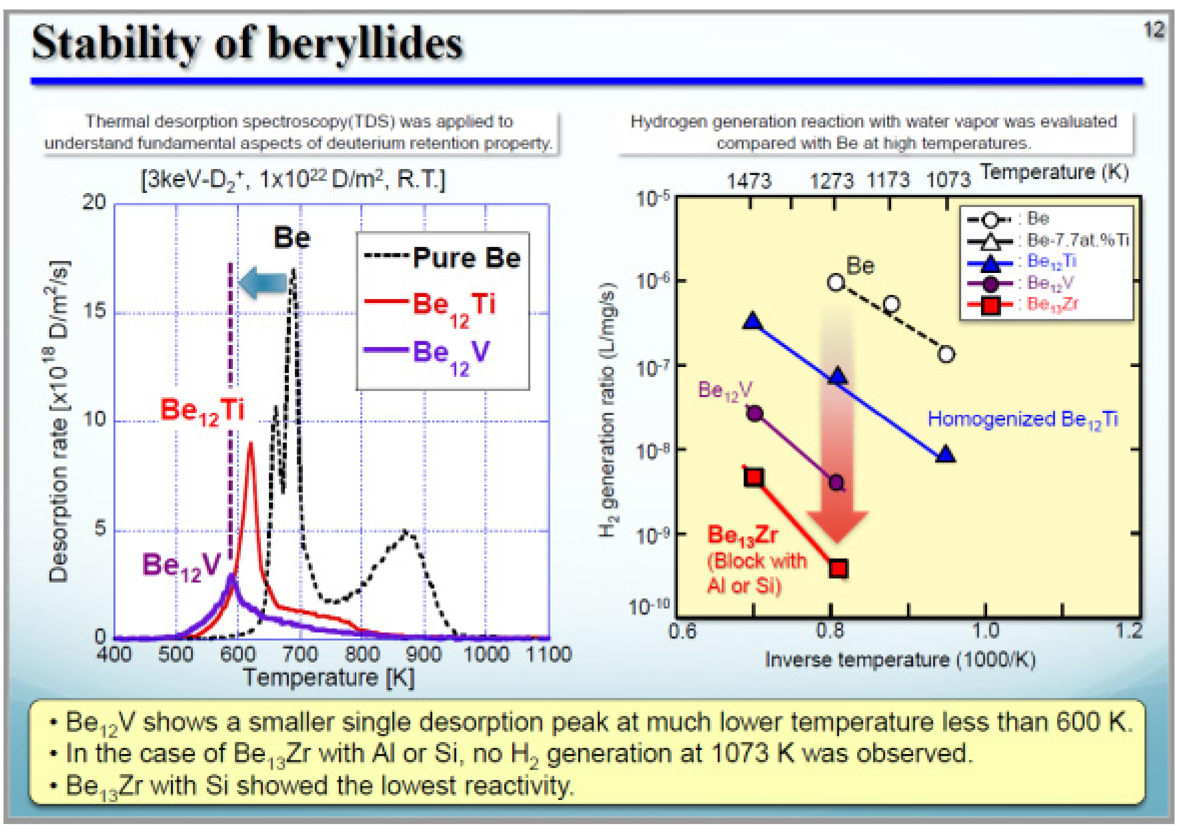

Facility for neutron multipliers granulation
[Blanket Engineering Test Building]
7
[Top view]
[1]
[Electric room]
[Beryllium handling area]
[2]
[4]
[3]
[Air supply system room]
[Toilet]
[Ventilation control room]
[Entrance area]
[Entry hall]
[Front view]
1) Beryllium handling area
[1] Granulation apparatus (x3)
[2] Synthesis apparatus
[3] Processing equipment
[4] Hood
2) Entrance area
Control panel
Air shower
Decontamination
Drainage equipment
Air conditioner
3) Air supply system room
Air compressor
Air supply system
4) Exhaust system room
Exhaust blower
HEPA filter


Granulation apparatus for mass production
8
ø3500mm
[Front view]
[Top view]
Large granulation apparatus with granulation chamber of 3,500mm in diameter has been designed for mass production of multipliers pebble.


9
Granulation technology for
beryllides as multipliers for DEMO


Novel granulation flow scheme of beryllide
10
Synthesis of beryllide
Granulation using beryllide rod
Beryllide rod has successfully fabricated by the plasma sintering.
Insert material (raw powder)
Die and punch (carbon)
Plasma-sintered disks
This results in powder surface activation that 1) enhances powder particle sinterability & 2) reduces high temperature exposure.
It has no affect of the surface oxidation layer.
Plasma sintering process:
1) Uniaxial pressure
2) Plasma generation for powder surface activation
3) Resistance heating
Beryllide block
ø20xL100mm
Plasma-sintered beryllide
Beryllide pebbles ø1mm has succeeded by the rotating electrode method (REM).
Electrode
Arc
Molten beryllide
Rotation
Beryllide electrode
The REM was selected because the experience base for its use is broad, not only for Be pebbles but also metallic pebbles in industry in general.
REM process:
1) Rotating of beryllide electrode
2) Discharge between beryllide and W electrode
3) Solidification to spherical particles
Beryllide pebble
• To fabricate the beryllide pebbles, a new granulation process has been established that combines a plasma sintering method and a rotating electrode method (REM).
• Be12V and Be13Zr single phase pebbles without the homogenization were successfully fabricated directly by the REM granulation.


Establishment of fundamental granulation technology
11
The task of QST is the establishment of the fundamental granulation technology of Be and Beryllide pebbles.
W electrode
Arc
Molten beryllide
Rotation
Plasma-sintered beryllide electrode
Beryllium granulation (Be)
Beryllide granulation (Be12V)
Average pebble diameter (µm)
Electrode rotation speed (rpm)
1mm
Relationship between electrode rotation speed and average pebble diameter of Be
Relationship between electrode rotation speed and average pebble diameter of Be12V


Stability of beryllides
12
Thermal desorption spectroscopy(TDS) was applied to understand fundamental aspects of deuterium retention property.
[3keV-D2+, 1x10^22 D/m², R.T.]
Desorption rate [x10^18 D/m²/s]
Temperature [K]
Be
Pure Be
Be12Ti
Be12V
Hydrogen generation reaction with water vapor was evaluated compared with Be at high temperatures.
Temperature (K)
1473 1273 1173 1073
H2 generation ratio (L/mg/s)
Inverse temperature (1000/K)
: Be
: Be-7.7at.%Ti
: Be12Ti
: Be12V
: Be13Zr
Homogenized Be12Ti
Be13Zr (Block with Al or Si)
• Be12V shows a smaller single desorption peak at much lower temperature less than 600 K.
• In the case of Be13Zr with Al or Si, no H2 generation at 1073 K was observed.
• Be13Zr with Si showed the lowest reactivity.

13

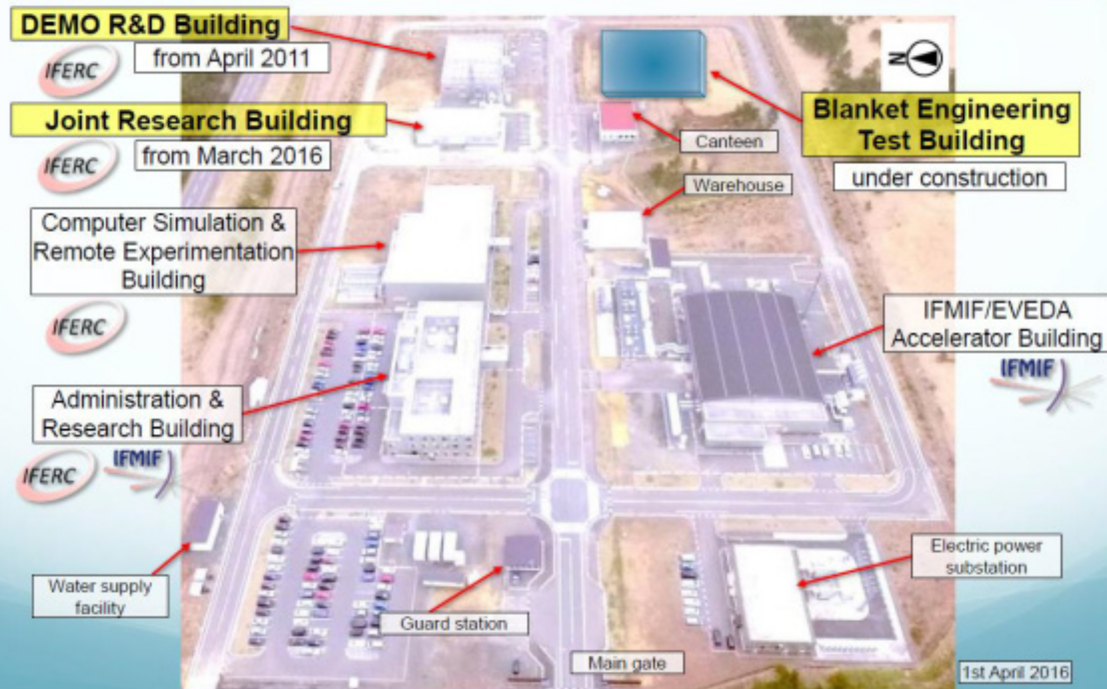
R&D facilities for multipliers in QST strategy for early realization of the DEMO beyond the ITER

14

International Fusion Energy Research Centre (IFERC)

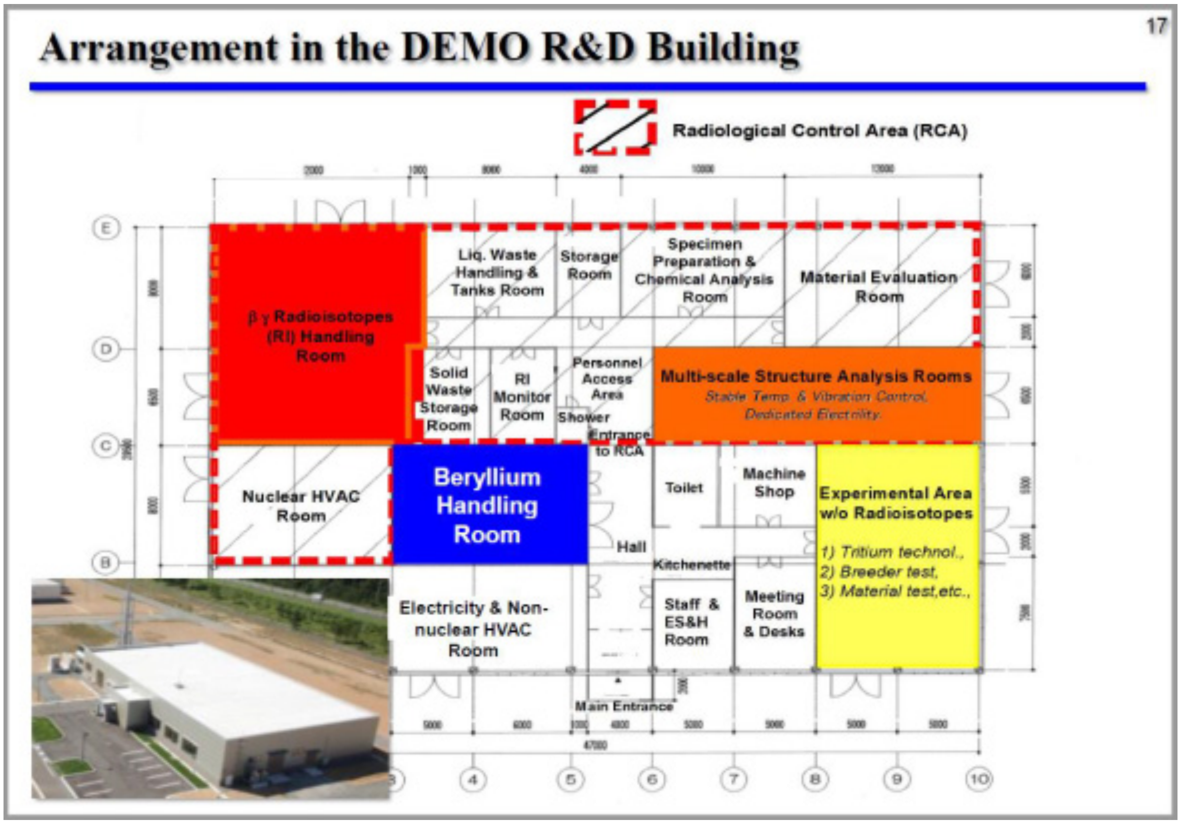
15

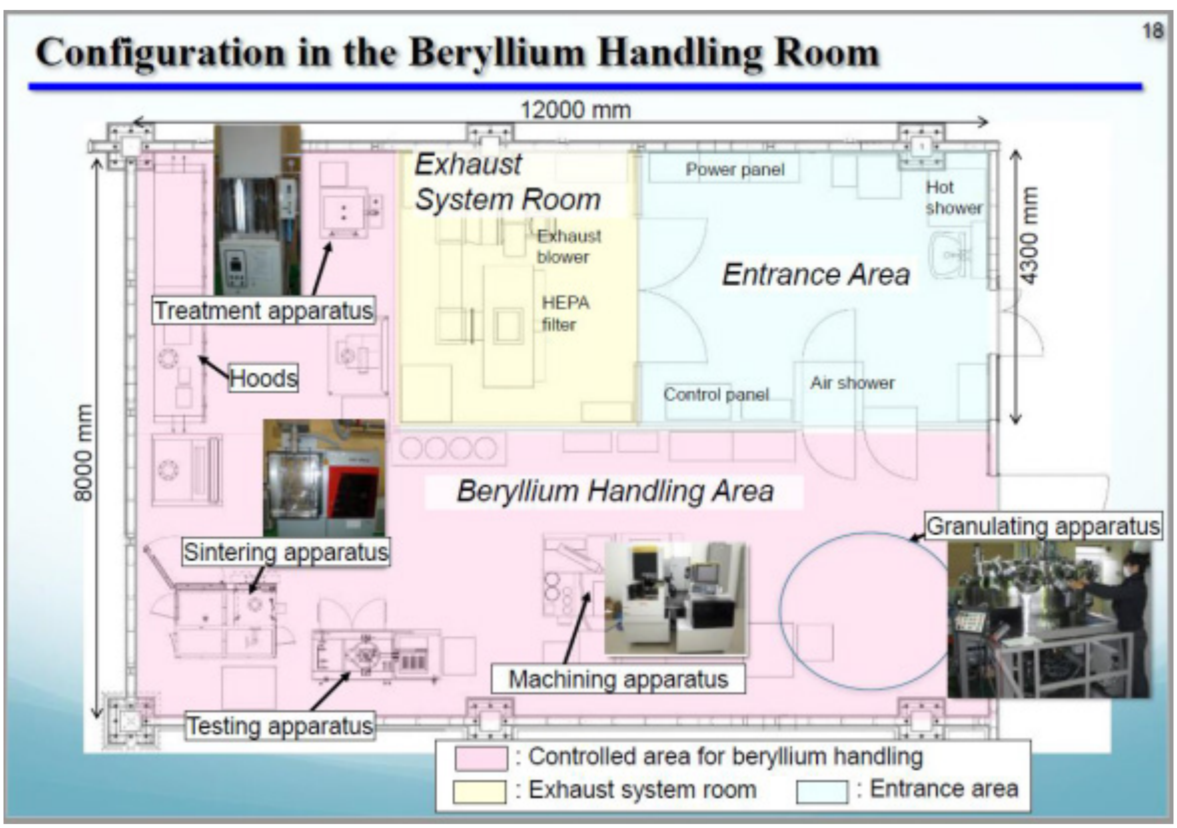
Arrangement in IFERC



16

Beryllium Handling Facility in the R&D building


Arrangement in the DEMO R&D Building
17
Radiological Control Area (RCA)
β γ Radioisotopes (RI) Handling Room
Liq. Waste Handling & Tanks Room
Storage Room
Specimen Preparation & Chemical Analysis Room
Material Evaluation Room
Solid Waste Storage Room
RI Monitor Room
Personnel Access Area
Shower
Entrance to RCA
Multi-scale Structure Analysis Rooms
Stable Temp. & Vibration Control, Dedicated Electrity
Nuclear HVAC Room
Beryllium Handling Room
Hall
Toilet
Machine Shop
Experimental Area w/o Radioisotopes
1) Tritium technol.,
2) Breeder test,
3) Material test,etc.,
Kitchenette
Electricity & Non-nuclear HVAC Room
Staff & ES&H Room
Meeting Room & Desks
Main Entrance


Configuration in the Beryllium Handling Room
18
12000 mm
8000 mm
4300 mm
Exhaust System Room
Exhaust blower
HEPA filter
Power panel
Hot shower
Entrance Area
Control panel
Air shower
Treatment apparatus
Hoods
Beryllium Handling Area
Sintering apparatus
Granulating apparatus
Machining apparatus
Testing apparatus
: Controlled area for beryllium handling
: Exhaust system room
: Entrance area

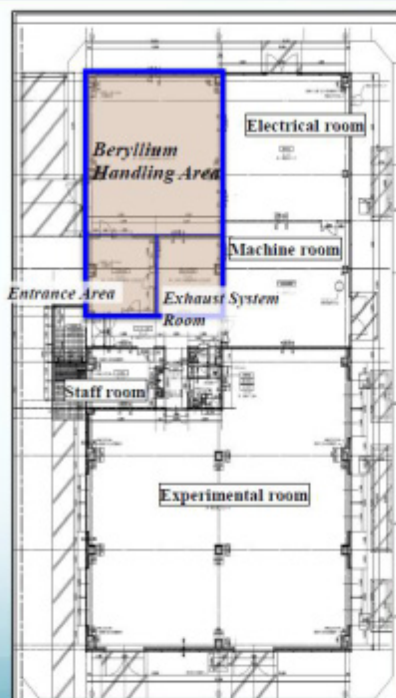
19

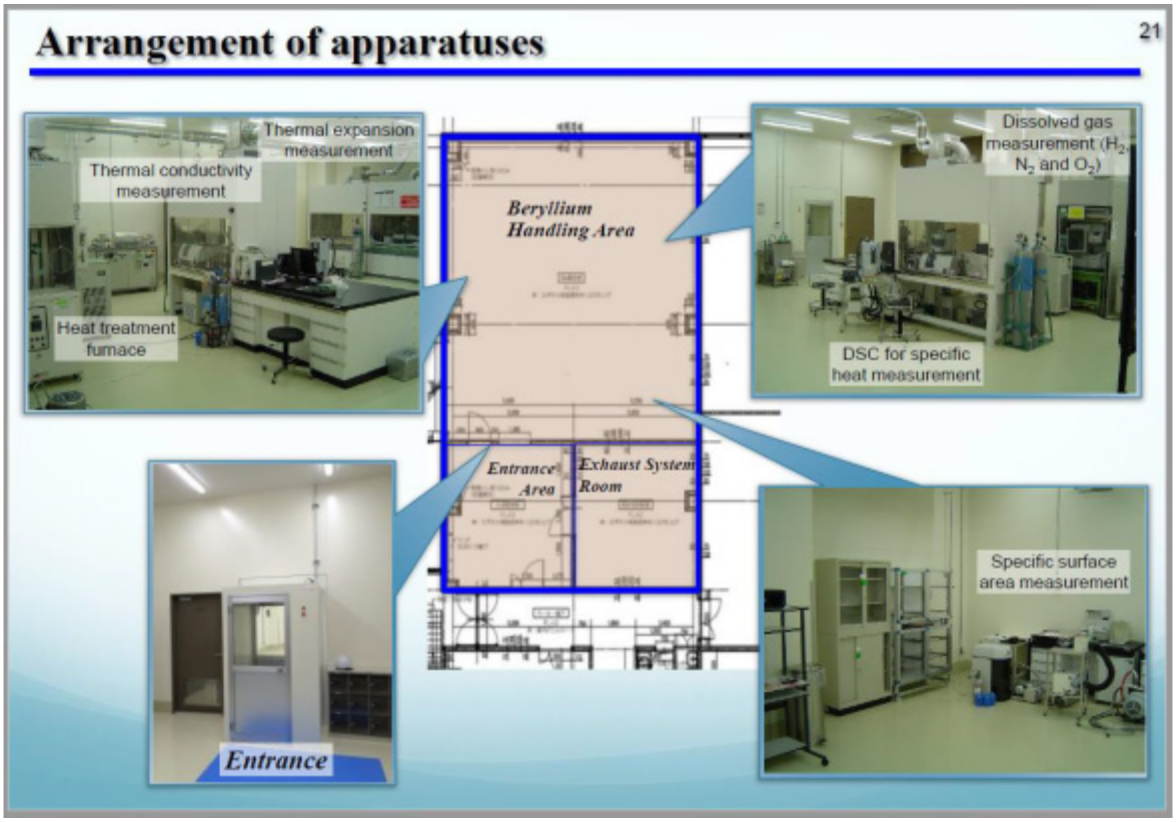
New Beryllium Handling Room in the Joint Research Building

20

Joint Research Building

Joint Research Building





Conclusion of Be production for ITER

22

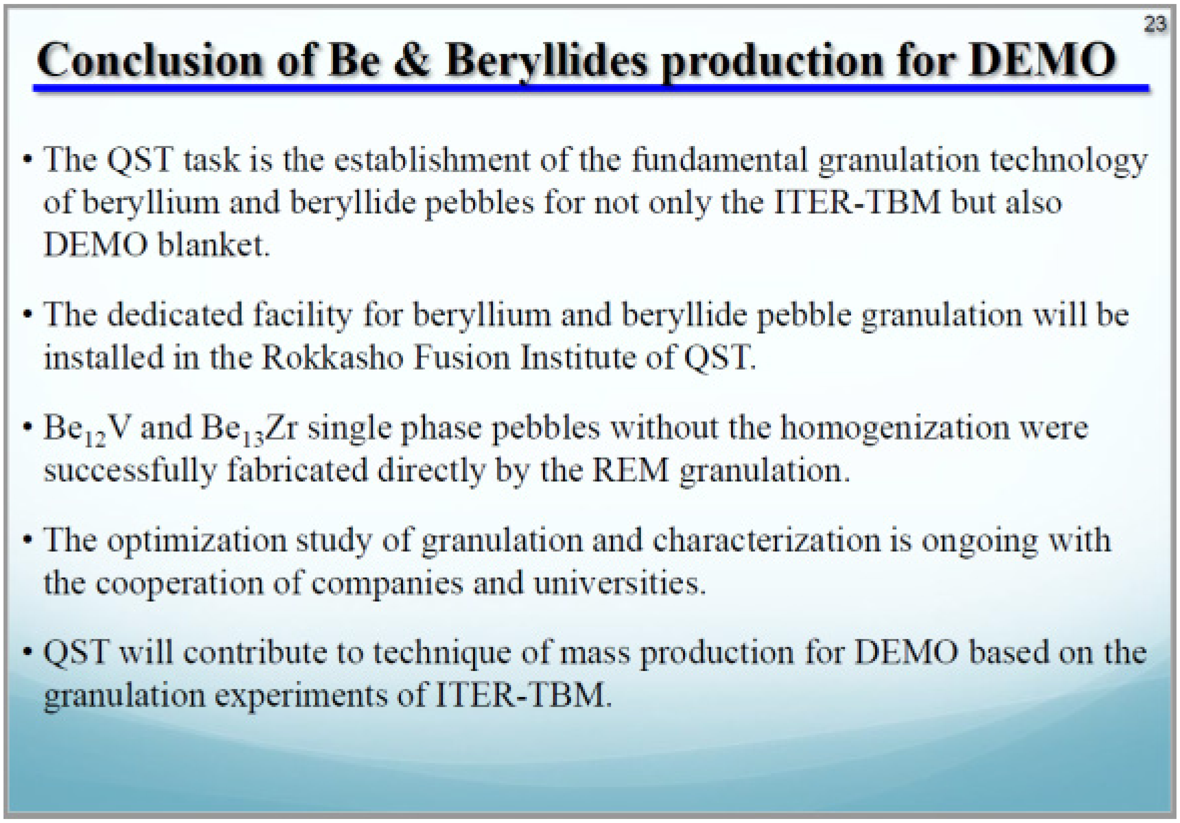
- From the viewpoints of establishment of

  - Fabrication technology,
  - Database of material properties, and
  - Stability of tritium recovery,

  beryllium metal as neutron multiplier and Li_2TiO_3 as tritium breeder have been selected for the candidate materials for ITER-TBM.

- Aiming at approval of the final design review (FDR) of the ITER-TBM, the safety verification experiments will be carried out in the Rokkasho Fusion Institute of QST.

- As to beryllium safety design, reactivity with water vapor will be evaluated in the new beryllium handling room.


23
Conclusion of Be & Beryllides production for DEMO
• The QST task is the establishment of the fundamental granulation technology of beryllium and beryllide pebbles for not only the ITER-TBM but also DEMO blanket.
• The dedicated facility for beryllium and beryllide pebble granulation will be installed in the Rokkasho Fusion Institute of QST.
• $Be_{12}V$ and $Be_{13}Zr$ single phase pebbles without the homogenization were successfully fabricated directly by the REM granulation.
• The optimization study of granulation and characterization is ongoing with the cooperation of companies and universities.
• QST will contribute to technique of mass production for DEMO based on the granulation experiments of ITER-TBM.

Development, characterization & modelling of neutron multiplier materials at KIT

P. Vladimirov[1], D. Bachurin[1], V.P. Chakin[1], R. Gaisin[1], A. Goraieb[2], F. Hernandez[1], M. Klimenkov[1], R. Rolli[1], C. Stihl[1], N. Zimber[1], S. Udartsev[3], M. Kolmakov[3], A. Vechkutov[3], and E. Frants[3]

[1]*Karlsruhe Institute of Technology, 76131 Karlsruhe, Germany*
[2]*Karlsruhe Beryllium Handling Facility, 76344, Eggenstein-Leopoldshafen, Germany*
[3]*Ulba Metallurgical Plant (UMP), Abay Avenue 102, 070005 Ust-Kamenogorsk, Kazakhstan*

Beryllium is an efficient neutron multiplier material, which permits compensation of unavoidable losses of neutrons in tritium breeding blanket and ensures closing fuel cycle of fusion reactor. Until recently, the pebble bed concept with interchanging layers of beryllium and lithium ceramic pebbles was considered for the Helium-Cooled Pebble Bed tritium-breeding module of the first experimental fusion reactor ITER as well as for the next demonstration fusion reactor DEMO.

Beryllium pebbles with a diameter of 1 mm produced by the rotating electrode method were tested in the HIDOBE irradiation campaign. The analytical TEM/EELS studies and tritium retention measurements suggest that significant fraction of generated tritium (up to 100% below 500°C) is trapped within helium bubbles and small gas-vacancy clusters. Being negligible in ITER, the total accumulated tritium inventory imposes critical safety issues as it may exceed the acceptable limits for the DEMO blanket. Thus, advanced neutron multiplier materials should be developed and characterized for their application in DEMO and beyond.

Usage of material with lower volumetric swelling and tritium retention, higher irradiation and chemical resistance as well as with higher melting temperature allows changing from the pebble bed concept to that based on solid hexagonal blocks made of titanium beryllide. Industrial route for fabrication of $Be_{12}Ti$ blocks was developed in cooperation with Ulba Metallurgical Plant, Kazakhstan. Fabricated blocks were successfully tested in 50 cycles of heating and cooling between 900°C and 200°C with heating rate of 12°C/s.

Our recent *ab-initio* calculations indicate lower tritium retention in $Be_{12}Ti$ than in pure beryllium in accordance with already published experimental results.

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Karlsruhe Institute of Technology

DEVELOPMENT AND CHARACTERIZATION OF ADVANCED NEUTRON MULTIPLIER MATERIALS

Pavel V. Vladimirov*, D. Bachurin, V. P. Chakin, R. Gaisin, A. Goraieb, F. Hernandez, M. Klimenkov, R. Rolli, C. Stihl, N. Zimber, S. Udartsev, M. Kolmakov, A. Vechkutov, E. Frants

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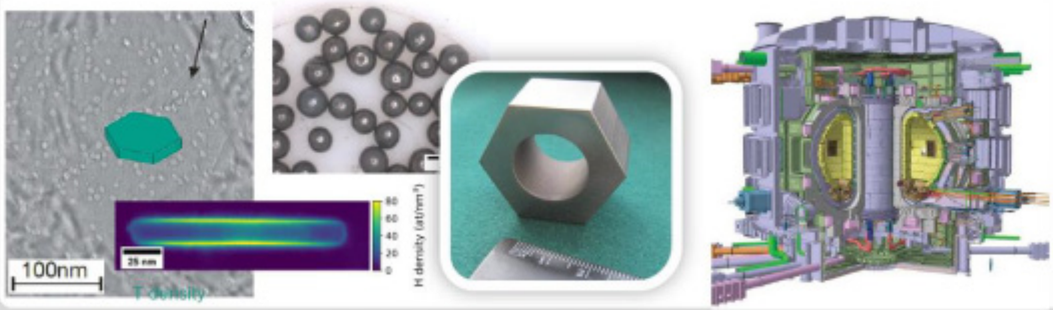


KIT – The Research University in the Helmholtz Association www.kit.edu

Outline

Karlsruhe Institute of Technology

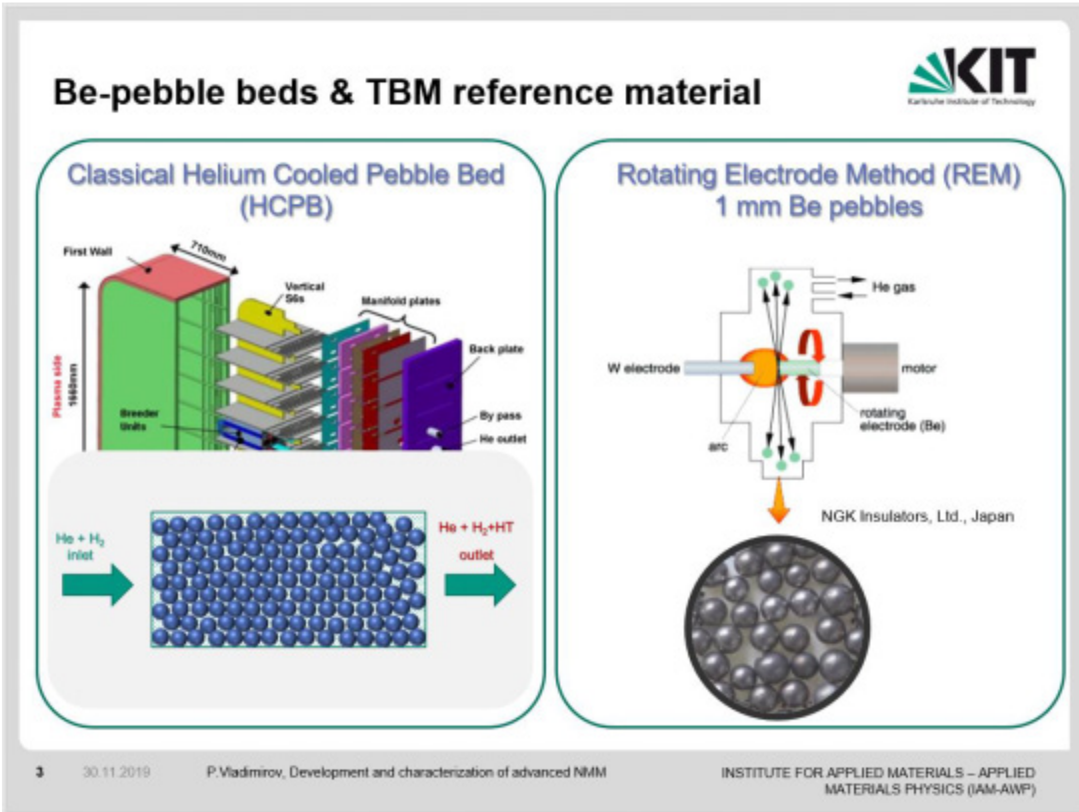
CONVENTIONAL NEUTRON MULTIPLIER - Beryllium

- Beryllium application in T-breeding blankets
  - Be-pebble beds
  - TBM reference material: 1mm NGK pebbles
- Beryllium pebble characterization
  - irradiation campaign HIDOBE
- Accumulation of tritium in Be – where is tritium?
  - Helium bubbles
  - T inside bubbles?
  - T-inventory
- ⇒ Consequences for blanket design

ADVANCED NEUTRON MULTIPLIER - Beryllides

- Advanced HCPB blanket concept
  - advanced neutron multiplier – beryllides
  - industrial scale fabrication?
  - inherent brittleness?
- Conclusions

2 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)


Be-pebble beds & TBM reference material
KIT
Classical Helium Cooled Pebble Bed (HCPB)
First Wall
710mm
Vertical Stiffening plates
Manifold plates
Back plate
Plasma side 1665mm
Breeder Units
By pass
He outlet
He + H2 inlet
He + H2+HT outlet
Rotating Electrode Method (REM) 1 mm Be pebbles
He gas
W electrode
motor
rotating electrode (Be)
arc
NGK Insulators, Ltd., Japan
3
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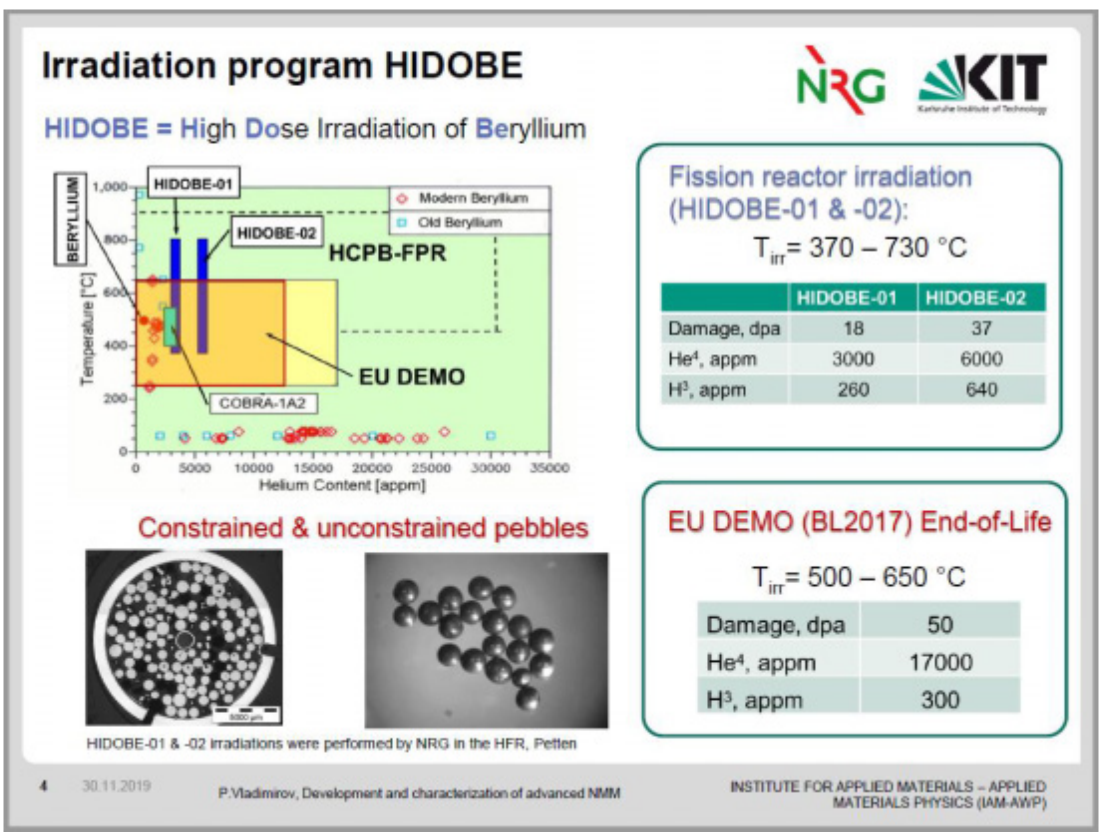
Irradiation program HIDOBE
NRG
KIT
HIDOBE = High Dose Irradiation of Beryllium
BERYLLIUM
HIDOBE-01
HIDOBE-02
HCPB-FPR
EU DEMO
COBRA-1A2
Modern Beryllium
Old Beryllium
Temperature [°C]
Helium Content [appm]
0
200
400
600
800
1,000
5000
10000
15000
20000
25000
30000
35000
Fission reactor irradiation (HIDOBE-01 & -02):
T_irr = 370 – 730 °C
HIDOBE-01 HIDOBE-02
Damage, dpa 18 37
He4, appm 3000 6000
H3, appm 260 640
EU DEMO (BL2017) End-of-Life
T_irr = 500 – 650 °C
Damage, dpa 50
He4, appm 17000
H3, appm 300
Constrained & unconstrained pebbles
HIDOBE-01 & -02 irradiations were performed by NRG in the HFR, Petten
4
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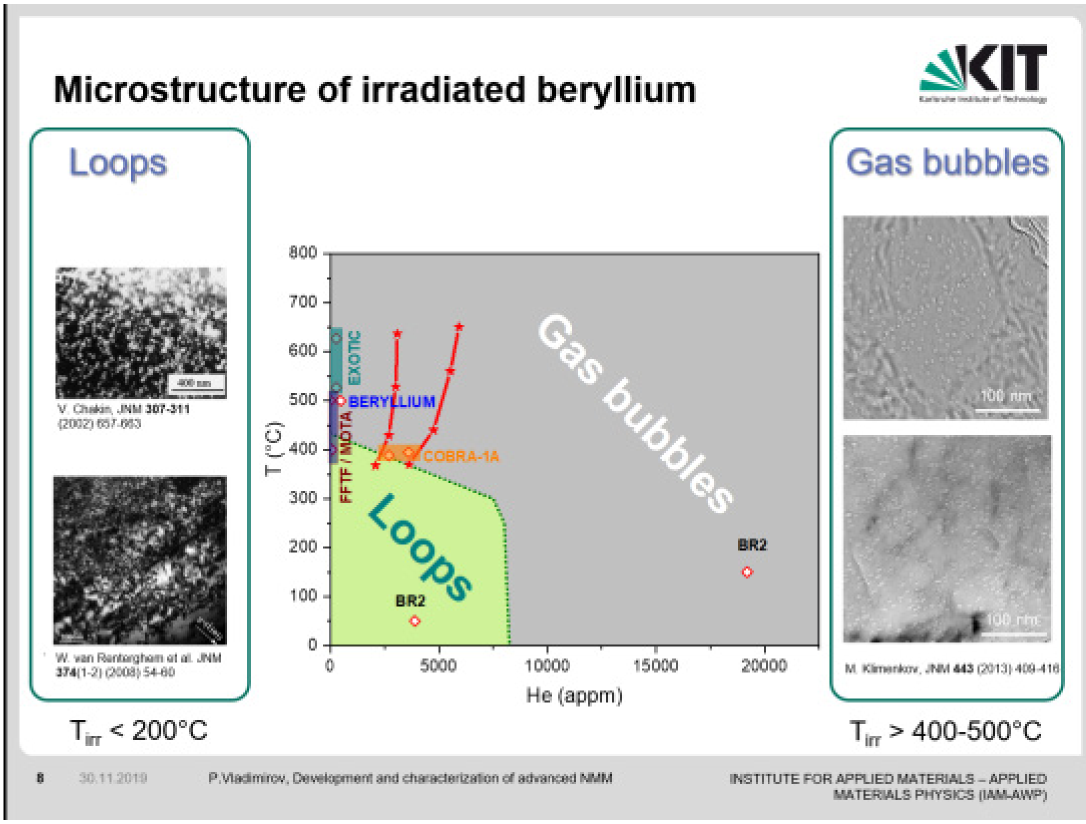
Microstructure of irradiated beryllium
KIT
Loops
V. Chakin, JNM 307-311 (2002) 657-663
W. van Renterghem et al. JNM 374(1-2) (2008) 54-60
T (°C)
He (appm)
Gas bubbles
Loops
EXOTIC
BERYLLIUM
FFTF / MOTA
COBRA-1A
BR2
BR2
Gas bubbles
100 nm
100 nm
M. Klimenkov, JNM 443 (2013) 409-416
T_{irr} < 200°C
T_{irr} > 400-500°C
8
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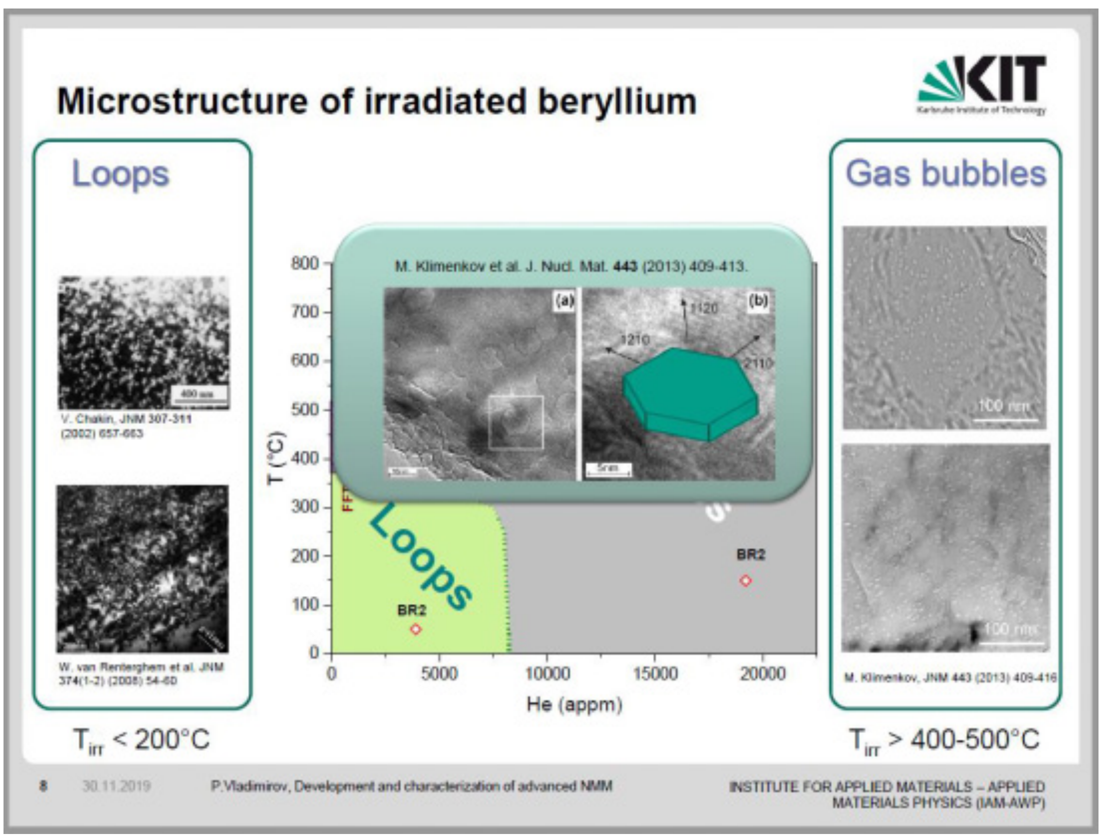
Microstructure of irradiated beryllium
KIT
Loops
V. Chakin, JNM 307-311 (2002) 657-663
W. van Renterghem et al. JNM 374(1-2) (2008) 54-60
M. Klimenkov et al. J. Nucl. Mat. 443 (2013) 409-413.
(a)
(b)
1120
1210
2110
5nm
T (°C)
He (appm)
Loops
BR2
BR2
Gas bubbles
100 nm
100 nm
M. Klimenkov, JNM 443 (2013) 409-416
T_{irr} < 200°C
T_{irr} > 400-500°C
8
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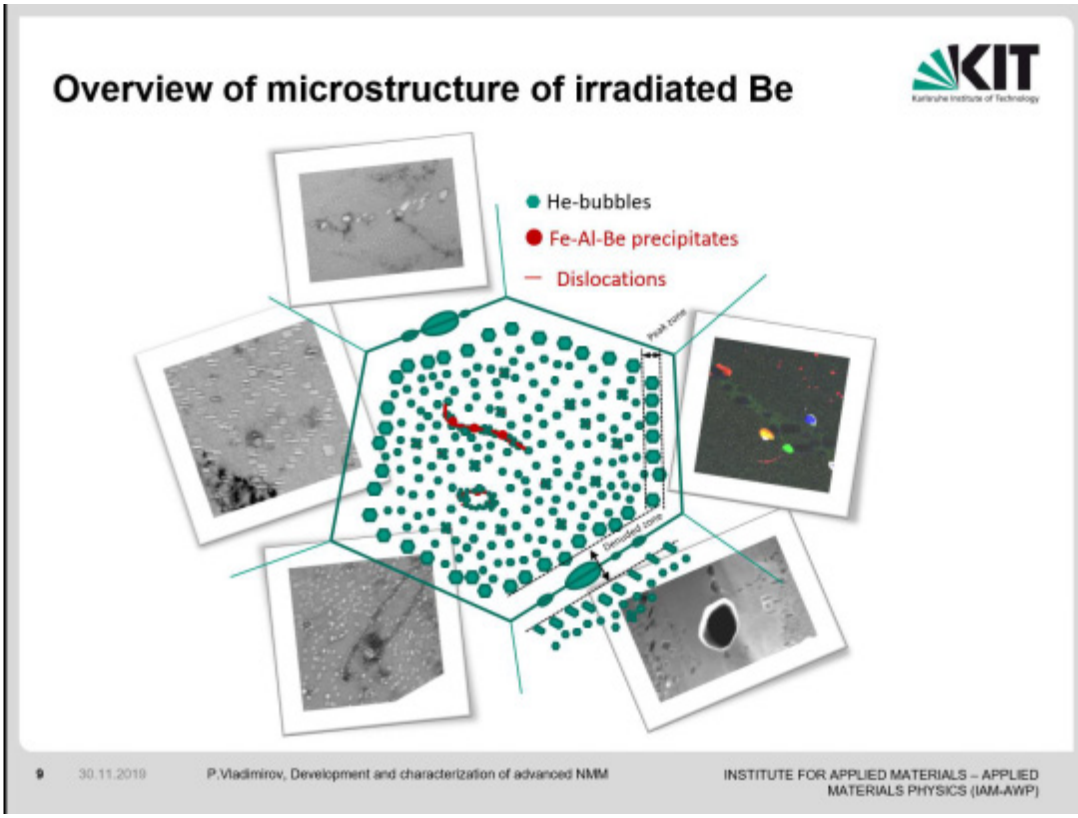
Overview of microstructure of irradiated Be
KIT
Karlsruhe Institute of Technology
He-bubbles
Fe-Al-Be precipitates
Dislocations
Peak zone
Denuded zone
9
30.11.2019
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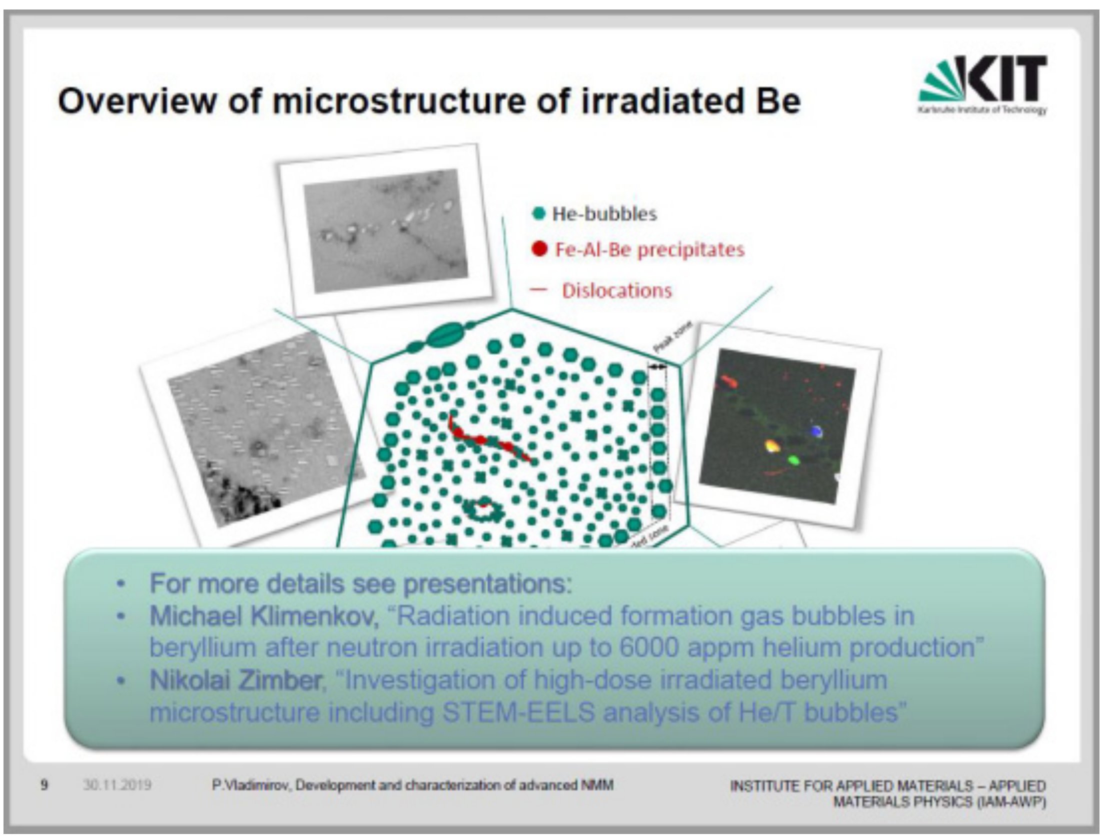
Overview of microstructure of irradiated Be
KIT
Karlsruhe Institute of Technology
He-bubbles
Fe-Al-Be precipitates
Dislocations
Peak zone
• For more details see presentations:
• Michael Klimenkov, "Radiation induced formation gas bubbles in beryllium after neutron irradiation up to 6000 appm helium production"
• Nikolai Zimber, "Investigation of high-dose irradiated beryllium microstructure including STEM-EELS analysis of He/T bubbles"
9
30.11.2019
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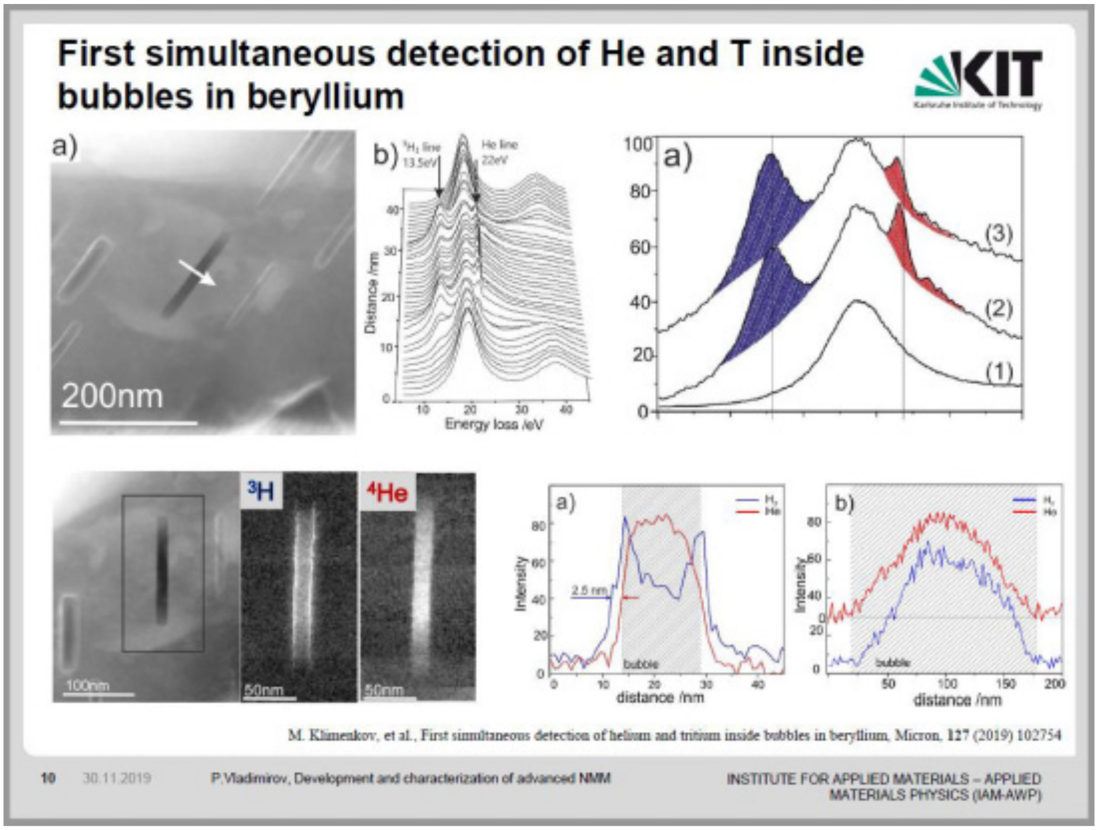
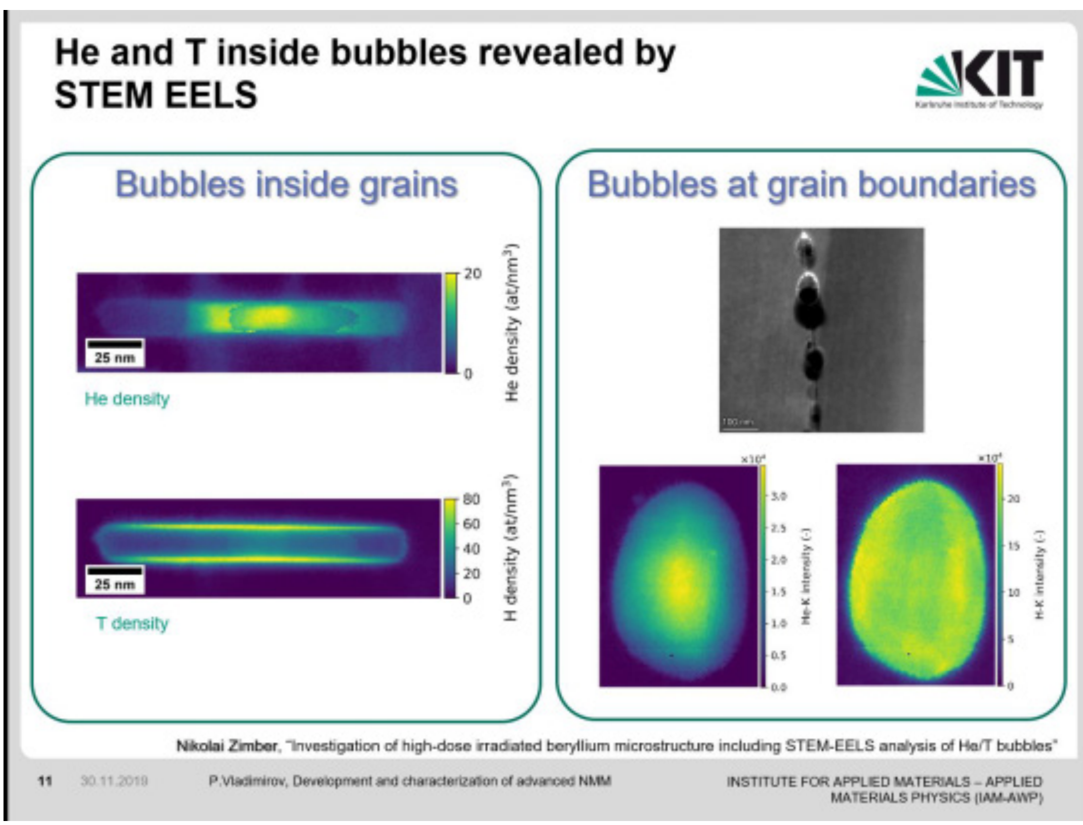
INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)

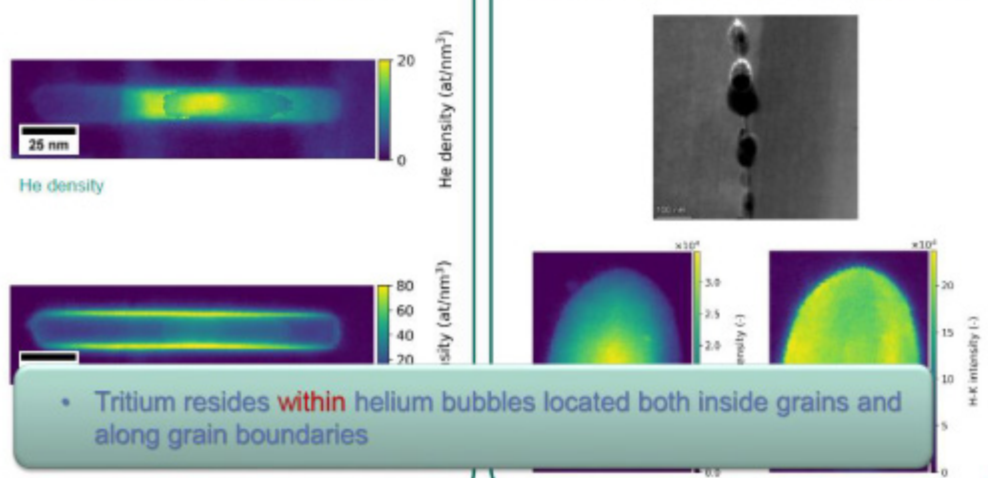

First simultaneous detection of He and T inside bubbles in beryllium
KIT
a)
200nm
b)
Energy loss /eV
Distance /nm
a)
(3)
(2)
(1)
³H
⁴He
100nm
50nm
50nm
a)
b)
Intensity
distance /nm
bubble
M. Klimenkov, et al., First simultaneous detection of helium and tritium inside bubbles in beryllium, Micron, 127 (2019) 102754
10 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM
INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)


He and T inside bubbles revealed by STEM EELS
KIT
Bubbles inside grains
Bubbles at grain boundaries
25 nm
He density
He density (at/nm³)
25 nm
T density
H density (at/nm³)
He-K intensity (-)
H-K intensity (-)
Nikolai Zimber, "Investigation of high-dose irradiated beryllium microstructure including STEM-EELS analysis of He/T bubbles"
11 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM
INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)

He and T inside bubbles revealed by STEM EELS

KIT Karlsruhe Institute of Technology

Bubbles inside grains | **Bubbles at grain boundaries**



- Tritium resides within helium bubbles located both inside grains and along grain boundaries

Nikolai Zimber, "Investigation of high-dose irradiated beryllium microstructure including STEM-EELS analysis of He/T bubbles"

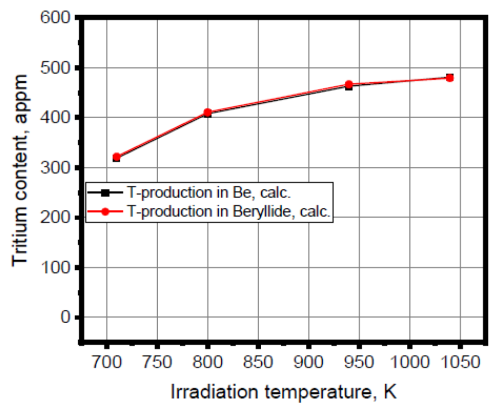
11 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)

T-production in beryllium

KIT Karlsruhe Institute of Technology

- Higher irradiation temperatures are reached near the reactor core, where n-flux is also higher. Tritium production follows the same trend.
- T-production is estimated based on the calculated and verified n-fluxes and evaluated neutron scattering x-sections, i.e. **has some uncertainty.**

Irradiation Temperature				
T_{irr}, K	710	800	940	1040
T_{irr}, °C	437	527	667	767



Vladimir Chakin, "Tritium release and retention behavior of beryllium and titanium beryllide irradiated up to high neutron doses"

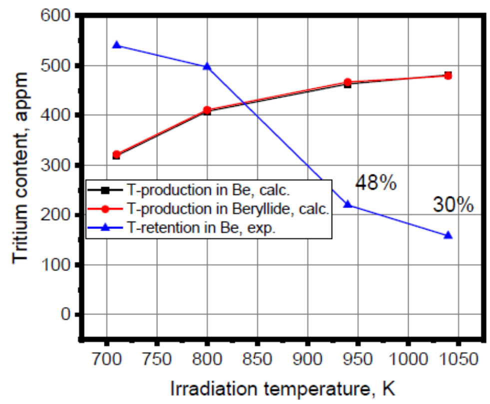
12 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)

T-retention in beryllium

KIT Karlsruhe Institute of Technology

- Actually measured T-retention at low irradiation temperatures exceeds calculated values.
- At two highest irradiation temperatures measured value is less than calculated, i.e. at such temperatures tritium escapes from Be already during irradiation.
- At T_{irr}=650°C, T-retention is more than 48% of T-production.
- Temperature profile in Be-pebble bed goes from less than 550°C near walls up to 650°C in the middle.

  ⇒ Excessive accumulation of T in Be is unavoidable



Vladimir Chakin, "Tritium release and retention behavior of beryllium and titanium beryllide irradiated up to high neutron doses"

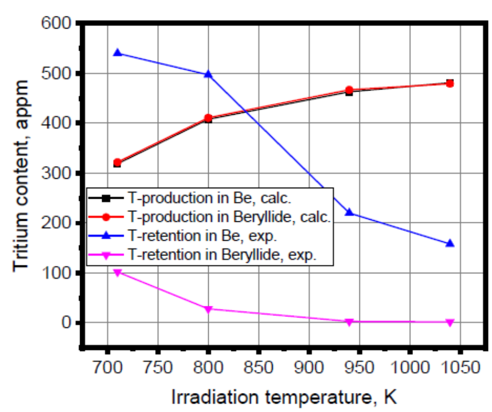
13 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)

T-retention in titanium beryllide

KIT Karlsruhe Institute of Technology

- For titanium beryllide T-retention drops down much quicker with T_{irr}.
- It is expected much lower overall T-inventory within blanket based on titanium beryllide.

⇒ Beryllides might be better alternative!



Vladimir Chakin, "Tritium release and retention behavior of beryllium and titanium beryllide irradiated up to high neutron doses"

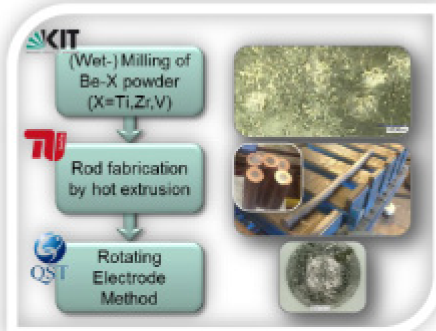
14 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)

Development of Advanced NM

- Advantages of beryllides
  - Higher melting points
  - Lower reactivity with air + steam
  - Lower tritium retention
  - Lower swelling under irradiation

- Intermetallic compounds on Be basis (e.g. $Be_{12}Ti$, $Be_{13}Zr$, $Be_{12}V$) are developed as advanced NM

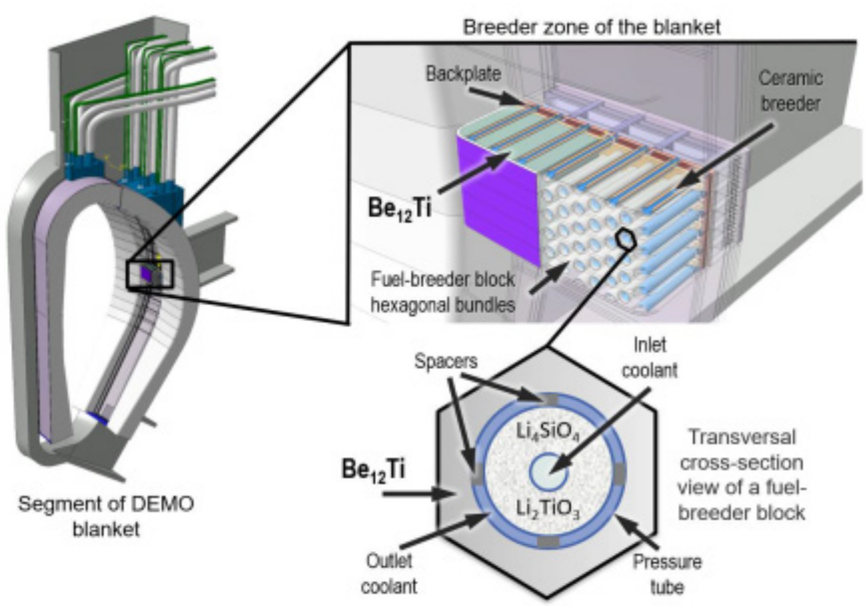
➢ Fabrication route developed at KIT
➢ Rod fabrication at TU Berlin
➢ Pilot production of $Be_{12}Ti$ pebbles at QST



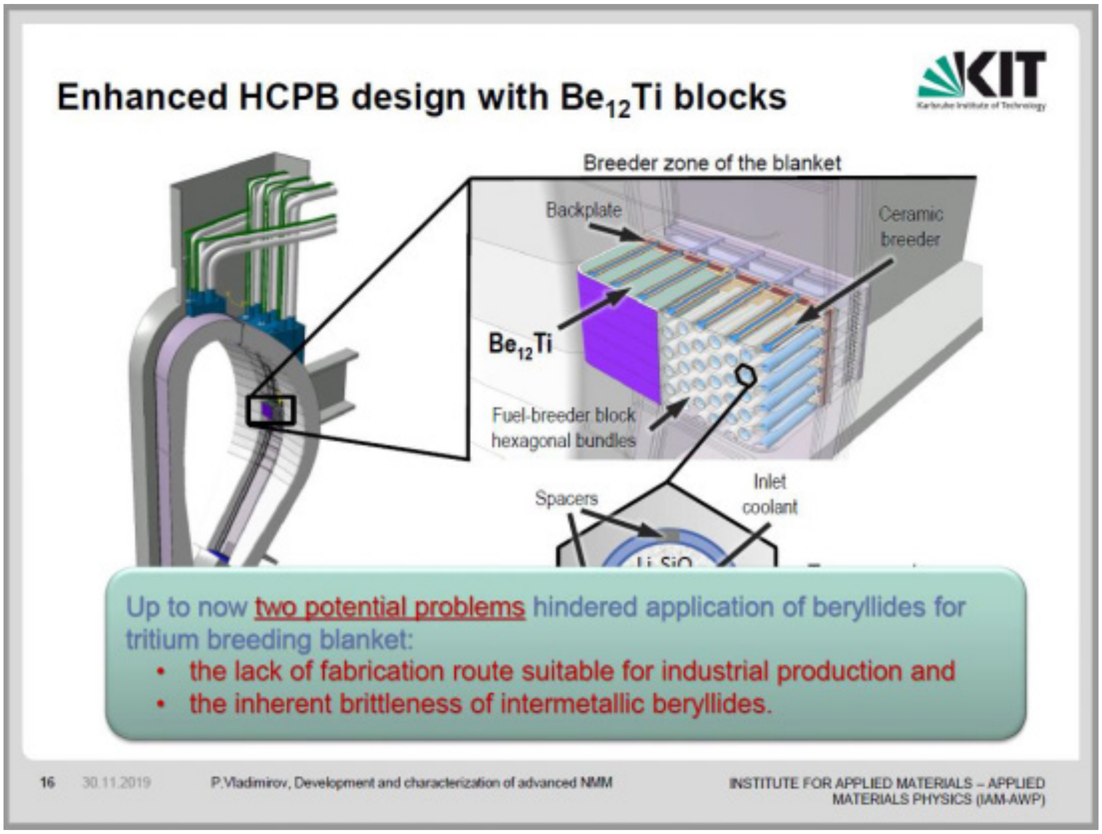
- Production of beryllides pebbles is possible for ITER, yet unrealistic for the quantities needed for DEMO

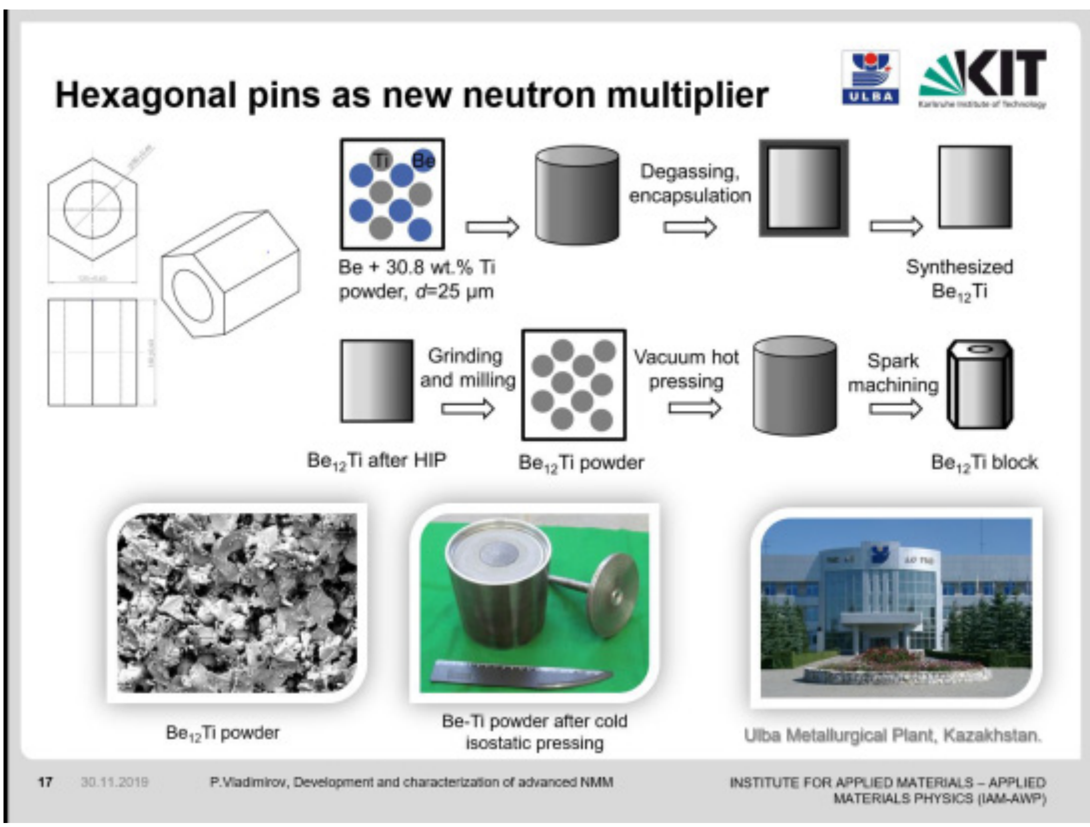
15 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)

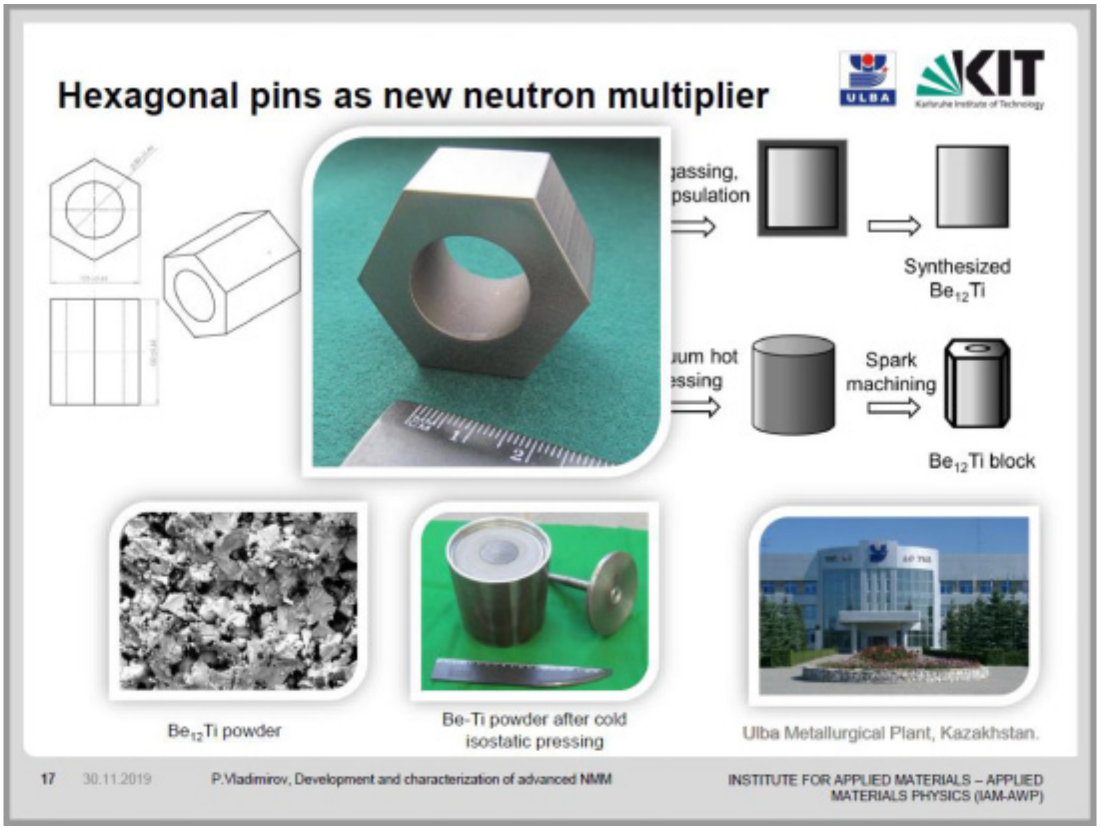
Enhanced HCPB design with $Be_{12}Ti$ blocks

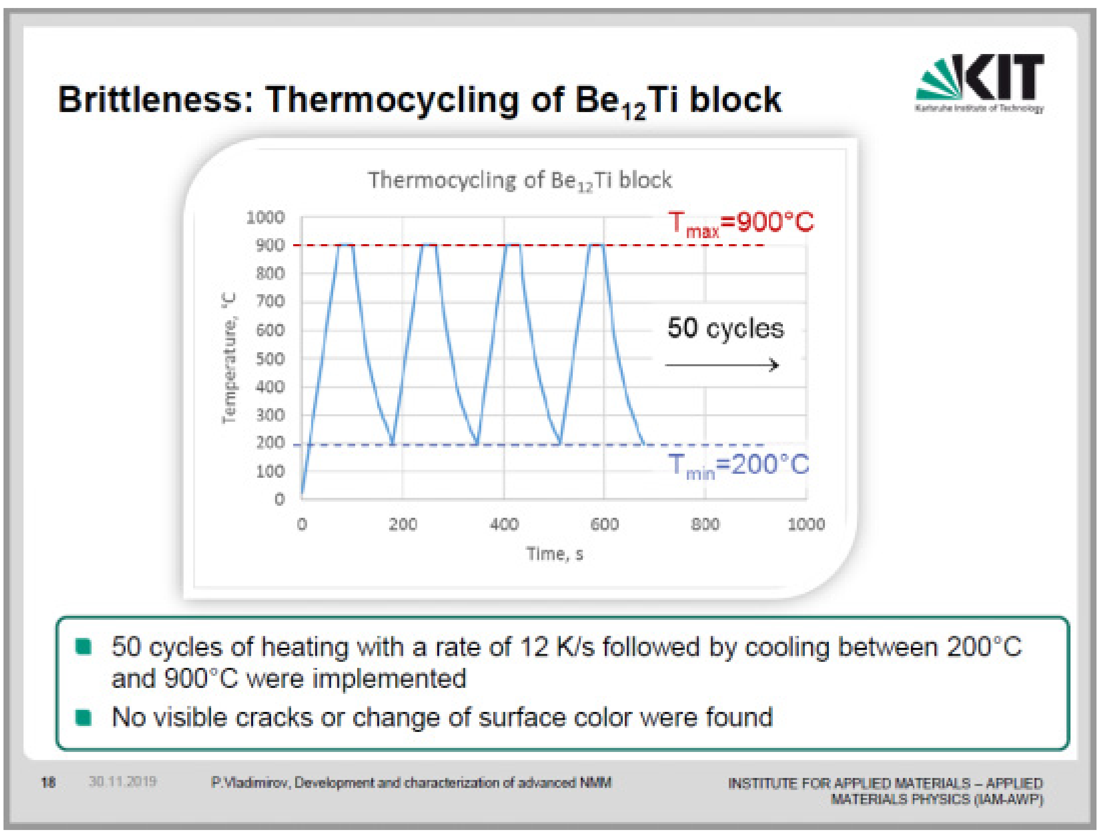


16 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)


Enhanced HCPB design with Be_{12}Ti blocks
KIT
Breeder zone of the blanket
Backplate
Ceramic breeder
Be_{12}Ti
Fuel-breeder block hexagonal bundles
Spacers
Inlet coolant
Up to now two potential problems hindered application of beryllides for tritium breeding blanket:
• the lack of fabrication route suitable for industrial production and
• the inherent brittleness of intermetallic beryllides.
16 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM
INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)

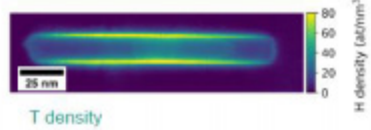

Hexagonal pins as new neutron multiplier
ULBA
KIT
Ti
Be
Be + 30.8 wt.% Ti powder, d=25 µm
Degassing, encapsulation
Synthesized Be_{12}Ti
Grinding and milling
Vacuum hot pressing
Spark machining
Be_{12}Ti after HIP
Be_{12}Ti powder
Be_{12}Ti block
Be_{12}Ti powder
Be-Ti powder after cold isostatic pressing
Ulba Metallurgical Plant, Kazakhstan.
17 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM
INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)


Hexagonal pins as new neutron multiplier
ULBA
KIT
Karlsruhe Institute of Technology
jassing,
psulation
Synthesized
Be12Ti
ıum hot
essing
Spark
machining
Be12Ti block
Be12Ti powder
Be-Ti powder after cold
isostatic pressing
Ulba Metallurgical Plant, Kazakhstan.
17 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM
INSTITUTE FOR APPLIED MATERIALS – APPLIED
MATERIALS PHYSICS (IAM-AWP)

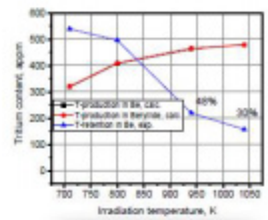

Brittleness: Thermocycling of Be12Ti block
KIT
Karlsruhe Institute of Technology
Thermocycling of Be12Ti block
Tmax=900°C
50 cycles
Tmin=200°C
Temperature, °C
Time, s
0
100
200
300
400
500
600
700
800
900
1000
0
200
400
600
800
1000
50 cycles of heating with a rate of 12 K/s followed by cooling between 200°C and 900°C were implemented
No visible cracks or change of surface color were found
18 30.11.2019 P.Vladimirov, Development and characterization of advanced NMM
INSTITUTE FOR APPLIED MATERIALS – APPLIED
MATERIALS PHYSICS (IAM-AWP)

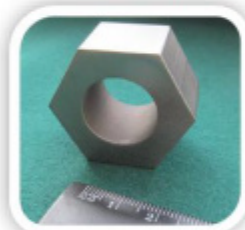
Conclusions & outlook

- T trapped inside He-bubbles in Be
- More that 40% of T is accumulated in Be
- New HCPB blanket design uses hexagonal $Be_{12}Ti$ blocks as neutron multiplier
- Industrial scale fabrication of solid blocks from $Be_{12}Ti$ is developed in cooperation with UMP, Kazakhstan
- Fabricated blocks are sufficiently strong to survive heating-cooling cycling
- Other beryllides ($Be_{12}V$, $Be_{13}Zr$, etc.) should be tested for fabrication of solid blocks as well



T density





R&Ds on Vanadium Beryllides as an advanced neutron multiplier for DEMO Fusion applications

Jae-Hwan Kim, Petr Kurinskiy, and Masaru Nakamichi

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National Institutes for Quantum and Radiological Science and Technology,
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Beryllium intermetallic compounds (beryllides) such as $Be_{12}Ti$, $Be_{12}V$, and $Be_{13}Zr$ are the most promising advanced neutron multipliers for demonstration (DEMO) fusion reactors owing to low reactivity with water vapor and low swelling at elevated temperatures.

By a novel process combined with a plasma sintering and a rotating electrode process for a rod fabrication and granulation, respectively, we have successfully fabricated the beryllides pebbles with 1mm in diameter for the first time in the world and attempted the optimization of the granulation conditions to control the pebble size and improve the granulation yield.

This presentation aimed to clarify the superiority of $Be_{12}V$ pebbles from viewpoints of easiness of fabrication process, excellent properties, low reactivity, low hydrogen retention, and verify the pebble packing fraction as well as the thermal conductivity for the pebble bed filled with $Be_{12}V$ pebbles.

It was concluded that $Be_{12}V$ pebbles with single phase were successfully fabricated by two processes, a plasma sintering and rotating electrode process with high granulation yield (including improvement of the yield by a reuse process), which were advantageous to mass production while the $Be_{12}V$ pebbles indicated much lower reactivity with H_2O as well as lower hydrogen isotope retention. Furthermore, the packing fractions of single-sized pebble with either 0.4 (0.3-0.5) or 2.5 (2.36-2.80) mm and binary-size $Be_{12}V$ pebbles with 0.4 (0.3-0.5) and 2.5 (2.36-2.80) mm in diameter reached at 65% and 81%, respectively, which are target packing fractions for the blanket designing. In addition, the effective thermal conductivity of the single-size packing of $Be_{12}V$ pebble bed with 1 (0.85-1.18) mm in diameter found out to be lower by 33 % than that of Be pebble bed at 873K, which this value is within an allowable range for the blanket designing.

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JAPAN

The 14th International Workshop on Beryllium Technology,
Queen Mary Hotel, Long Beach, CA, USA, 24-25 Oct, 2019

R&Ds on Vanadium Beryllides as an advanced neutron multiplier for DEMO fusion applications

Outline

1. Necessity of advanced neutron multipliers
2. Synthesis of single-phased beryllides
3. Granulation of beryllide pebbles
4. Characterization of beryllide pebbles
5. Summary

Jae Hwan KIM, Masaru NAKAMICHI

National Institutes for Quantum and Radiological Science and Technology, QST

Jae-Hwan Kim | BeWS-14, CA, USA | 24th Oct. 2019 | page (1/14)

1. Necessity of advanced neutron multipliers
2. Synthesis of single-phased beryllides
3. Granulation of beryllide pebbles
4. Characterization of beryllide pebbles
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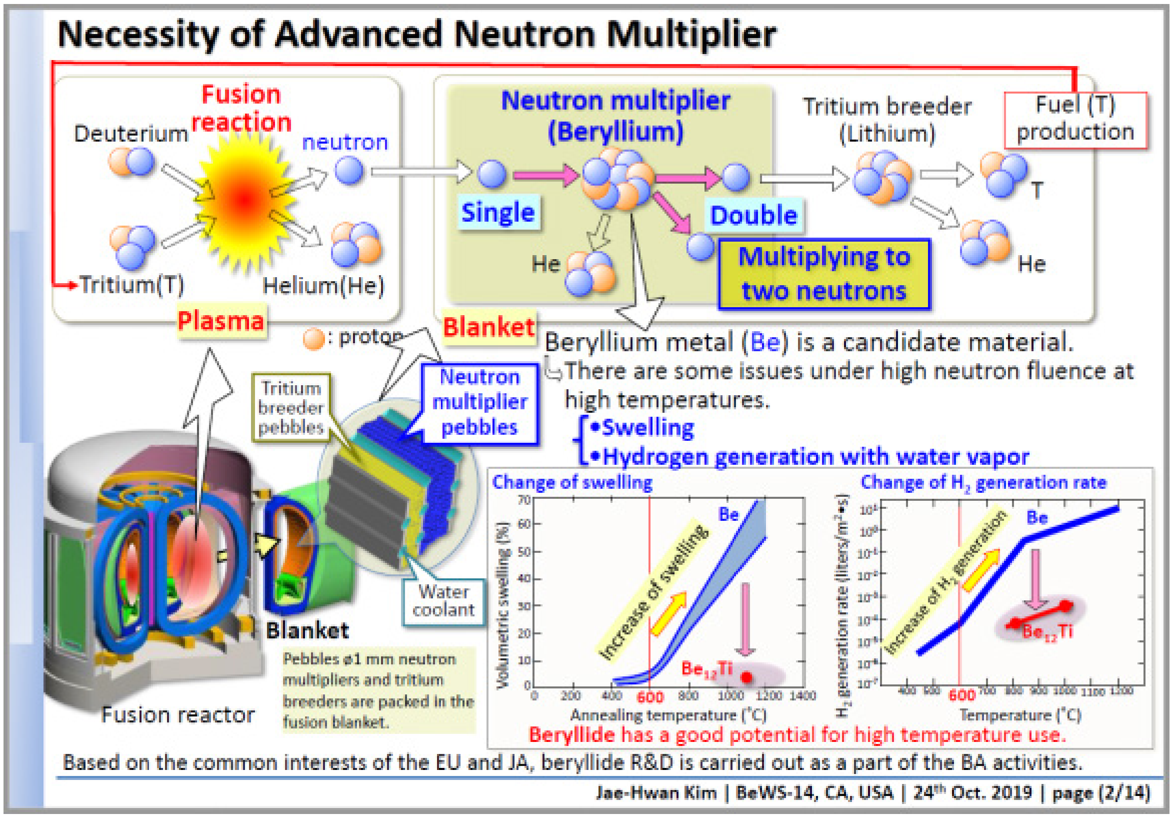
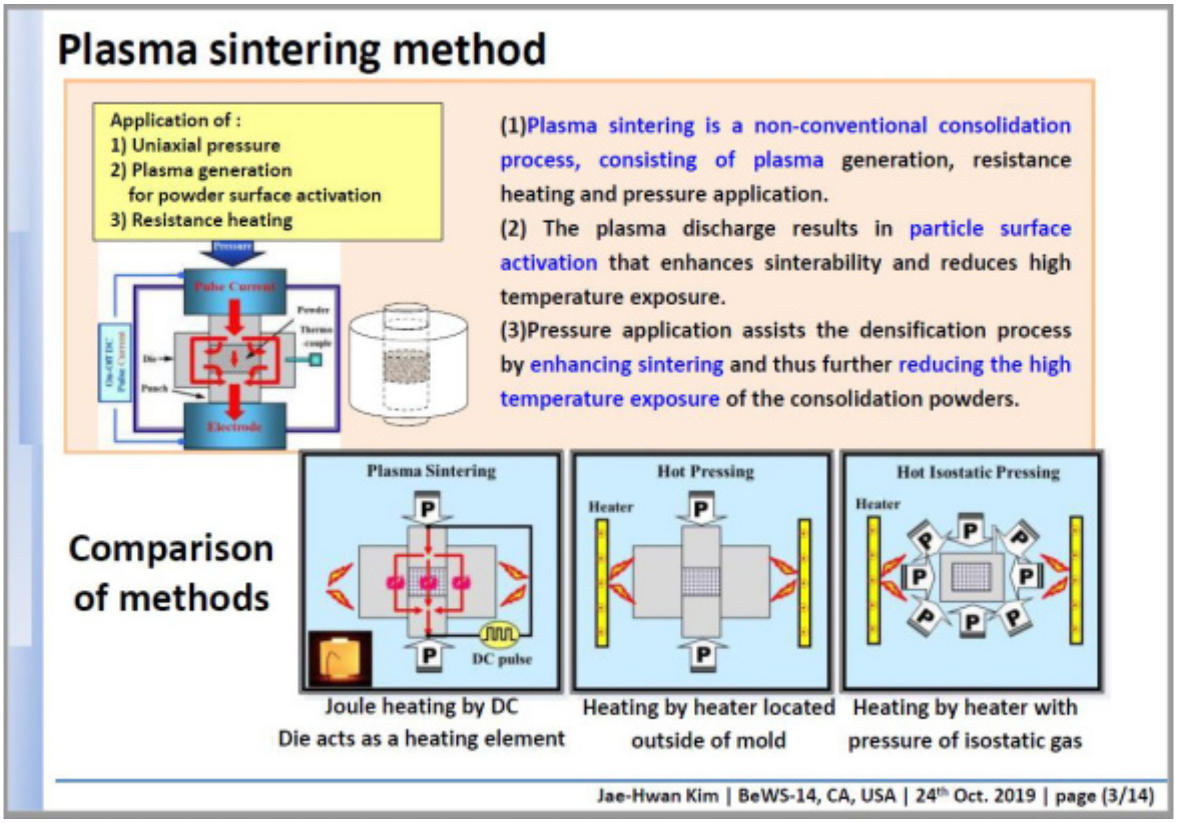
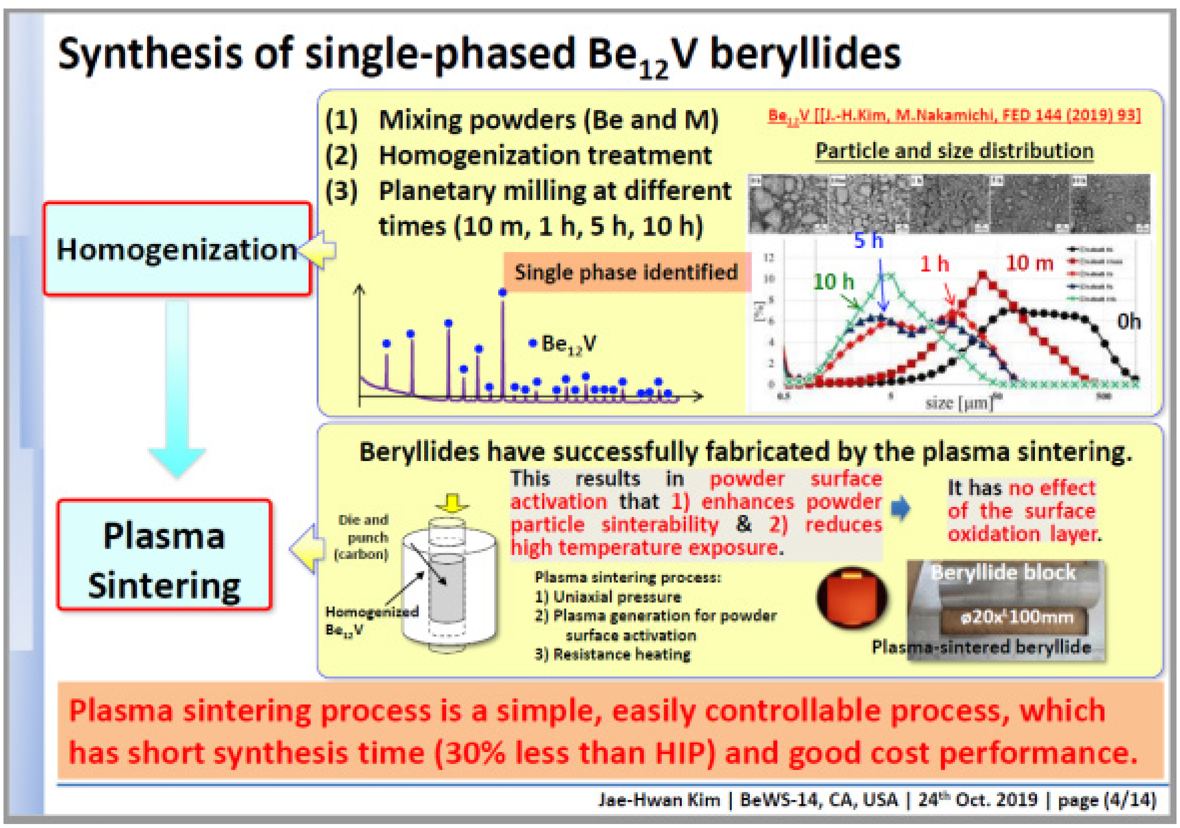
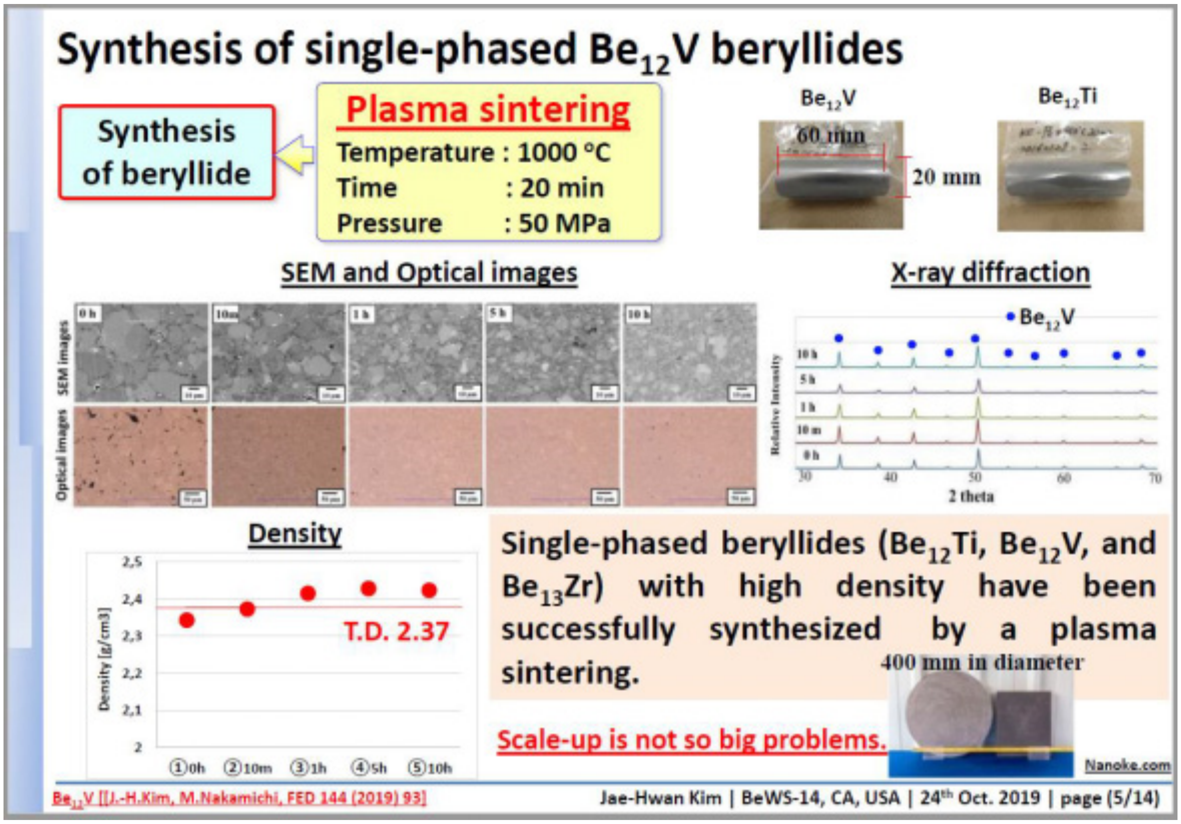


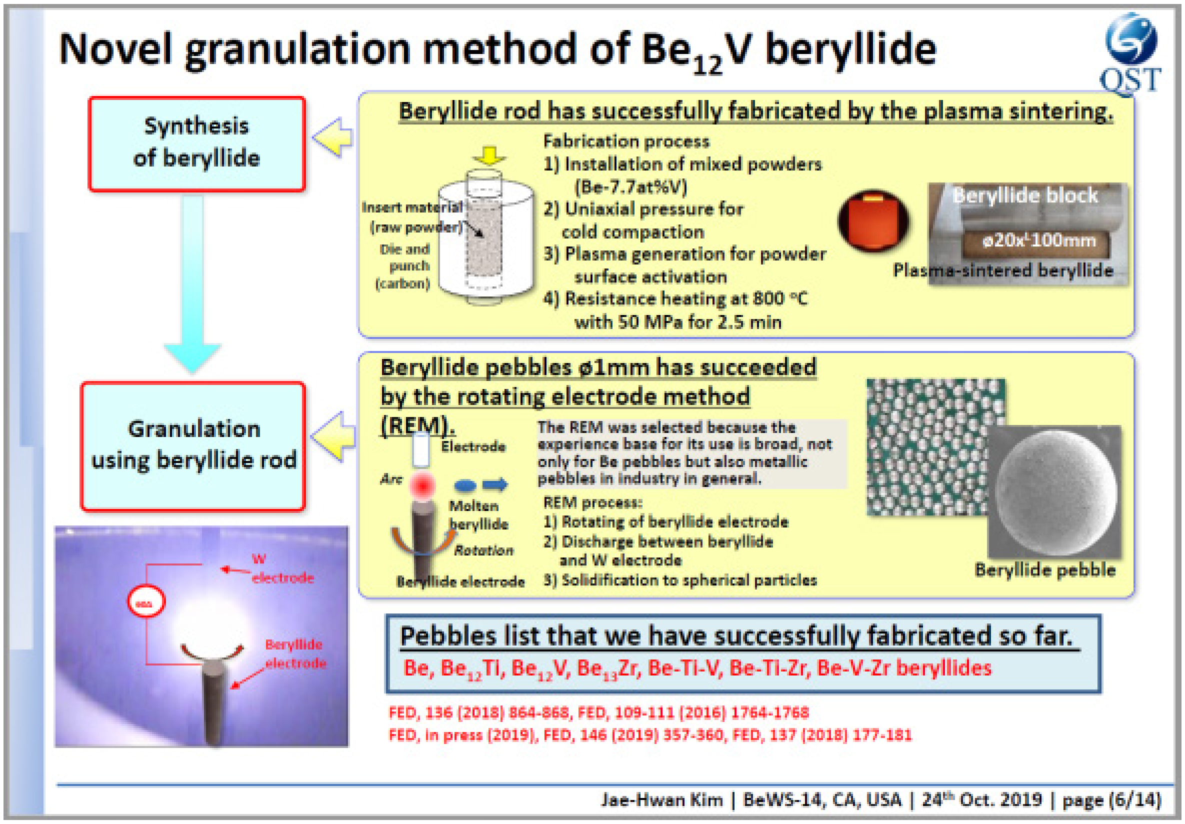
1. Necessity of advanced neutron multipliers
2. **Synthesis of single-phased beryllides**
3. Granulation of beryllide pebbles
4. Characterization of beryllide pebbles
5. Summary

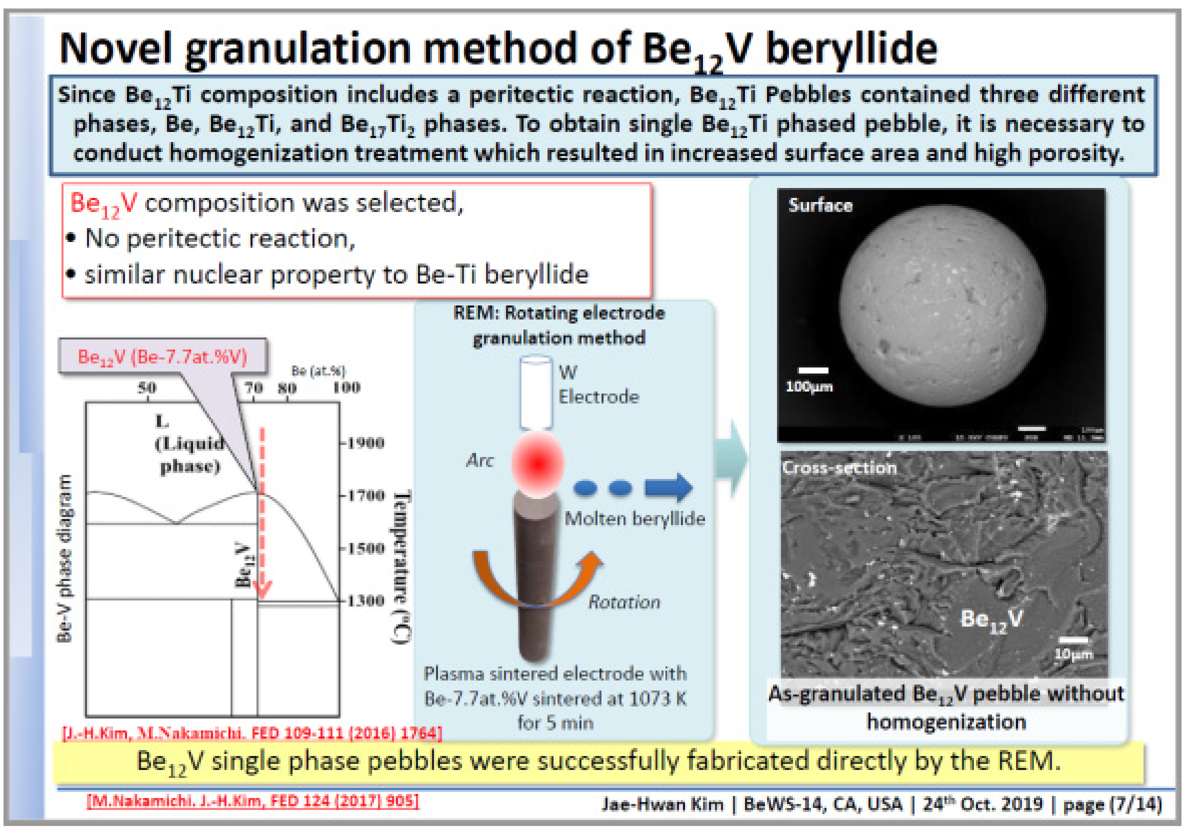

Plasma sintering method
Application of :
1) Uniaxial pressure
2) Plasma generation for powder surface activation
3) Resistance heating
(1)Plasma sintering is a non-conventional consolidation process, consisting of plasma generation, resistance heating and pressure application.
(2) The plasma discharge results in particle surface activation that enhances sinterability and reduces high temperature exposure.
(3)Pressure application assists the densification process by enhancing sintering and thus further reducing the high temperature exposure of the consolidation powders.
Comparison of methods
Plasma Sintering
Hot Pressing
Hot Isostatic Pressing
Heater
DC pulse
Joule heating by DC
Die acts as a heating element
Heating by heater located outside of mold
Heating by heater with pressure of isostatic gas
Jae-Hwan Kim | BeWS-14, CA, USA | 24th Oct. 2019 | page (3/14)

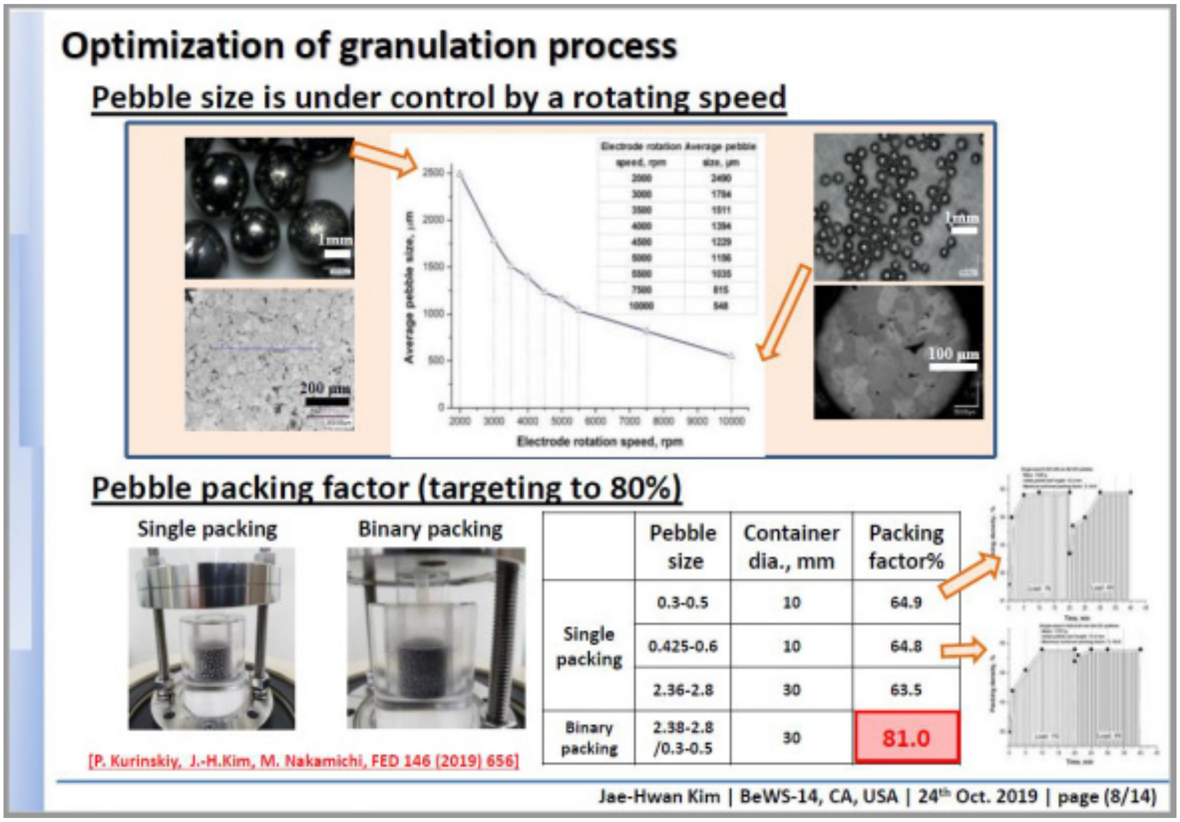

Synthesis of single-phased Be12V beryllides
(1) Mixing powders (Be and M)
(2) Homogenization treatment
(3) Planetary milling at different times (10 m, 1 h, 5 h, 10 h)
Be12V [[J.-H.Kim, M.Nakamichi, FED 144 (2019) 93]
Particle and size distribution
Homogenization
Single phase identified
Be12V
5 h
10 h
1 h
10 m
0h
size [μm]
Beryllides have successfully fabricated by the plasma sintering.
This results in powder surface activation that 1) enhances powder particle sinterability & 2) reduces high temperature exposure.
It has no effect of the surface oxidation layer.
Die and punch (carbon)
Homogenized Be12V
Plasma Sintering
Plasma sintering process:
1) Uniaxial pressure
2) Plasma generation for powder surface activation
3) Resistance heating
Beryllide block
ø20x100mm
Plasma-sintered beryllide
Plasma sintering process is a simple, easily controllable process, which has short synthesis time (30% less than HIP) and good cost performance.
Jae-Hwan Kim | BeWS-14, CA, USA | 24th Oct. 2019 | page (4/14)

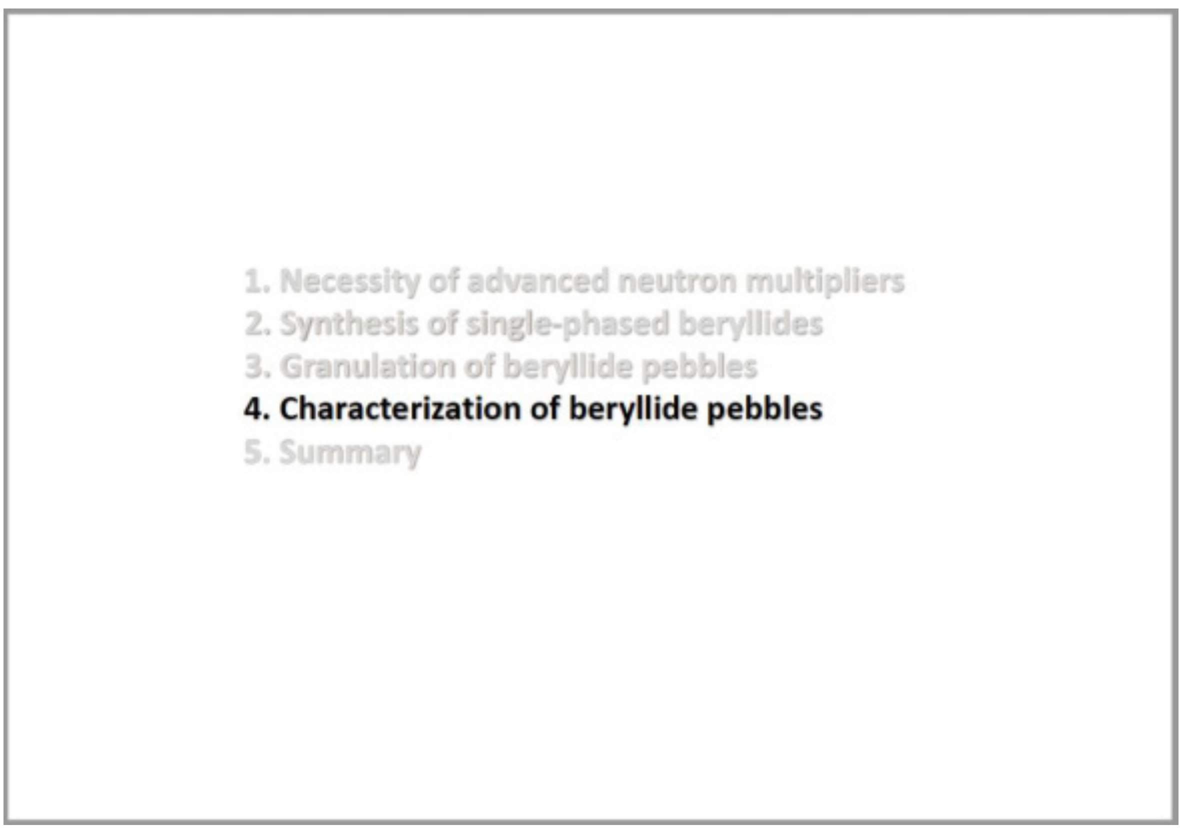


1. Necessity of advanced neutron multipliers
2. Synthesis of single-phased beryllides
3. **Granulation of beryllide pebbles**
4. Characterization of beryllide pebbles
5. Summary


Novel granulation method of $Be_{12}V$ beryllide
Synthesis of beryllide
Granulation using beryllide rod
Beryllide rod has successfully fabricated by the plasma sintering.
Fabrication process
1) Installation of mixed powders (Be-7.7at%V)
2) Uniaxial pressure for cold compaction
3) Plasma generation for powder surface activation
4) Resistance heating at 800 °C with 50 MPa for 2.5 min
Insert material (raw powder)
Die and punch (carbon)
Beryllide block
ø20x^{t}100mm
Plasma-sintered beryllide
Beryllide pebbles ø1mm has succeeded by the rotating electrode method (REM).
The REM was selected because the experience base for its use is broad, not only for Be pebbles but also metallic pebbles in industry in general.
REM process:
1) Rotating of beryllide electrode
2) Discharge between beryllide and W electrode
3) Solidification to spherical particles
Electrode
Arc
Molten beryllide
Rotation
Beryllide electrode
Beryllide pebble
W electrode
Beryllide electrode
Pebbles list that we have successfully fabricated so far.
Be, $Be_{12}Ti$, $Be_{12}V$, $Be_{13}Zr$, Be-Ti-V, Be-Ti-Zr, Be-V-Zr beryllides
FED, 136 (2018) 864-868, FED, 109-111 (2016) 1764-1768
FED, in press (2019), FED, 146 (2019) 357-360, FED, 137 (2018) 177-181
Jae-Hwan Kim | BeWS-14, CA, USA | 24th Oct. 2019 | page (6/14)


Novel granulation method of $Be_{12}V$ beryllide
Since $Be_{12}Ti$ composition includes a peritectic reaction, $Be_{12}Ti$ Pebbles contained three different phases, Be, $Be_{12}Ti$, and $Be_{17}Ti_2$ phases. To obtain single $Be_{12}Ti$ phased pebble, it is necessary to conduct homogenization treatment which resulted in increased surface area and high porosity.
$Be_{12}V$ composition was selected,
• No peritectic reaction,
• similar nuclear property to Be-Ti beryllide
$Be_{12}V$ (Be-7.7at.%V)
Be (at.%)
50
70
80
100
L (Liquid phase)
1900
1700
1500
1300
Temperature (°C)
$Be_{12}V$
Be-V phase diagram
[J.-H.Kim, M.Nakamichi. FED 109-111 (2016) 1764]
REM: Rotating electrode granulation method
W Electrode
Arc
Molten beryllide
Rotation
Plasma sintered electrode with Be-7.7at.%V sintered at 1073 K for 5 min
Surface
100μm
Cross-section
$Be_{12}V$
10μm
As-granulated $Be_{12}V$ pebble without homogenization
$Be_{12}V$ single phase pebbles were successfully fabricated directly by the REM.
[M.Nakamichi. J.-H.Kim, FED 124 (2017) 905]
Jae-Hwan Kim | BeWS-14, CA, USA | 24th Oct. 2019 | page (7/14)


Optimization of granulation process
Pebble size is under control by a rotating speed
1mm
200 μm
Electrode rotation speed, rpm
Average pebble size, μm
Electrode rotation speed, rpm
Average pebble size, μm
2000
2490
3000
1784
3500
1511
4000
1394
4500
1229
5000
1106
5500
1035
7500
815
10000
548
1mm
100 μm
Pebble packing factor (targeting to 80%)
Single packing
Binary packing
Pebble size
Container dia., mm
Packing factor%
Single packing
0.3-0.5
10
64.9
0.425-0.6
10
64.8
2.36-2.8
30
63.5
Binary packing
2.38-2.8 /0.3-0.5
30
81.0
[P. Kurinskiy, J.-H.Kim, M. Nakamichi, FED 146 (2019) 656]
Jae-Hwan Kim | BeWS-14, CA, USA | 24th Oct. 2019 | page (8/14)


1. Necessity of advanced neutron multipliers
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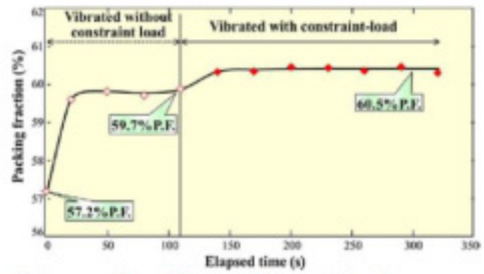
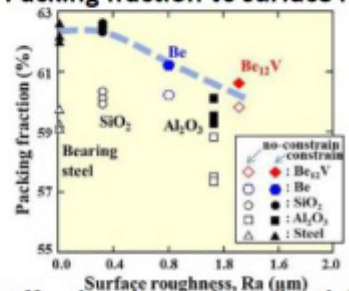
5. Summary

Thermal conductivity of Be and $Be_{12}V$ pebble beds

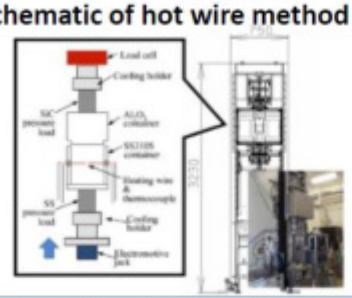
Packing fraction variation by vibration



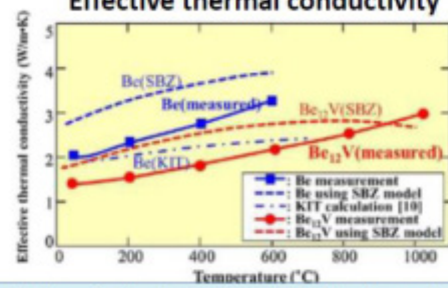
Packing fraction vs surface roughness



Schematic of hot wire method



Effective thermal conductivity



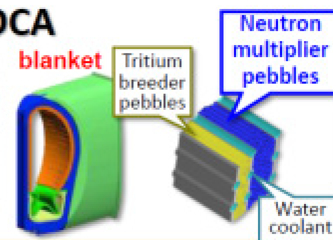
It was clear that the effective thermal conductivity of the $Be_{12}V$ pebble bed was lower by approximately 30 % than that of Be pebble bed at 873 K.

[M. Nakamichi, J.-H.Kim, P. Kurinskiy, FED 136 (2018) 125-127]

Jae-Hwan Kim | BeWS-14, CA, USA | 24th Oct. 2019 | page (9/14)

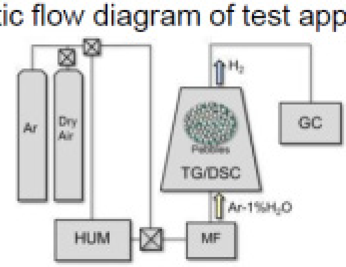
Critical issues on H_2 generation reaction in LOCA

Japan has adopted water coolant solid breeder blanket concept for ITER and DEMO. Assuming Loss Of Coolant Accident in the blanket, H_2 generation behavior of neutron multiplier (beryllium and beryllides) should be clarified.



H_2 generation reaction of beryllides pebbles under Ar with the addition of H_2O (1~15%) were examined using a Thermo-Gravimetry/Differential Scanning Calorimetry apparatus connected with a gas chromatography.

Schematic flow diagram of test apparatus

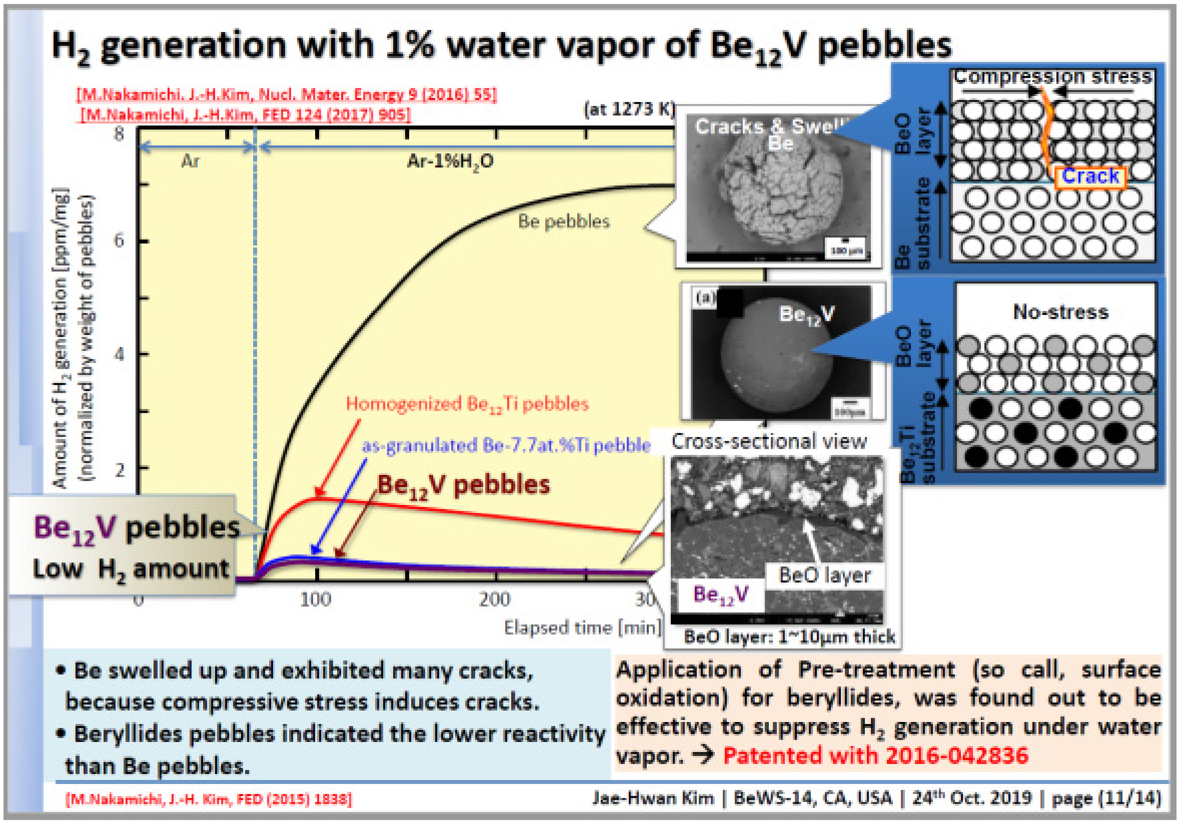


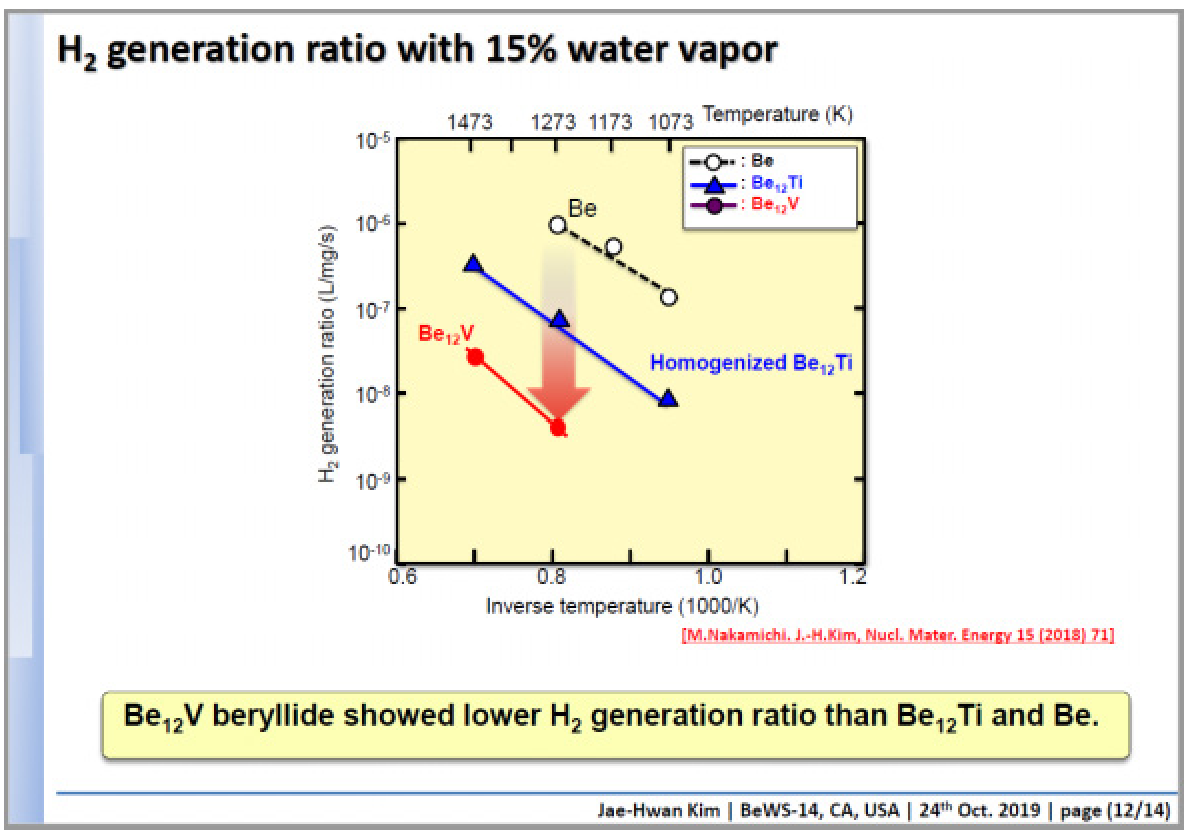
DSC : Differential Scanning Calorimetry apparatus
GC : Gas Chromatographic apparatus
HUM: Water vapor generation apparatus
MF : Mass flow controller

Conditions

- Apparatus :
  - DSC : STA-499, Netzsch, Japan.
  - GC : CP-490, Agilent, USA.
  - HUM : HC-9800, Netzsch, Japan.
- Heating rate : 10 K/min
- Atmosphere : Ar with 1~15% H_2O
- Temperature: ~1473 K
  (Ar gas flow during temperature ramping)

Jae-Hwan Kim | BeWS-14, CA, USA | 24th Oct. 2019 | page (10/14)


H2 generation with 1% water vapor of Be12V pebbles
[M.Nakamichi, J.-H.Kim, Nucl. Mater. Energy 9 (2016) 55]
[M.Nakamichi, J.-H.Kim, FED 124 (2017) 905]
(at 1273 K)
Ar
Ar-1%H2O
Be pebbles
Homogenized Be12Ti pebbles
as-granulated Be-7.7at.%Ti pebble
Be12V pebbles
Amount of H2 generation [ppm/mg] (normalized by weight of pebbles)
Elapsed time [min]
Be12V pebbles
Low H2 amount
Cracks & Swell
Be
Be12V
Cross-sectional view
BeO layer
Be12V
BeO layer: 1~10μm thick
Compression stress
Crack
BeO layer
Be substrate
No-stress
BeO layer
Be12Ti substrate
• Be swelled up and exhibited many cracks, because compressive stress induces cracks.
• Beryllides pebbles indicated the lower reactivity than Be pebbles.
Application of Pre-treatment (so call, surface oxidation) for beryllides, was found out to be effective to suppress H2 generation under water vapor. → Patented with 2016-042836
[M.Nakamichi, J.-H. Kim, FED (2015) 1838]
Jae-Hwan Kim | BeWS-14, CA, USA | 24th Oct. 2019 | page (11/14)


H2 generation ratio with 15% water vapor
Temperature (K)
1473
1273
1173
1073
: Be
: Be12Ti
: Be12V
Be
Be12V
Homogenized Be12Ti
H2 generation ratio (L/mg/s)
Inverse temperature (1000/K)
[M.Nakamichi, J.-H.Kim, Nucl. Mater. Energy 15 (2018) 71]
Be12V beryllide showed lower H2 generation ratio than Be12Ti and Be.
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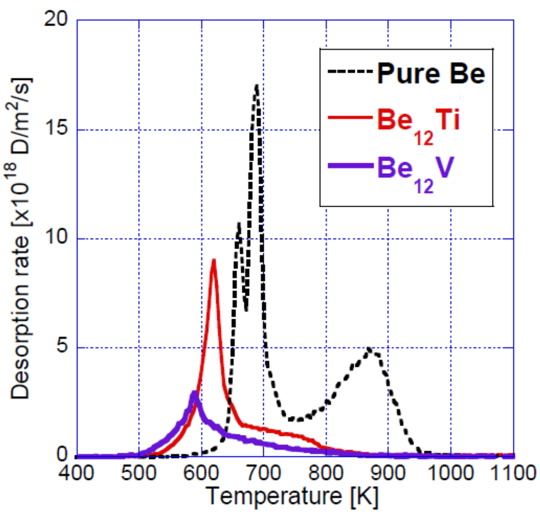
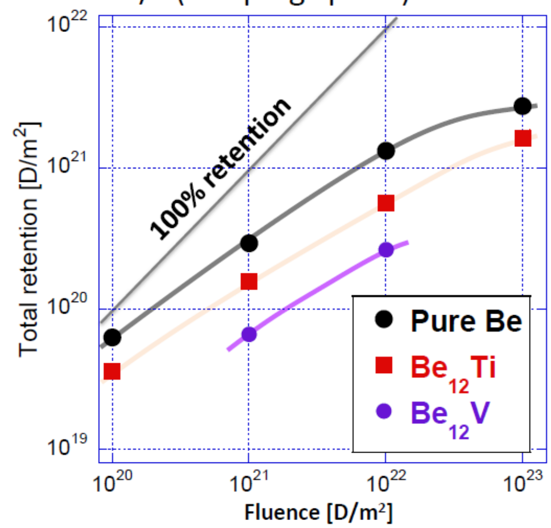
Deuterium Desorption and retention properties

- 3keV-D_2^+, 1×10^{22} D/m^2, R.T. and TDS with 1 K/s (ramping speed)





[M.Nakamichi, J.-H.Kim, Nucl. Mater. Energy 9 (2016) 55]

Due to the small desorption from beryllides, the total retentions of deuterium in beryllides were evaluated to be ~50% in $Be_{12}Ti$ and ~20% in $Be_{12}V$ of that in pure Be.

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Summary

- Single phased $Be_{12}V$ beryllide blocks and pebbles were successfully fabricated directly either by a plasma sintering and by the rotating electrode granulation method (REM) using the plasma-sintered beryllides electrodes, respectively.
- Optimization of granulation for $Be_{12}V$ pebbles led to being able to fabricate not only small (0.5mm) but also big (2.5mm) pebbles and binary packing fraction reached into over 80 %.
- Beryllides ($Be_{12}V$) had much lower H_2 generation ratio under H_2O than Be and lower D retention than Be.
- A new neutron irradiation campaign will be performed for newly developed beryllides (for instance, single phase $Be_{12}V$, ternary beryllides etc.) to verify superiority of swelling and tritium retention over Be.

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Beryllium and its Alloys as Neutron Multiplying Materials

MasaruNakamichi, Jae-HwanKim, Makoto M.Nakamura
National Institutes for Quantum and Radiological Science and Technology, Chiba, Japan

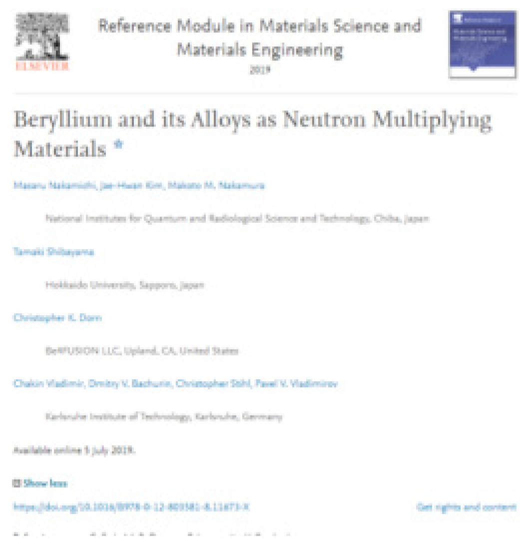
TamakiShibayama
Hokkaido University, Sapporo, Japan

Christopher K.Dorn
Be4FUSION LLC, Upland, CA, United States

Chakin Vladimir, Dmitry V.Bachurin, Christopher Stihl, Pavel V.Vladimirov
Karlsruhe Institute of Technology, Karlsruhe, Germany

Available online 5 July 2019.

Outline


Reference Module in Materials Science and Materials Engineering
2019

Beryllium and its Alloys as Neutron Multiplying Materials

Available online 5 July 2019.

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Acknowledgements

As a collaborative study within a framework of Broader Approach (BA) activities,
Publications,

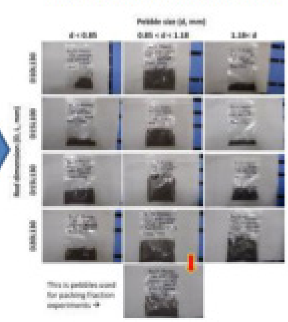
- Mechanical behavior of Be-Ti pebbles at blanket relevant temperatures, FED (2016)
- Effect of heat treatment of titanium beryllide on tritium/hydrogen release, FED (2018)
- Beryllium and its Alloys as Neutron Multiplying Materials, electronic book (2019)

And, also devoting to

- Pebble fabrication and shipping in QST using by the hot extruded beryllide rods (KIT)

KIT rod

Granulation at Rokkasho



Thank you for your kind attention

Microstructure and properties of intermetallic $Be_{12}Ti$ fabricated by arc melting or hot isostatic pressing

Ramil Gaisin[1], Vladimir Chakin[1], Aniceto Goraieb[2], and Pavel Vladimirov[1]

[1]Karlsruhe Institute of Technology (KIT), 76344 Eggenstein-Leopoldshafen, Germany
[2]Karlsruhe Beryllium Handling Facility (KBHF GmbH), 76344 Eggenstein-Leopoldshafen, Germany

Recently, Karlsruhe Institute of Technology has proposed an updated design of helium-cooled breeding blanket for the DEMO fusion reactor. In the new design, solid titanium beryllide blocks ($Be_{12}Ti$) are considered as neutron multiplier instead of 1mm beryllium pebbles. In comparison with pure beryllium, intermetallic titanium beryllide swell less, retain less tritium under irradiation and have higher heat and corrosion resistances. A key issue for titanium beryllide blocks is the lack of production technology on an industrial scale. Further advancement of the new breeding blanket design requires the development of a $Be_{12}Ti$ blocks production technology.

The work compares two different ways of $Be_{12}Ti$ manufacture: conventional casting and hot isostatic pressing (HIP). Conventional casting in laboratory argon arc-melting furnace resulted in composition shift towards titanium owing to extensive evaporation of beryllium. X-ray analysis showed the presence of other Be-Ti phases in the composition. The obtained ingot shows higher porosity and lower density. Because of these shortcomings, the casting of industrial-size titanium beryllide ingots seems impractical. Combination of hot extrusion for preliminary powder consolidation and HIP for further densification was chosen as an alternative way of $Be_{12}Ti$ fabrication following powder metallurgy route. Hot extruded rods produced from mixture of pure Be and Ti powders were subjected to HIP in 800-1200°C temperature range.

After HIP, a fine-grained titanium beryllide microstructure is formed with traces of beryllium, beryllium oxide and other titanium beryllide phases. Transmission electron microscopy suggests that BeO particles pin grain boundaries and suppress grain growth during HIP. $Be_{12}Ti$ phase obeys very high hardness of 1400-1500 HV, but rather low fracture toughness. Therefore, fast heating and cooling during HIP should be avoided to eliminate possible cracking of the workpiece. To avoid crack formation during finishing, electrical discharge machining was used for cutting and final machining of the surfaces. As a result, the $Be_{12}Ti$ mock-up of the neutron multiplier block was successfully manufactured. The presented method of a titanium beryllide mock-up manufacturing by combination of hot extrusion and HIP can be scaled up using larger extruded rods and industrial equipment. If necessary, the hot extrusion step can be replaced by other consolidation method (e.g. cold pressing).

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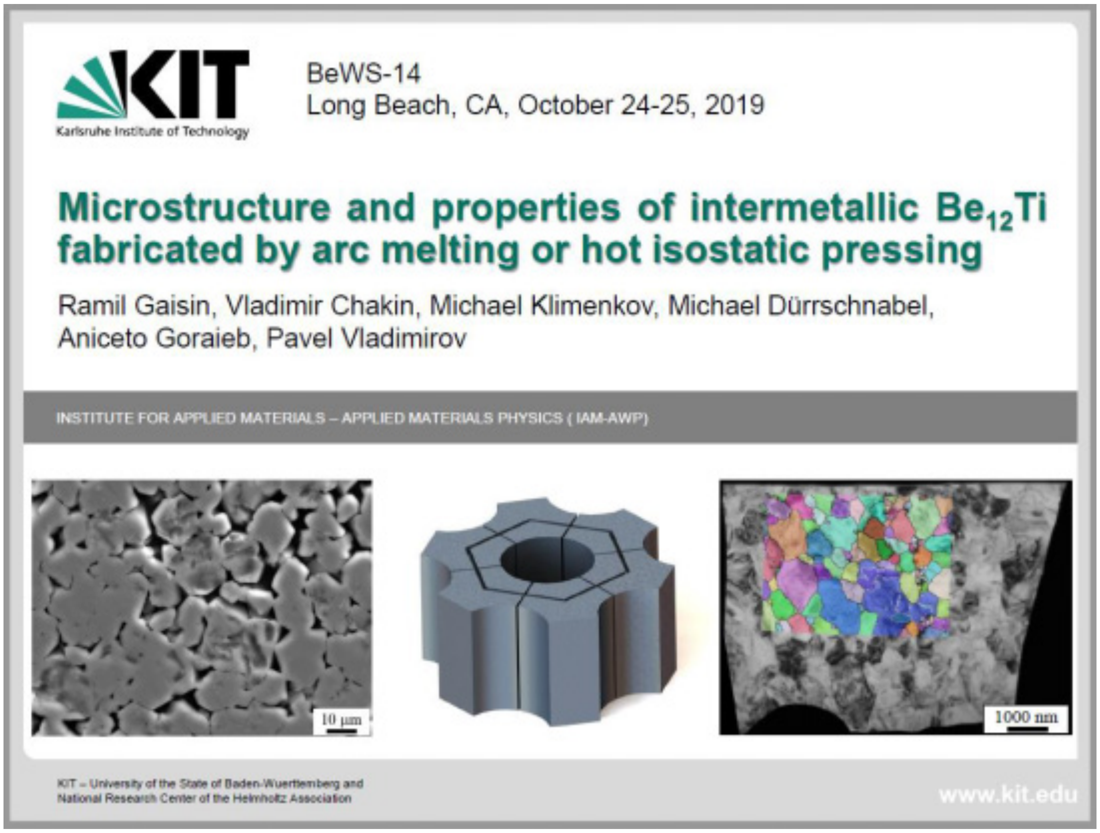
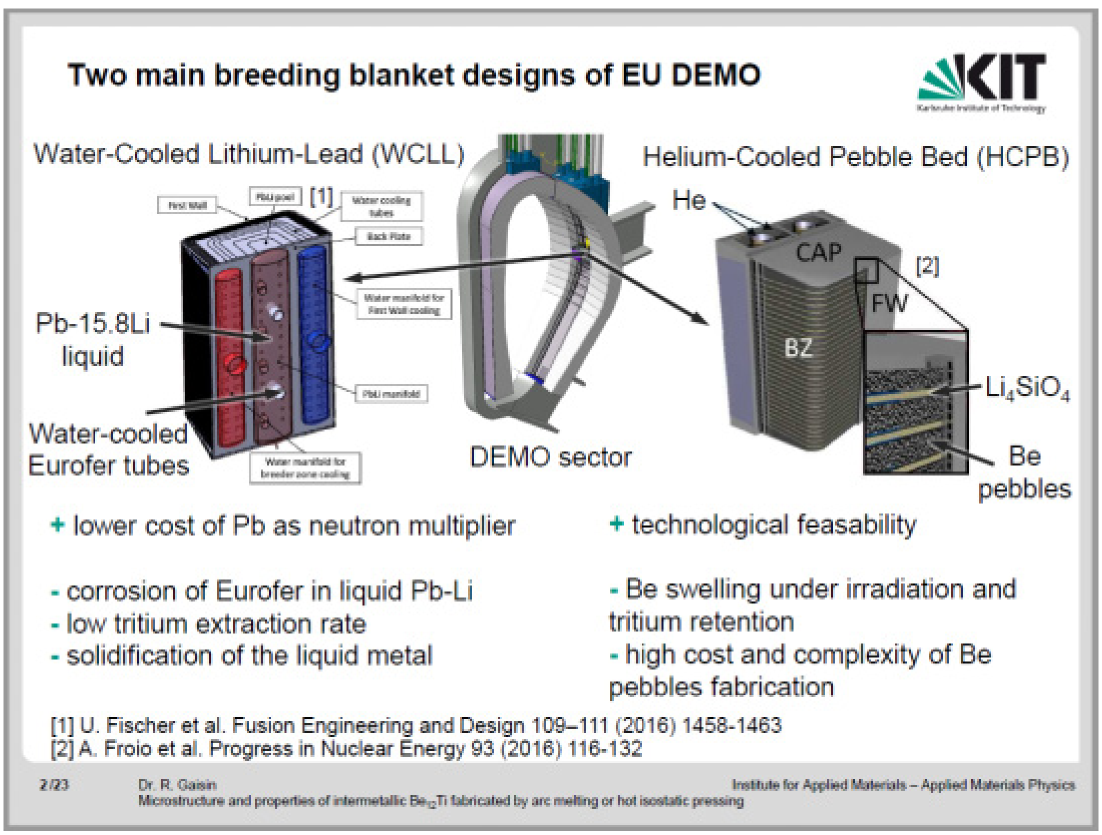

Karlsruhe Institute of Technology
BeWS-14
Long Beach, CA, October 24-25, 2019
Microstructure and properties of intermetallic $Be_{12}Ti$ fabricated by arc melting or hot isostatic pressing
Ramil Gaisin, Vladimir Chakin, Michael Klimenkov, Michael Dürrschnabel, Aniceto Goraieb, Pavel Vladimirov
INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-AWP)
10 µm
1000 nm
KIT – University of the State of Baden-Wuerttemberg and National Research Center of the Helmholtz Association
www.kit.edu


Two main breeding blanket designs of EU DEMO
Water-Cooled Lithium-Lead (WCLL)
First Wall
PbLi pool
[1]
Water cooling tubes
Back Plate
Water manifold for First Wall cooling
PbLi manifold
Water manifold for breeder zone cooling
Pb-15.8Li liquid
Water-cooled Eurofer tubes
DEMO sector
Helium-Cooled Pebble Bed (HCPB)
He
CAP
[2]
FW
BZ
Li_4SiO_4
Be pebbles
+ lower cost of Pb as neutron multiplier
- corrosion of Eurofer in liquid Pb-Li
- low tritium extraction rate
- solidification of the liquid metal
+ technological feasability
- Be swelling under irradiation and tritium retention
- high cost and complexity of Be pebbles fabrication
[1] U. Fischer et al. Fusion Engineering and Design 109–111 (2016) 1458-1463
[2] A. Froio et al. Progress in Nuclear Energy 93 (2016) 116-132
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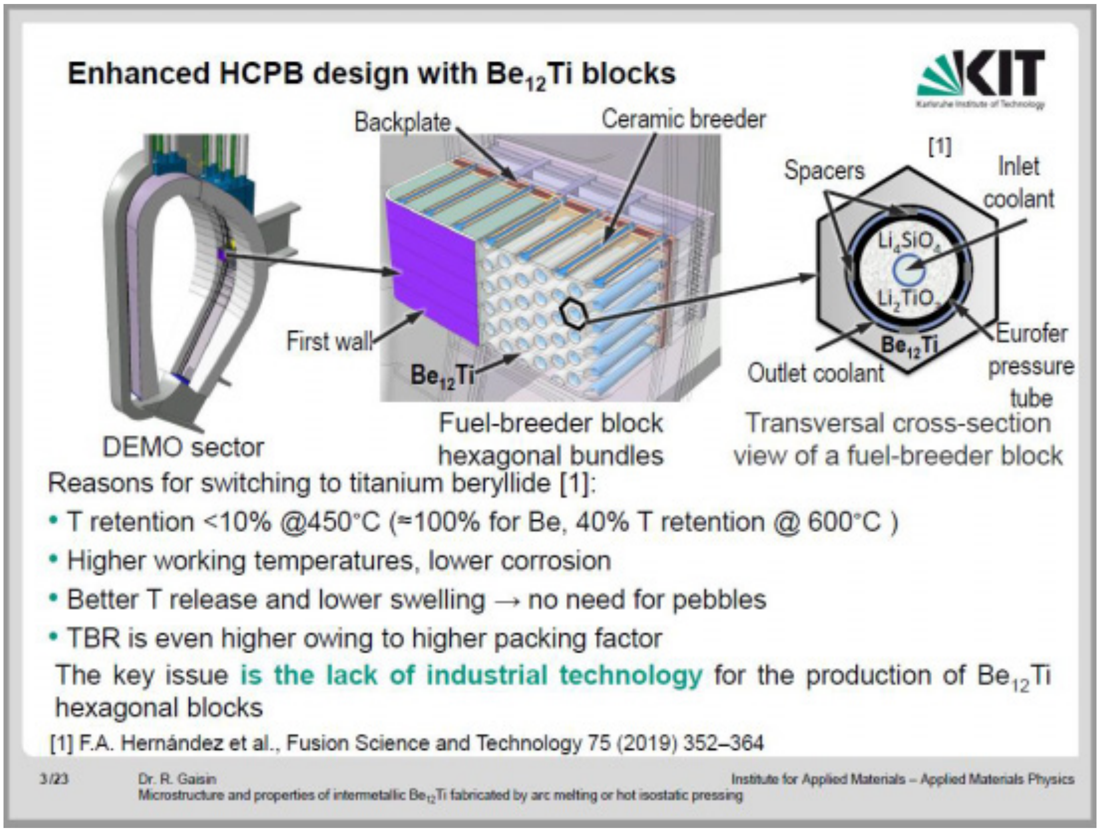
Enhanced HCPB design with $Be_{12}Ti$ blocks
KIT
Backplate
Ceramic breeder
[1]
Spacers
Inlet coolant
Li_4SiO_4
Li_2TiO_3
$Be_{12}Ti$
Eurofer pressure tube
Outlet coolant
First wall
$Be_{12}Ti$
DEMO sector
Fuel-breeder block hexagonal bundles
Transversal cross-section view of a fuel-breeder block
Reasons for switching to titanium beryllide [1]:
• T retention <10% @450°C (≈100% for Be, 40% T retention @ 600°C)
• Higher working temperatures, lower corrosion
• Better T release and lower swelling → no need for pebbles
• TBR is even higher owing to higher packing factor
The key issue is the lack of industrial technology for the production of $Be_{12}Ti$ hexagonal blocks
[1] F.A. Hernández et al., Fusion Science and Technology 75 (2019) 352–364
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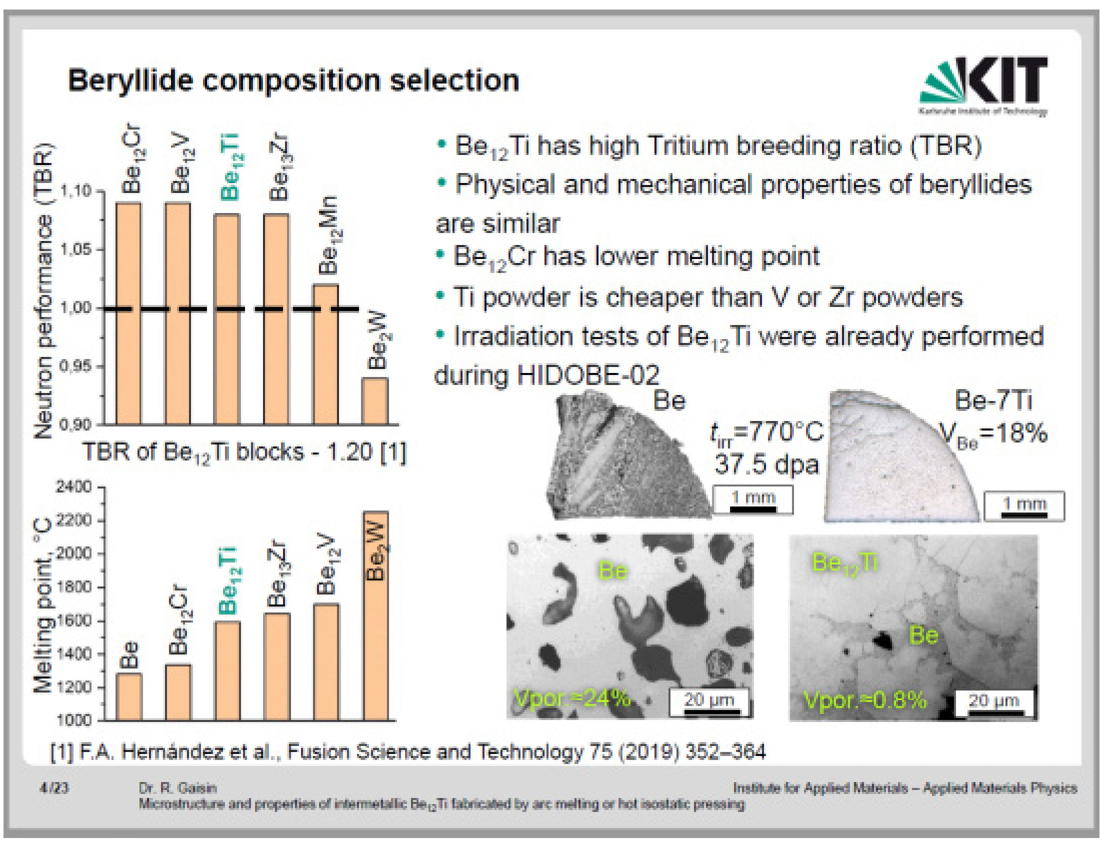
Beryllide composition selection
KIT
Neutron performance (TBR)
1,10
1,05
1,00
0,95
0,90
$Be_{12}Cr$
$Be_{12}V$
$Be_{12}Ti$
$Be_{13}Zr$
$Be_{12}Mn$
Be_2W
TBR of $Be_{12}Ti$ blocks - 1.20 [1]
Melting point, °C
2400
2200
2000
1800
1600
1400
1200
1000
Be
$Be_{12}Cr$
$Be_{12}Ti$
$Be_{13}Zr$
$Be_{12}V$
Be_2W
• $Be_{12}Ti$ has high Tritium breeding ratio (TBR)
• Physical and mechanical properties of beryllides are similar
• $Be_{12}Cr$ has lower melting point
• Ti powder is cheaper than V or Zr powders
• Irradiation tests of $Be_{12}Ti$ were already performed during HIDOBE-02
Be
t_{irr}=770°C
37.5 dpa
1 mm
Be-7Ti
V_{Be}=18%
1 mm
Be
Vpor.=24%
20 µm
$Be_{12}Ti$
Be
Vpor.=0.8%
20 µm
[1] F.A. Hernández et al., Fusion Science and Technology 75 (2019) 352–364
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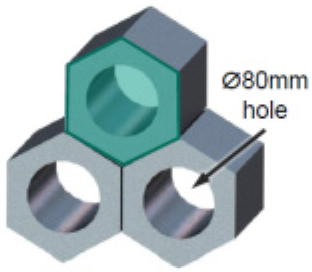
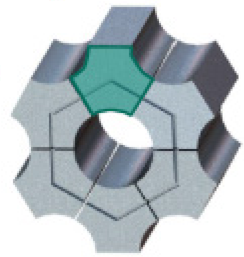
Manufacturing process selection

Hexagonal block with hole

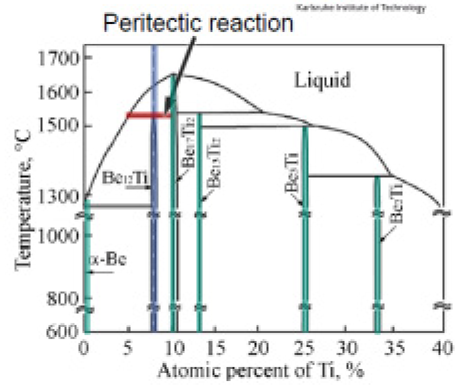


Ø144mm×150mm

Complex shape block



Ø85mm×50-100mm



$Be_{12}Ti$ blocks manufacturing

- Arc melting (KIT+IMSP)
- Powder metallurgy
  - Extrusion+HIP (KIT+KBHF)
  - Vacuum Hot Pressing (Ulba Metallurgical Plant)

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Be-Ti ingots arc melting

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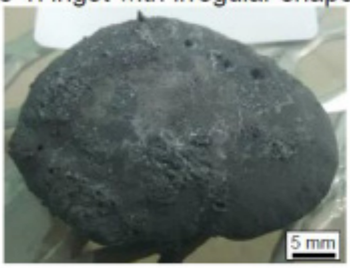
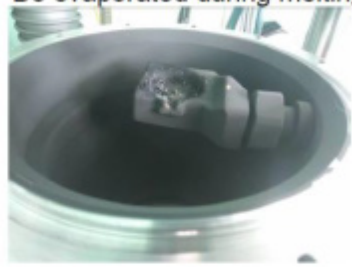
Arc Melting of $Be_{12}Ti$

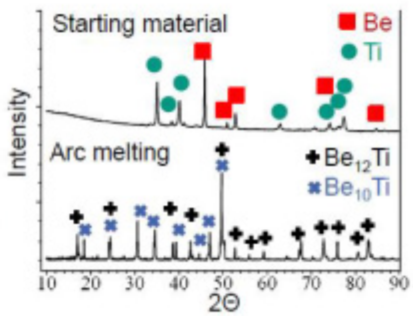
Be-Ti ingot with irregular shape



Be evaporated during melting



- 20-gram ingot was arc-melted
- Density is 2.111 g/cm³ or 92% of TD
- Microhardness is 980±140 HV0.1
- Be-Ti melting was accompanied by Be evaporation.
- Weight loss was 4%, and the composition could shift from Be-7.3Ti to Be-8.2Ti at.%
- $Be_{12}Ti$+$Be_{10}Ti$ phase composition after casting



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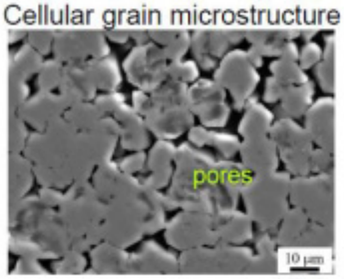
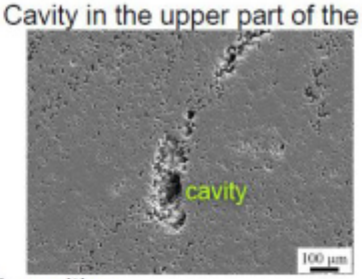
Microstructure of arc-melted Be-Ti

Cellular grain microstructure

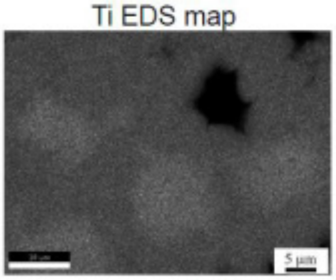


Cavity in the upper part of the ingot

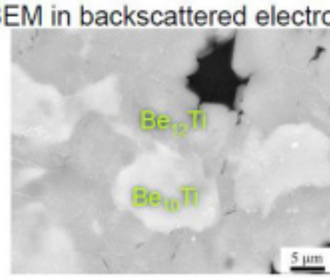


- High porosity ≈20%, cavities

Ti EDS map



SEM in backscattered electrons



- Two-phase metastable microstructure

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$Be_{12}Ti$ casting in Ulba Metallurgical Plant

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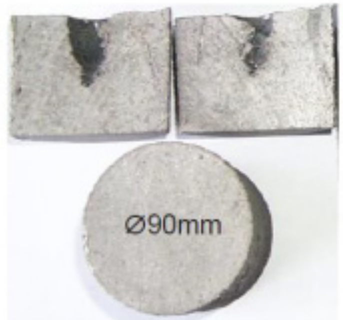
$Be_{12}Ti$ after Vacuum arc remelting

- In 2000-s Ulba casted $Be_{12}Ti$ ingots for JAEA
- Total amount of the melted $Be_{12}Ti$ was 60 kg
- Even after zone melting beryllide ingots have high porosity and cavities
- Casting of Be-Ti is impractical due to the evaporation of Be and peritectic reaction

$Be_{12}Ti$ after zone melting



[1-3] Courtesy of UMP

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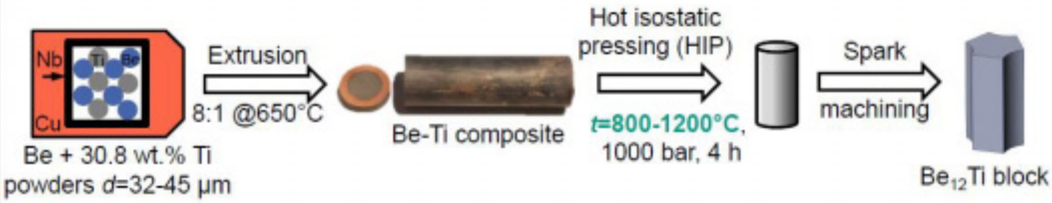
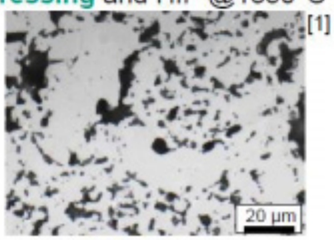
$Be_{12}Ti$ synthesis by Hot Isostatic Pressing

Fabrication of $Be_{12}Ti$ using extrusion and HIP

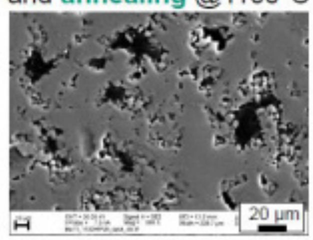


Be-Ti microstructure after **cold pressing** and HIP @1350°C



23% porosity

Be-Ti microstructure after extrusion and **annealing** @1100°C



25% porosity

[1] P. Kurinskiy et al. Fusion Engineering and Design 84 (2009) 1136–1139

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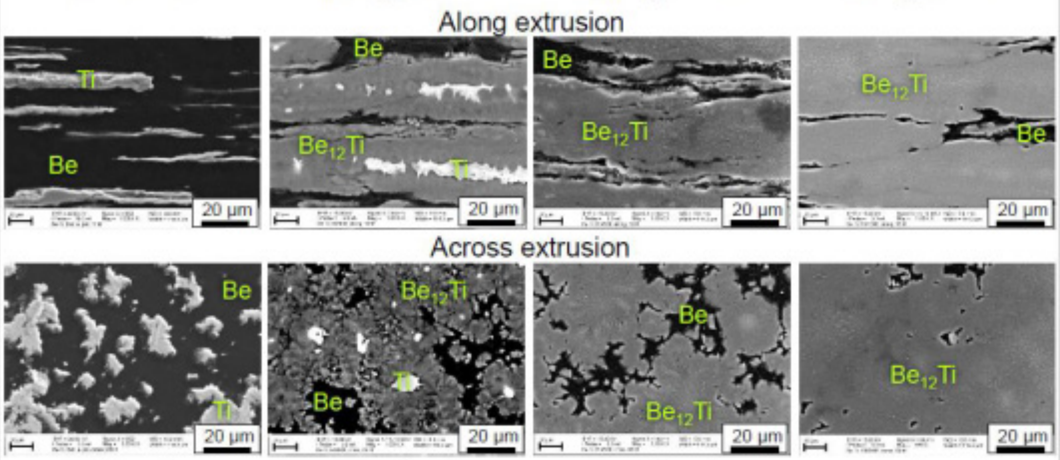
Effect of HIP on the microstructure of Be-7.3Ti

After extrusion | HIP @800°C | HIP @900°C | HIP @1000°C



After extrusion:

Phase	V. %	HV
Be	75	320
Ti	25	315

HIP @800°C:

Phase	V. %	HV
$Be_{12}Ti$	70	1130
Be+pores	25	370
Ti	5	-

HIP @900°C:

Phase	V. %	HV
$Be_{12}Ti$	79	1680
Be+pores	20	380
Ti	1	-

HIP @1000°C:

Phase	V. %	HV
$Be_{12}Ti$	90	1480
Be+pores	10	360

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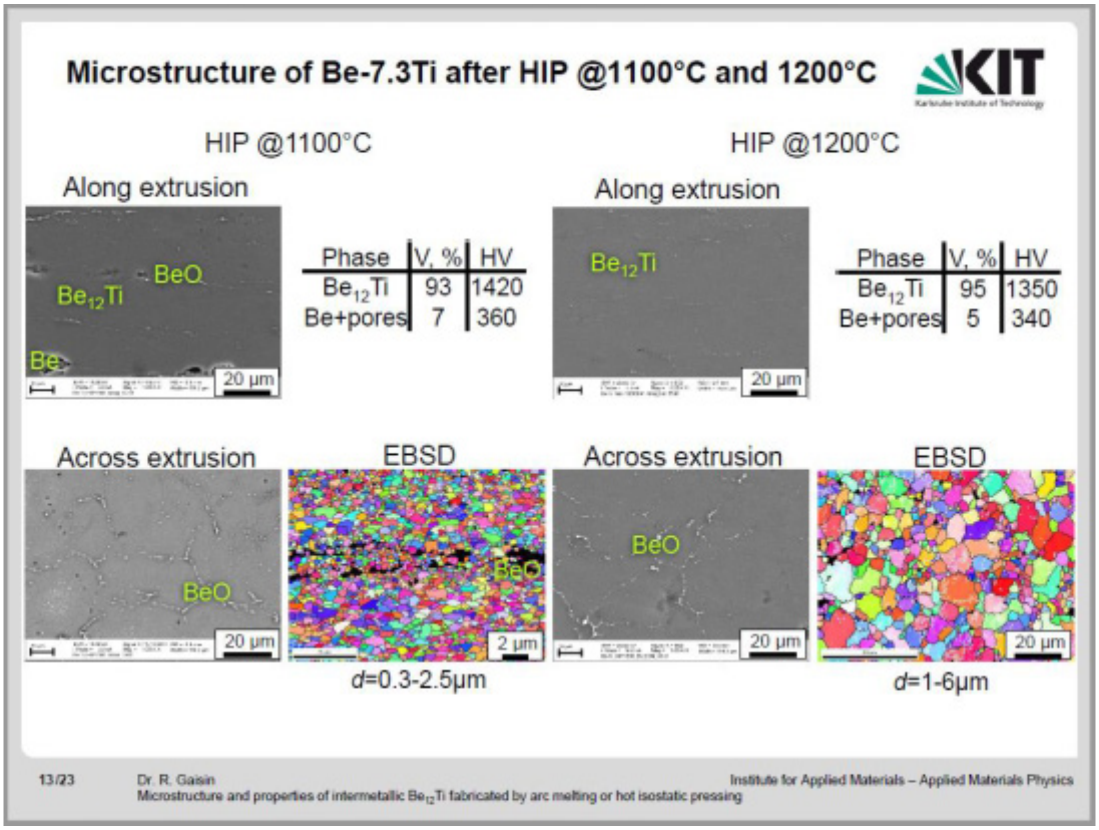
Microstructure of Be-7.3Ti after HIP @1100°C and 1200°C
KIT
HIP @1100°C
HIP @1200°C
Along extrusion
BeO
Be12Ti
Be
20 µm
Phase | V, % | HV
Be12Ti | 93 | 1420
Be+pores | 7 | 360
Along extrusion
Be12Ti
20 µm
Phase | V, % | HV
Be12Ti | 95 | 1350
Be+pores | 5 | 340
Across extrusion
BeO
20 µm
EBSD
BeO
2 µm
d=0.3-2.5µm
Across extrusion
BeO
20 µm
EBSD
20 µm
d=1-6µm
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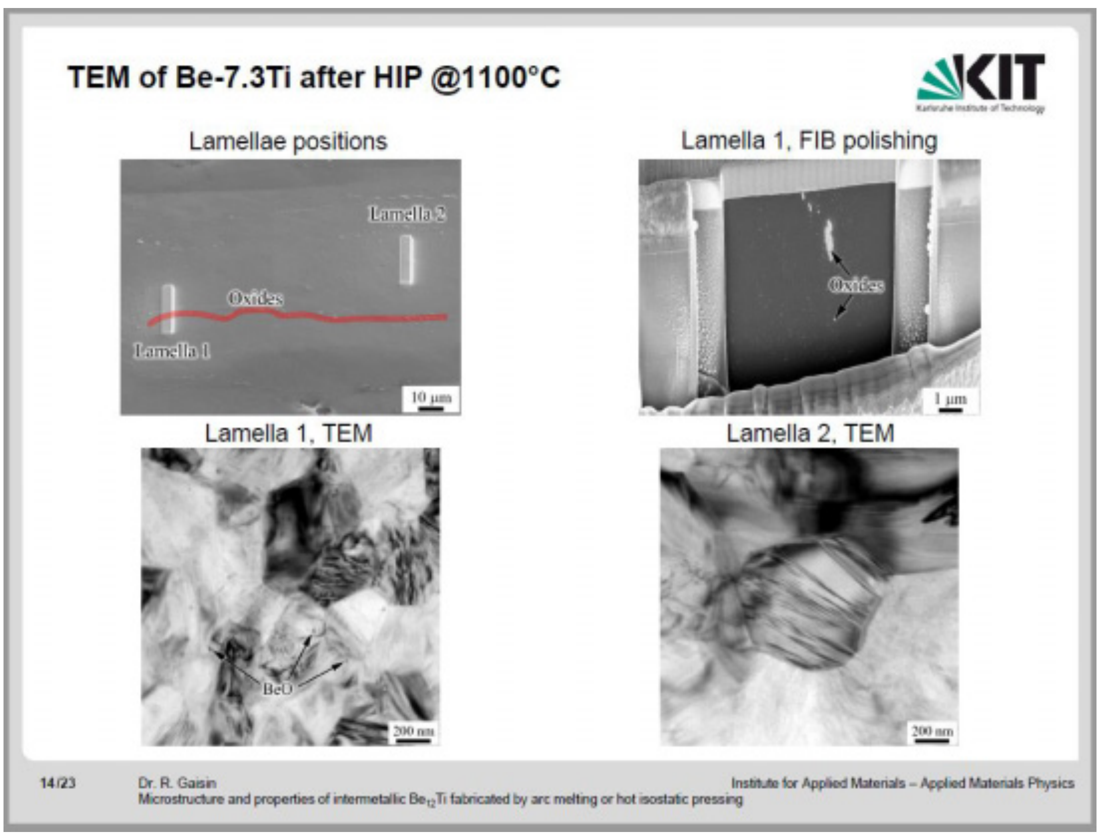
TEM of Be-7.3Ti after HIP @1100°C
KIT
Lamellae positions
Lamella 2
Oxides
Lamella 1
10 µm
Lamella 1, FIB polishing
Oxides
1 µm
Lamella 1, TEM
BeO
200 nm
Lamella 2, TEM
200 nm
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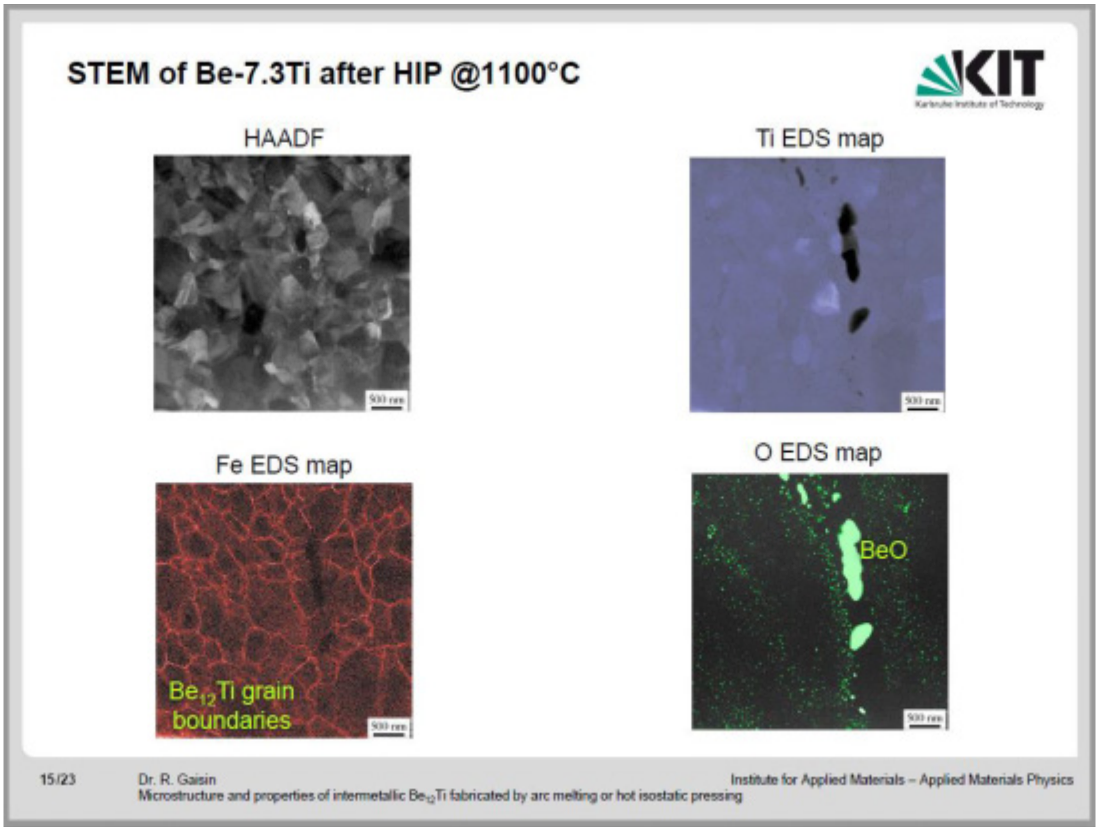
STEM of Be-7.3Ti after HIP @1100°C
KIT
Karlsruhe Institute of Technology
HAADF
500 nm
Ti EDS map
500 nm
Fe EDS map
Be12Ti grain boundaries
500 nm
O EDS map
BeO
500 nm
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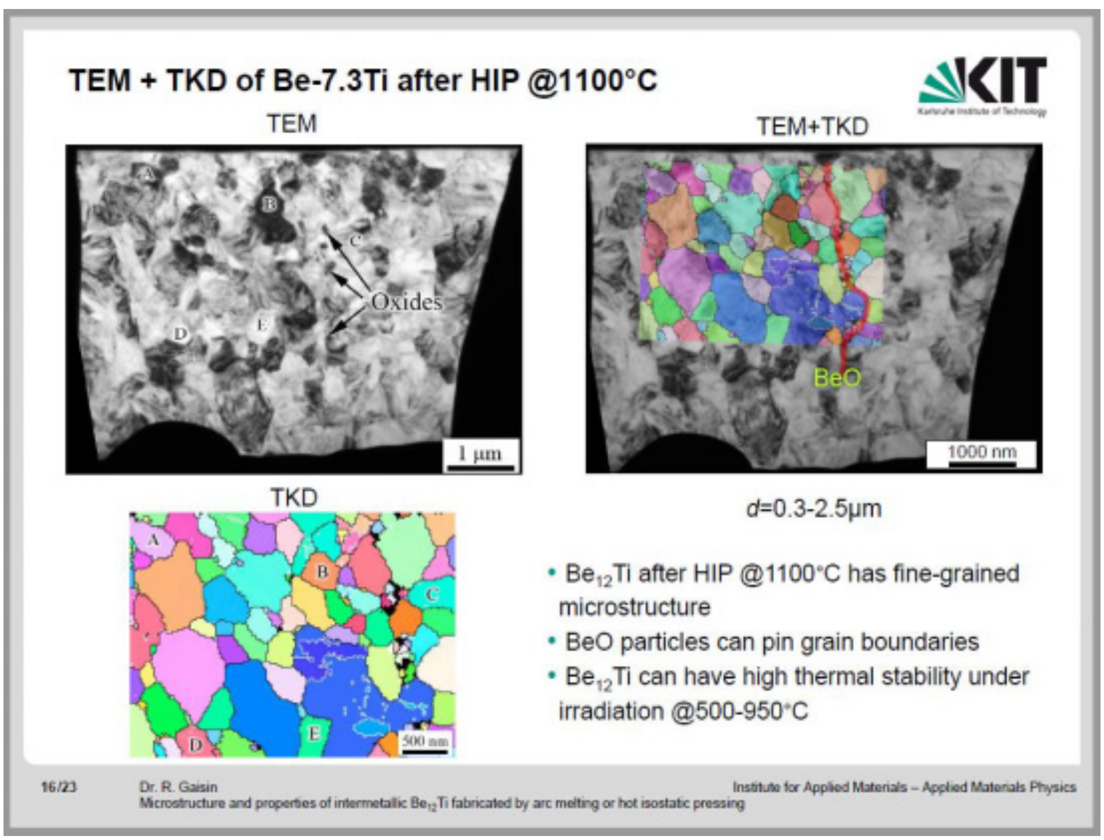
TEM + TKD of Be-7.3Ti after HIP @1100°C
KIT
Karlsruhe Institute of Technology
TEM
A
B
C
Oxides
D
E
1 µm
TEM+TKD
BeO
1000 nm
TKD
A
B
C
D
E
500 nm
d=0.3-2.5µm
• Be12Ti after HIP @1100°C has fine-grained microstructure
• BeO particles can pin grain boundaries
• Be12Ti can have high thermal stability under irradiation @500-950°C
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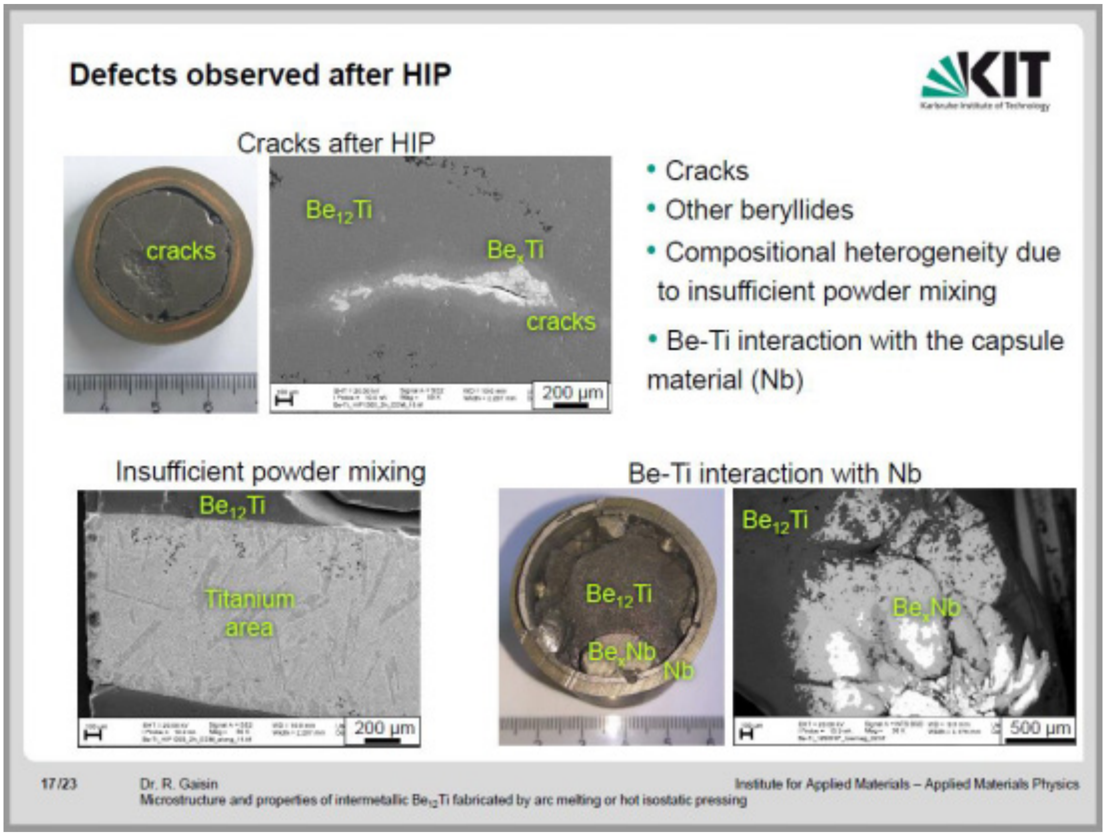
Defects observed after HIP
KIT
Cracks after HIP
cracks
Be12Ti
BexTi
cracks
200 µm
• Cracks
• Other beryllides
• Compositional heterogeneity due to insufficient powder mixing
• Be-Ti interaction with the capsule material (Nb)
Insufficient powder mixing
Be12Ti
Titanium area
200 µm
Be-Ti interaction with Nb
Be12Ti
BexNb
Nb
Be12Ti
BexNb
500 µm
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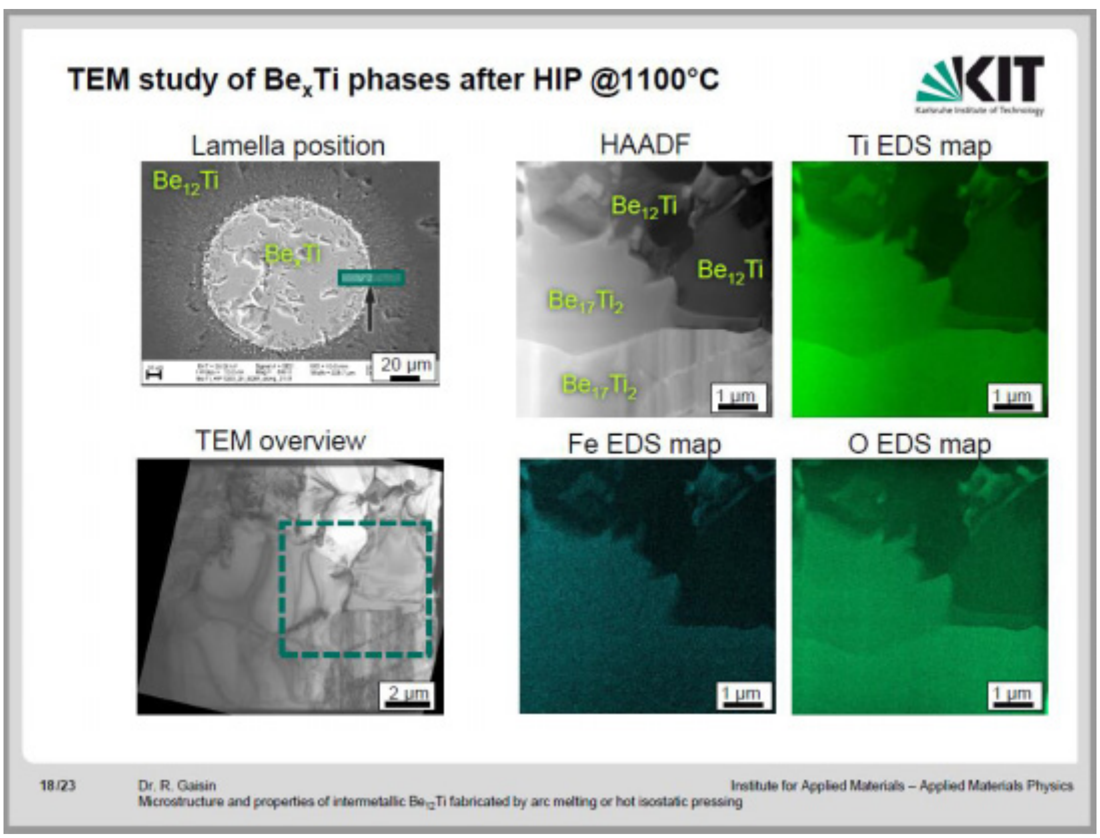
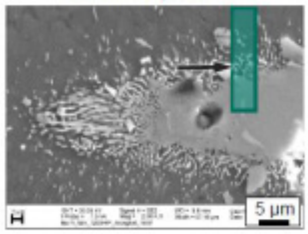
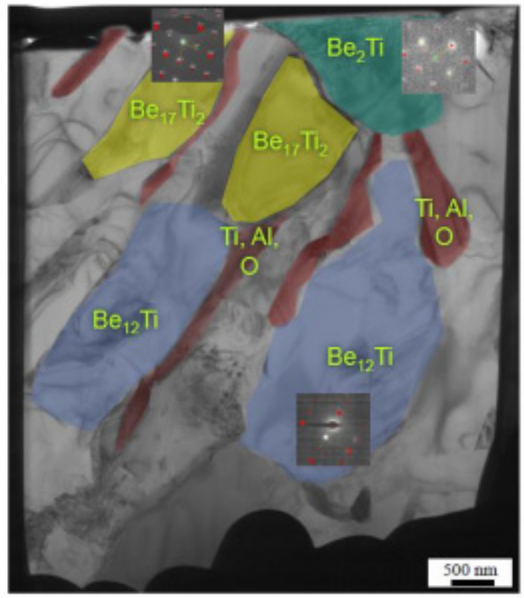

TEM study of BexTi phases after HIP @1100°C
KIT
Lamella position
Be12Ti
BexTi
20 µm
HAADF
Be12Ti
Be12Ti
Be17Ti2
Be17Ti2
1 µm
Ti EDS map
1 µm
TEM overview
2 µm
Fe EDS map
1 µm
O EDS map
1 µm
18/23
Dr. R. Gaisin
Microstructure and properties of intermetallic Be12Ti fabricated by arc melting or hot isostatic pressing
Institute for Applied Materials – Applied Materials Physics

TEM study of Be_xTi phases after HIP @1100°C

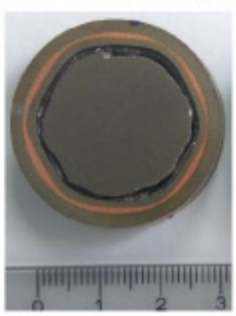
Lamella position





- $Be_{12}Ti$ grows from $Be_{17}Ti_2$ and Be_2Ti phases
- Be_2Ti decomposes to areas with higher Be content and areas with higher Ti, Al, O content

$Be_{12}Ti$ block mockup after HIP



$Be_{12}Ti$ after HIP@1100°C

Spark machining

$Be_{12}Ti$ block mockup
Ø25mm×35mm

- Extrusion should be carried out after precise mixing of the powders
- HIP temperature should be >1100°C
- Slow heating and cooling rates are required during HIP to avoid cracks

Outlook

- Characterisation of the mockup
- Irradiation tests 2020-2025
- Rapid heating-cooling experiments

Conclusions

Karlsruhe Institute of Technology

- Owing to intense beryllium evaporation and peritectic reaction during solidification, arc melting resulted in two phase $Be_{12}Ti$ and $Be_{10}Ti$ microstructure with high porosity
- Hot isostatic pressing after extrusion can provide $Be_{12}Ti$ microstructure with residue Be phase. $Be_{12}Ti$ after HIP @1100°C has fine-grained microstructure (d=0.3–2.5 µm) and high hardness (1420HV)
- To avoid defects, HIP should be performed @t>1100°C with slow heating/cooling rates after extrusion of precisely mixed powders

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Acknowledgments

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- The authors gratefully acknowledge S. Kuksenko, J. Claridge (UK Atomic Energy Authority, Culham Science Centre), and U. Jäntsch (KIT) for their assistance with the FIB sample preparation.

22/23 Dr. R. Gaisin Institute for Applied Materials – Applied Materials Physics
Microstructure and properties of intermetallic $Be_{12}Ti$ fabricated by arc melting or hot isostatic pressing


Karlsruhe Institute of Technology
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23
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Hot Extrusion of Be-Ti Powder Mixtures: Recent Achievements in Advanced Neutron-Multiplier Materials Production

Aniceto Goraieb[1], Pavel Vladimirov[2], and Christopher Dorn[3]

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[2]*Karlsruhe Institute of Technology, Institute for Applied Materials – Applied Materials Physics*
[3]*Be4FUSION LLC*

In future fusion reactors, lithium will be needed for the generation of the fuel, tritium. In taking on a leadership role in the development of critical materials for fusion energy, the Karlsruhe Institute of Technology (KIT) is specifically working on materials for blankets that will be used in a Helium-Cooled Pebble Bed (HCPB).

The Karlsruhe Beryllium Handling Facility (KBHF), a "Spin In" on the KIT North Campus. The necessary beryllium safety was built up with the support of KIT and commercial partner HIMA (Brühl, Germany). Novel production methods and new beryllium-based materials are now being qualified under cooperative agreements with the IAM-WPT (KIT, Germany), the world market leader for Beryllium Products, Materion Brush Inc. (Ohio, USA), and the Research Center for Hot Extrusion of the TU-Berlin.

Based on current conceptual designs for an ITER Solid Breeder Test Blanket Module (TBM), about 150kg of breeder and 300 kg of multiplier will be needed for a single TBM. Imagining an economically and logistically viable production process, a production rate of about one pebble per second is envisioned. Recycling is not being considered at this stage.

Over the past few years, the Rotating Electrode Method (REM) is being developed for production of titanium beryllide pebbles, using 1kg powder-derived Be-Ti extruded rods as the electrodes. These composite rods have a density of >90% from the hot-extrusion process. With the support of QST in Japan, initial yields of over 50% of pebble material in the needed size range have been achieved. Using this process, a production rate of 100 kg of beryllide pebbles per year seems realistic. This will be sufficient for the filling of an ITER TBM.

In contrast to ITER, a DEMO reactor will have a full-scale blanket, so the needed quantity of materials will be a thousand times greater than for ITER TBMs. Under the contract to KIT, KBHF, and Be4FUSION have written a white paper, which gives an overview of possible production methods for advanced neutron-multiplier materials, including the necessary safety conditions. As a result, of that analysis, the latest DEMO Blanket Design at KIT is based on beryllide material in block-form, rather than pebbles. Beryllide compounds will be preferred for use in DEMO over beryllium metal, as they have a much lower swelling rate under neutron irradiation and much lower reactivity than beryllium metal has under the envisioned DEMO conditions.

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State of the art

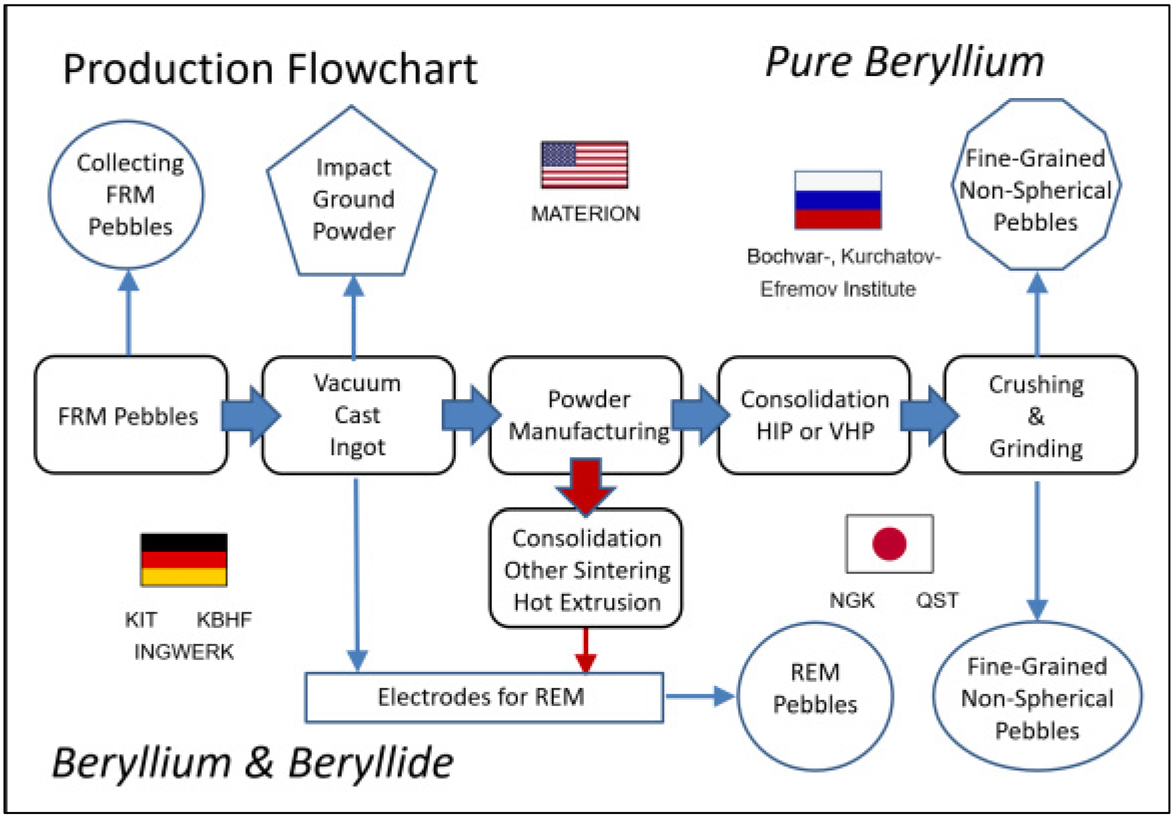
- NGK pebbles produced by REM are reference material for a Helium-Cooled-Pebble-Bed-Blanket
- For ITER a water-cooled version of TBM is foreseen but pure Beryllium causes safety problems
- Beryllides based on Beryllium and Titanium have gone through a long qualification process
- The production of pebbles by REM process seems promising for production of ITER amounts
- Electrodes of different sizes have been produced and about 180g pebbles were made of those

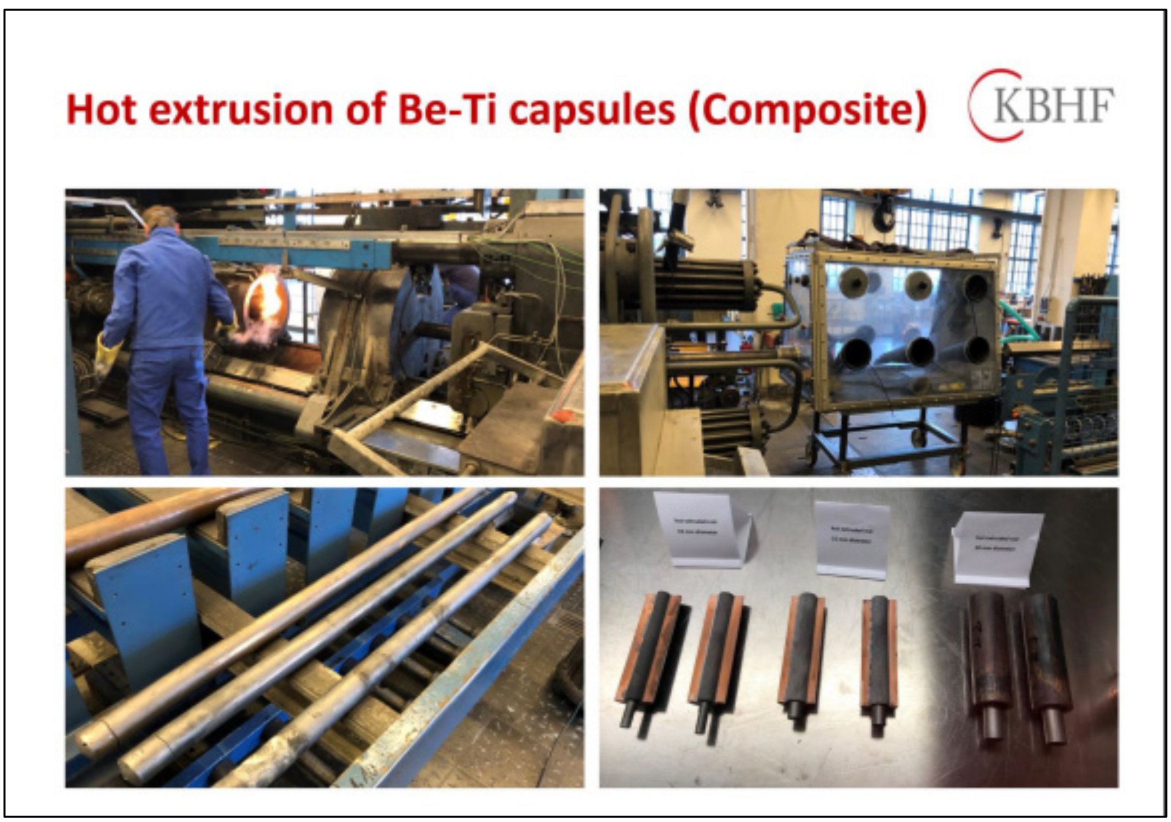
Thanks to Your support we could go a big step towards production conditions

- Together with Chris Dorn from Be4FUSION, a white paper “pebble production” was written
- The assumptions were confirmed by our international partners QST, NGK and MATERION
- A decision was made at KIT towards Beryllides and Be-Ti was chosen as promising candidate
- For DEMO a parallel concept is being looked at using “Honeycomb”-Structures made of Be-Ti

Now, it is about time to involve industry and start a feasibility study

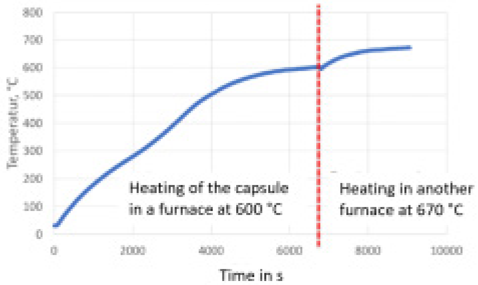
- Tests to produce them are performed by TUB/INGWERK, KBHF and from ULBA in Kazakhstan

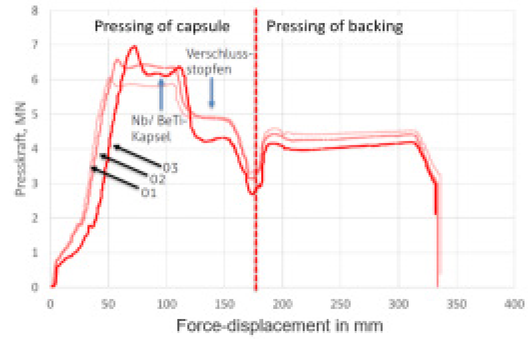

Production Flowchart
Pure Beryllium
Collecting FRM Pebbles
Impact Ground Powder
MATERION
Bochvar-, Kurchatov-Efremov Institute
Fine-Grained Non-Spherical Pebbles
FRM Pebbles
Vacuum Cast Ingot
Powder Manufacturing
Consolidation HIP or VHP
Crushing & Grinding
Consolidation Other Sintering Hot Extrusion
KIT KBHF
INGWERK
NGK QST
Electrodes for REM
REM Pebbles
Fine-Grained Non-Spherical Pebbles
Beryllium & Beryllide


Hot extrusion of Be-Ti capsules (Composite)
KBHF

Results from our hot extrusion campaigns INGWERK

Heating curve and comparison of the total forces





Increasing Be-Ti fraction leads to an increasing pressing force and the decreasing off the stationary press area (reason: increasing compaction energy)

Curve number 03:

- Maximum pressure force 7 MN is reached with the 27/28 mm core
- Homogeneous hot extrusion of the Niobium capsule containing the Be-Ti powder mixture
- Increasing of the press force when the end of the inner capsule is reached

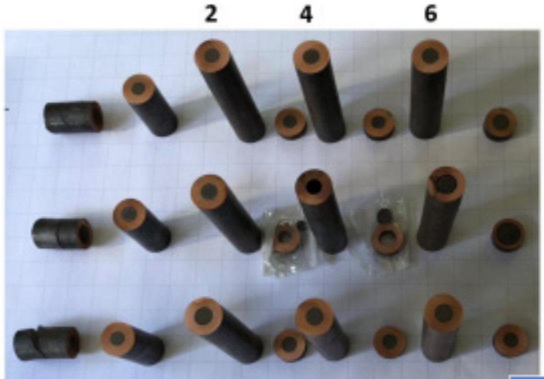
Optimizing the roundness of the core material

2 4 6

I 2α = 90°

II 2α = 120°

III 2α = 150°



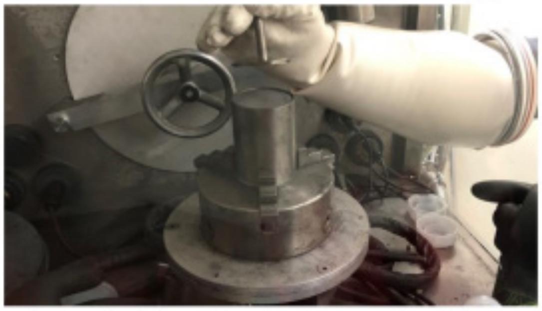
120° was our standard for most tests but it seems that 90° or 150° is giving better results.

Rotation tests at 5.000 RPM should be performed with the dismantled rods to find out which length stays mechanically stable and courses low "good" or acceptable vibrations.

	2 A	2 B	4 A	4 B	6 A	6 B
I y	12,2	11,8	12,3	12,3	12,4	13,3
I x	12,8	11,2	12,7	12,5	12,2	13,8
II y	11,8	10,6	12,3	12,2	12,0	18,2
II x	12,4	10,6	12,4	12,2	12,3	18,9
III y	12,4	10,7	12,0	12,2	11,4	11,5
III x	12,9	11,3	12,5	12,5	12,5	12,1

Production of Electrodes for REM-method

Production of Beryllide blocks or rods of bigger size:



- Green density reached in the capsule with powder is smaller than 50 %
- Therefore a direct HIP of the capsule would course extreme deformation
- HOT-Extrusion reaches densities of about 90 % in the Be-Ti-composite (≈ Hot Vacuum Pressing)
- HIP as the second step converts the composite in a Beryllide with high density (tests performed)

For pebbles HIP is not necessary, the material can be direct converted by REM

- If we can produce the right shape by hot-extrusion we can dismantle it using Niobium-capsule
- If we have to cut out the structure by electrical-wire-cutting we can simplify the capsule

The integral way of viewing things

KBHF

- ✓ We have successful produced rods with core diameter of about 28 mm
- ✓ It can be easily dismantled due to the Niobium inner capsule
- ✓ Over 1 kg of composite can be produced at a density > 90%
- ✓ For ITER it might be a possible alternative to Plasma Sintering (QST)

But:

- ➢ Over the thumb 25% was waste material that would be difficult to recycle
- ➢ Including REM > 50% of material can be converted
- ➢ This doubles the **price** of the basic material **$$$**

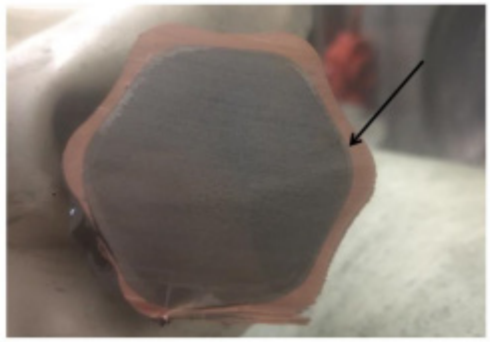
	1	2	3	4	5	6	7	8	9	10	11
I	/	/	/	/	31,92 31,92	30,09 30,38	30,23 30,23	28,46 28,46	28,12 28,45	27,51 28,19	28,21 28,04
II	27,34 27,27	27,87 27,63	27,67 26,83	28,16 28,09	28,4 28,0	28,05 28,05	28,42 28,84	28,87 28,71	28,94 28,21	28,14 28,64	28,43 28,56
III	26,92 26,18	26,45 27,15	27,8 27,05	○	Δ	/	/	/	/	/	/

HOT-extrusion campaign in January 2019

KBHF

Experiments performed for the production of angular shaped rods using indirect hot-extrusion

- ✓ It seems to be possible to produce other than round shapes depending of the wall thickness
- ➢ This process would only make sense if direct followed by HIP
- ➢ It is a batch process and the size will surely vary over the length of the rod
- ➢ Therefore HVP followed by HIP and electrical wire cutting of the structure might be better
- ✓ ULBA has successfully produced such parts, we wait now for qualifying them at KIT



Results confirm the validity of the assumption

- For ITER it will be possible to produce the relevant amounts of Beryllium and Beryllide pebbles
- If pure Beryllium pebbles are needed they can be produced by REM, as well (NGK)
- Alternatively MATERION can collect them from their existing pebble plant (FRM) by sieving
- Maximum capacity we can get seems to be in the range of 100kg/year, **price $$**
- If we can tolerate non spherical material it can be produced by crashing (& grinding), **price $**
- In this case even bigger amounts are realistic but a part off the material has to be reprocessed
- For DEMO we need several hundred tons (1 particle = about 1 mg) **1 Billion particles per ton!**
- Therefore an alternative concept for the DEMO-Blanket should be taken in account

- **Together with the TU-Berlin we have produced several rods of different sizes by hot extrusion**

- We found out that we will reach most limits with a rod of 30mm diameter and 150cm length
- It would contain max. 1.5kg Beryllium-Titanium composite
- This is about the maximum we can handle with our equipment at KBHF

INGWERK has already offered to produce 100 pieces per year

What about near to end-shape production?

KBHF

- Beryllium and Beryllides can only be produced by using powder processing
- Beryllium powder needs the handling under high H&S conditions
- To get from the powder to a pebble needs several steps like Electrodes and REM
- The use of binder for sintering (PIM) is not working in most cases
- Pebbles have an advantage when you have high swelling under irradiation
- To produce pebbles is difficult and cost intensive, especially for DEMO amounts
- Handling of pebbles causes dust production and therefore high H&S conditions
- Pure Beryllium has a disadvantage if water cooling is foreseen for the Blanket
- Beryllide has low swelling under irradiation compared to pure Beryllium
- It has also better oxidation behavior, but pebbles are even more difficult to produce
- "Near to end-shape" would allow to build complex structures with a **low price $**
- It might be possible to build the whole Blanket-Box and fill it with Li-ceramics
- Even for repairing parts or in case of recycling it might be a future solution



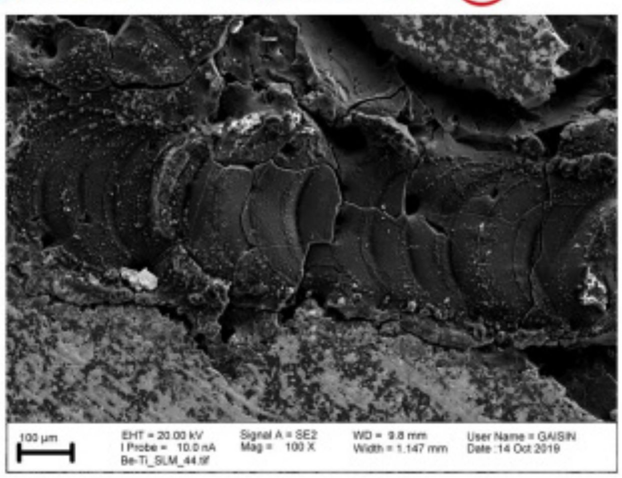
First test Laser Sintering of Be-Ti Beryllide at

KBHF

✓ We have Beryllide from the HIP experiments that we mill to powder
✓ A Thesis was performed showing that it should be theoretically possible
✓ A capsule has been designed and tested with Titanium powder before
✓ We were able to perform one test with $Be_{12}Ti$ Beryllide

But:

➢ The material seems to have cracks from thermal stress
➢ More tests have to be done under different conditions
➢ The microstructure has to be looked at



➢ **To get reproduceable results we would need professional equipment for Laser Sintering**
➢ **We have the Laboratory at KBHF available with the necessary safety standards**
➢ **It would also be a chance to look to recycling or repair of parts under "hot conditions"**

Strength

- KBHF has the Beryllium Facility
- Expertise in the production of Beryllide
- KIT has expertise in Laser Sintering
- The qualification can be done at KIT, too
- Laser-Sintering takes place in a box
- Handling under inert-gas conditions
- Low risk of gaining oxygen, high H&S
- A Bachelor Theses has been performed

Weaknesses

- Laser Sintering devices are expensive
- We would need sponsors for the project
- Not much work has been performed

Opportunities

- "Near to end-shape" would allow to build complex structures for other customers
- A sponsor could sell these parts (cash-back)
- Even for repairing parts it might work
- It might be possible to find new markets for all kinds of Beryllides
- Rapid-Prototyping will be the future concerning any product and material

Threats

- Does the "alloying" of Be-Ti work by LS?
- Laser-sintered parts have lower density
- Additional HIP process might be necessary (cost intensive $$)

Aims and time table for our efforts

Pure Beryllium (should be available in 2025 for a first "dummy" ITER TBM):

- **Qualifying the mentioned and available materials** for the use in an ITER-Test-Blanket
- **Calculating the overall costs and production efficiency** together with our partners from industry

Be-Ti-Beryllide pebbles (should be available in 2030 for a second ITER TBM):

- Automation of rod production by hot extrusion together with INGWERK and the TU-Berlin
- **Test how the amount and the range of produced pebbles depend on the electrode diameter**
- Optimize, simplify and scale-up the capsules to the requested size of electrodes needed for REM

Be-Ti-Beryllide blocks or rods (should be tested in a third TBM and be available for DEMO):

- **Producing single phase Be-Ti-Beryllides** in a bigger block or rod (15cm diameter, 50cm long)
- **Building up a Modell-scale "Honeycomb"-Structure** for thermal and irradiation tests

Suggested strategy:

2019: Find cooperation partners from industry and possible sponsors for a Laser-sintering device
2020: Starting the test production of 100kg (per year) of the qualified Beryllium material
*2021: R&D work on **"3D printing"** of complex Demo-Blanket relevant structures made **of Beryllide***


Catch the Sun: How Fusion Energy can Benefit our Societies
Event Information
4 November 2019,
6:30 PM to 8:00 PM
New York City
Organizer: German Center for Research and Innovation (DWIH NY), University of Freiburg, KIT & ITER
In the United States and Germany, interest in fusion energy is growing, as evidenced by the increase in both publications, start-ups on and public interest in the topic.
But in both the US and Germany, questions remain about the feasibility and practicality of fusion energy as a cost-effective and viable power source for industry and civil society.
These questions will be addressed by representatives of science, politics, companies and other stakeholders in the moderated panel discussion (Nov 4), followed by an expert meeting (Nov 5).
The aim is to foster a dialogue – also with the audience – about the role of fusion energy in both American and German society and consider how further developments in this field might be coordinated.
Please register here:
https://www.dwih-newyork.org/en/event/catch-the-sun/
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Session 3: Beryllium Materials under Extreme Conditions

Radiation-Induced Formation of Gas Bubbles in Beryllium after Neutron Irradiation up to 6000apm Helium Production

M. Klimenkov (KIT, Germany) et al.

Radiation-induced formation of gas bubbles in beryllium after neutron irradiation up to 6000appm helium production

Michael Klimenkov, Ute Jäntsch, Jan Hoffmann, Pavel Vladimirov, and Anton Möslang

Karlsruhe Institute of Technology, Hermann-von-Helmholtz-Platz 1,
76344 Eggenstein-Leopoldshafen, Germany

The current interest in mechanical properties and microstructure of neutron-irradiated beryllium refers to its planned application in the Helium-Cooled Pebble Bed (HCPB) European concept of a breeding blanket of DEMO [1,2]. Irradiation experiments in high-neutron-flux nuclear research reactors yield information about microstructural evolution of beryllium under conditions relevant to fusion (temperature, damage dose, helium and tritium productions) excluding 14MeV neutrons impact which is not present in the neutron spectra of fission reactors. The HIDOBE-02 irradiation campaign accomplished at the HFR, Petten corresponds to 1246.5 Full Power Days at a reactor power level of 45MW in the temperature range from 410°C to 680°C. Transmission electron microscopy (TEM) has been to study the evolution of voids during neutron irradiation at different temperatures. The target preparation of specimens was performed using focused ion beam (FIB).

TEM study shows the formation of radiation-induced hexagonal flat gas bubbles inside the grains, however at the lowest irradiation temperature of 410°, the pebbles show a uniform shape. The diameters of the bubbles increase from a few nanometers at 410°C to more than hundred nanometers at 680°C. The number density of bubbles decreases, correspondingly, by more than two orders of magnitude. The preferable formation of bubbles along the grain boundary and dislocation lines was observed. Analytical investigations using electron energy loss spectroscopy show the presence of He and H23 inside bubbles.

Spherical second-phase precipitates formed at GBs and inside grains were observed at the two lowest (370°C and 440°C) irradiation temperatures only. EDX mapping reveals that precipitates inside grain and at the GB have increased iron and aluminum content suggesting formation of Fe-Al-Be phase. In the material irradiated at 440°C, most precipitates have also Fe-Al-Be composition, while several other single- and multi-phase precipitates were found. The Fe-Al-Be phase is observed as 10-15nm precipitates inside grains and as 200 nm particle attached to a gas bubble at the GB. The present study shows detailed microstructural changes induced by neutron irradiation in beryllium.

[1] M. Klimenkov et al., Journal of Nuclear Materials 455 (2014), pp. 660–664.

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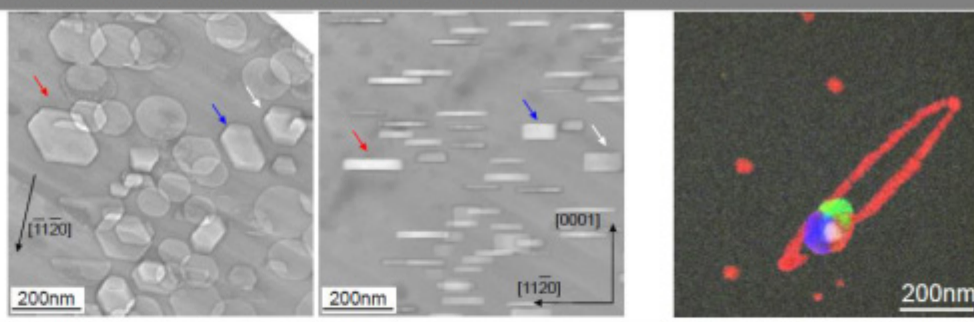
Radiation induced formation gas bubbles in beryllium after neutron irradiation up to 6000 appm helium production

M. Klimenkov, P. Vladimirov, N. Zimber, U. Jäntsch and A. Möslang

Karlsruhe Institute of Technology , Institute for Applied Materials - Applied Materials Physics (IAM-AWP)



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Outline

Karlsruhe Institute of Technology

- Introduction
- HIDOBE I and HIDOBE II radiation programs
- TEM analysis of irradiated beryllium
  - Bubbles inside grains
  - Microstructure of grain boundaries
- Detection of He and ^{3}H inside bubbles
- Conclusion

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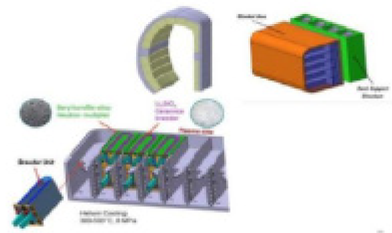
Motivation – application of beryllium in fusion technology

Helium Cooled Pebble Bed Blanket



Production of helium and tritium (^3H) by transmutation reactions

$^9Be + n \rightarrow 2\,^4He + 2n$ (E_n>2.7 MeV)

$^9Be + n \rightarrow {}^7Li + {}^3He$ (E_n>10.7 MeV)

$^7Li + n \rightarrow {}^4He + {}^3H + n$

$^9Be + n \rightarrow {}^6He + {}^3H$ (E_n> 0.6 MeV)

$^6He \rightarrow {}^6Li + b^- + \nu$

$^6Li + n \rightarrow {}^4He + {}^3H$

- Application as a "First Wall" material in ITER.
- As neutron multiplier material in different tritium-breeding blanket concepts for the future demonstration fusion power plant DEMO.

Prediction of irradiation resistance of beryllium pebbles under close-to-fusion conditions.

- operation temperature,
- accumulated damage dose,
- amount of helium and tritium generated by neutron-induced transmutation.

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Introduction

Neutron irradiation programs **Fusion for Energy**

High Dose Beryllium irradiation program **(HIDOBE-I)**
(2005-2007)
18 dpa displacement per atom — **3000 appm ^4He**
Irradiation temperatures — **300 appm ^3H**
686K (410°C), 753K (480°C), 861K (590°C), 968K (700°C)

High Dose Beryllium irradiation program **(HIDOBE-II)**
(2005-2011)
High-Flux Reactor (HFR), Petten, Netherlands
38 dpa displacement per atom — **5900 appm ^4He**
Irradiation temperatures: — **640 appm ^3H**
643K(370°C), 713K(440°C), 813K(540°C), 923K (650°C)

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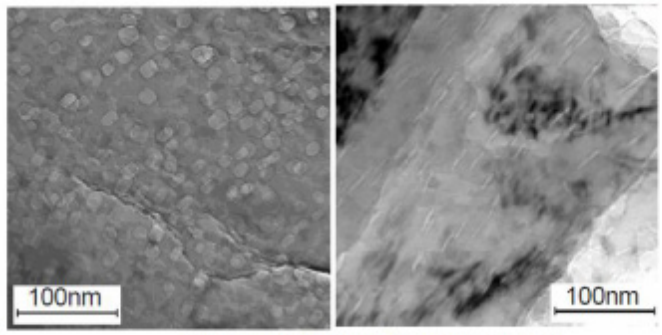
Review - HIDOBE I

Sample preparation
using special crushing tool.

Beryllium powder (mostly small pieces of 5-10 µm) was deposited on a TEM copper grid covered by a thin perforated carbon film.

Developmen and fabrication in Fusion Materials Laboratory (KIT).

TEM images of Be irradiated at 861K (590°C) in different orientations

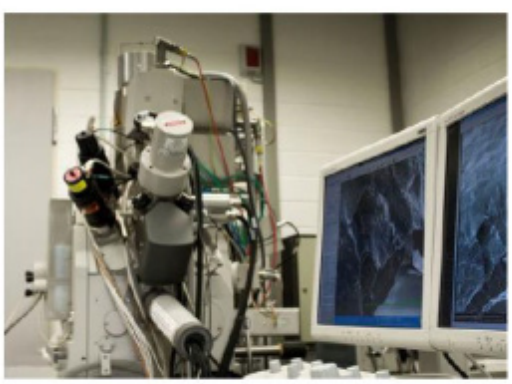


No possibility for thickness control
Often deformed pieces
Very few pieces with prismatic orientation
No piece with grain boundary

M. Klimenkov, et. al Journal of Nuclear Materials 455 (2014) 660–664

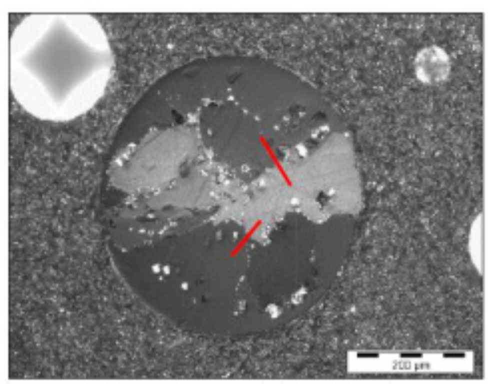
Preparation of TEM specimens

New Focused Ion Beam (FIB) in Fusion Materials Laboratory at KIT



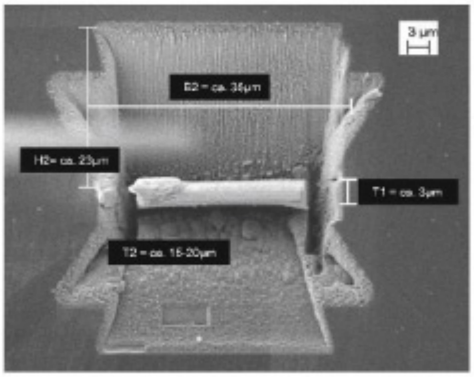
FIB (Nova 200)
Production ThermoFisher Scientific

Targeted preparation on the grain boundaris



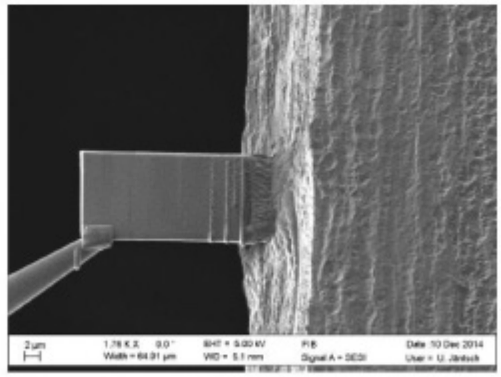
Specimen preparation using Foccused Ion Beam

Preparation of thin lamella



Removal of larger amounts of surrounding material using Ga ion beam.

Deposition on the Cu greed



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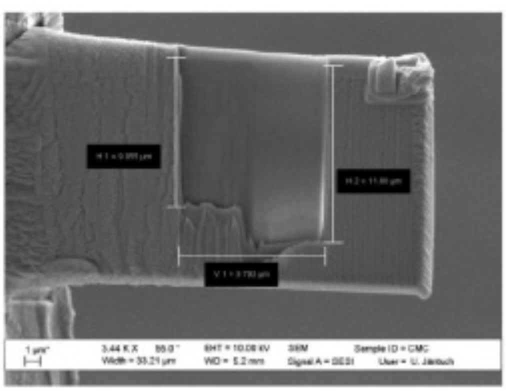
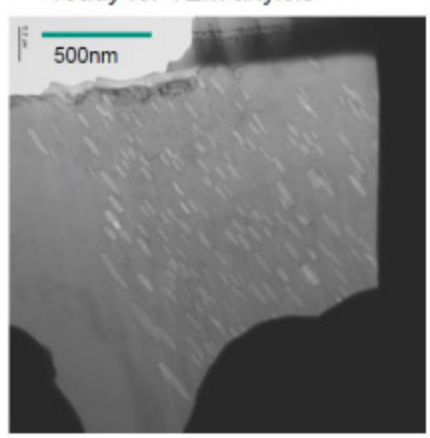
Specimen preparation using Foccused Ion Beam (FIB)

Thinning procedure
Formation of thin window



Final result – lamella is ready for TEM anylsis



The lamella thickness varried from 120 to 350nm mean path in beryllium ≈0,6-2.1λ

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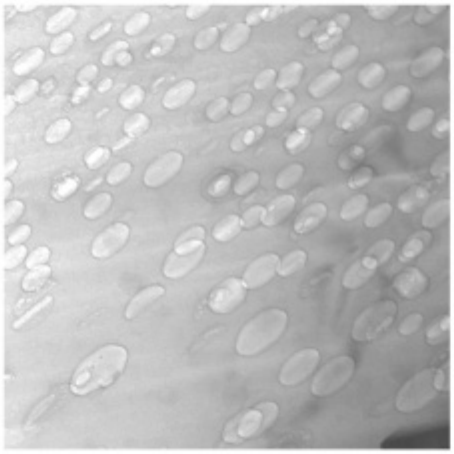
Shape of the bubbles

Identification of the real shape of the bubbles.

Difference between projection and real 3D shape.



Oval projection = elipsoidal bubbles?

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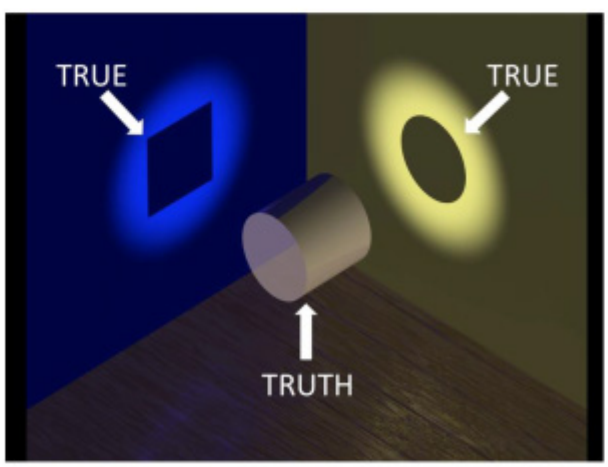
Shape of the bubbles

Identification of the real shape of the bubbles.

Difference between projection and real 3D shape.



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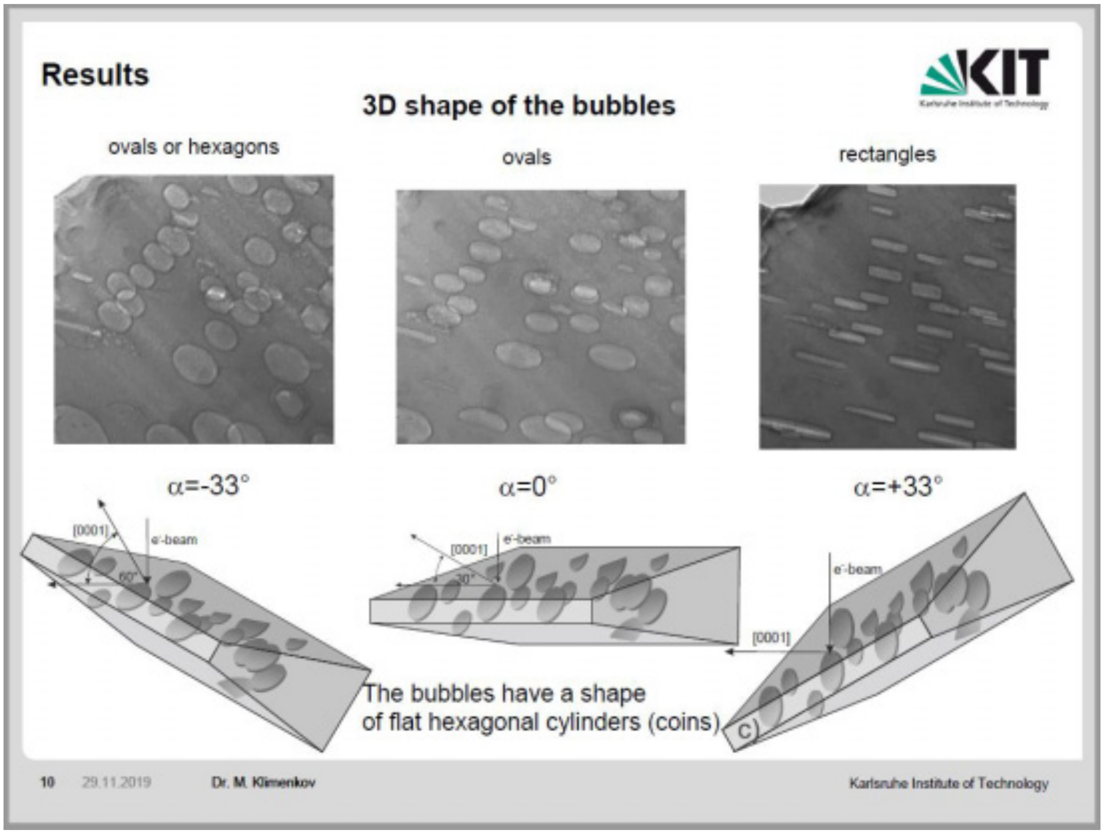
Results
3D shape of the bubbles
KIT
ovals or hexagons
ovals
rectangles
α=-33°
α=0°
α=+33°
[0001]
e-beam
60°
30°
The bubbles have a shape
of flat hexagonal cylinders (coins)
C
10
29.11.2019
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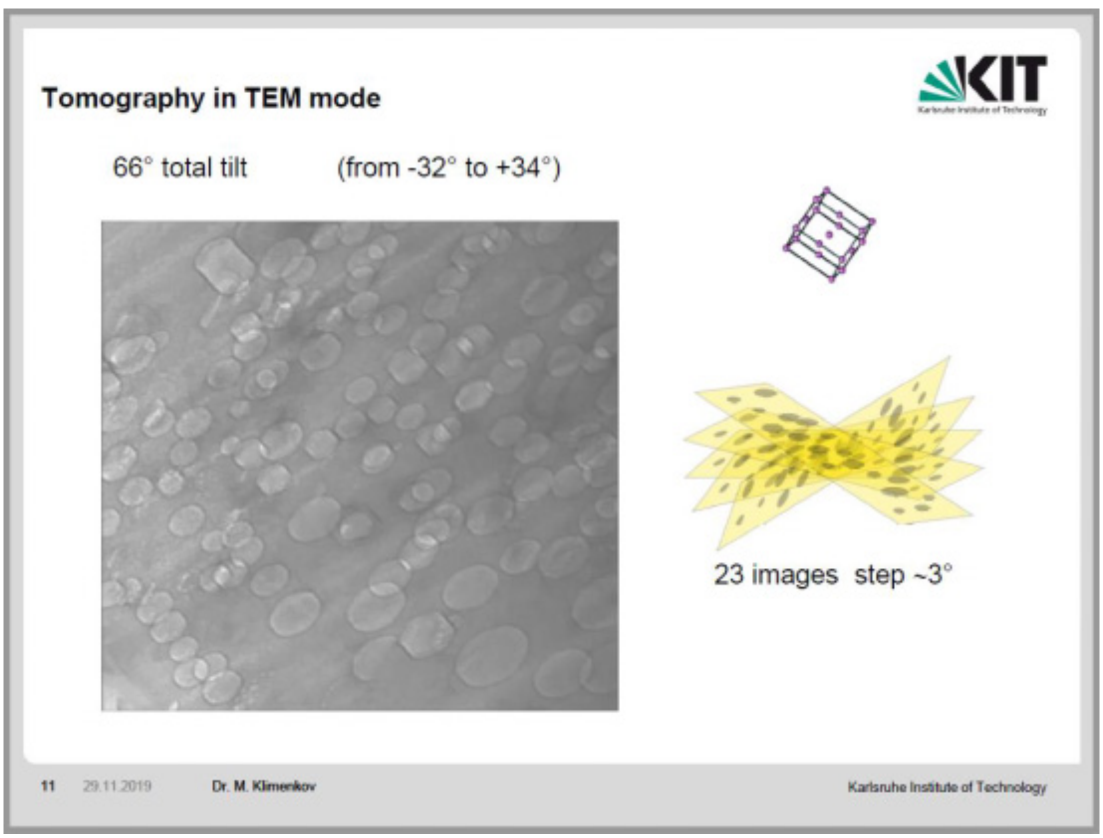
Karlsruhe Institute of Technology


Tomography in TEM mode
KIT
66° total tilt
(from -32° to +34°)
23 images step ~3°
11
29.11.2019
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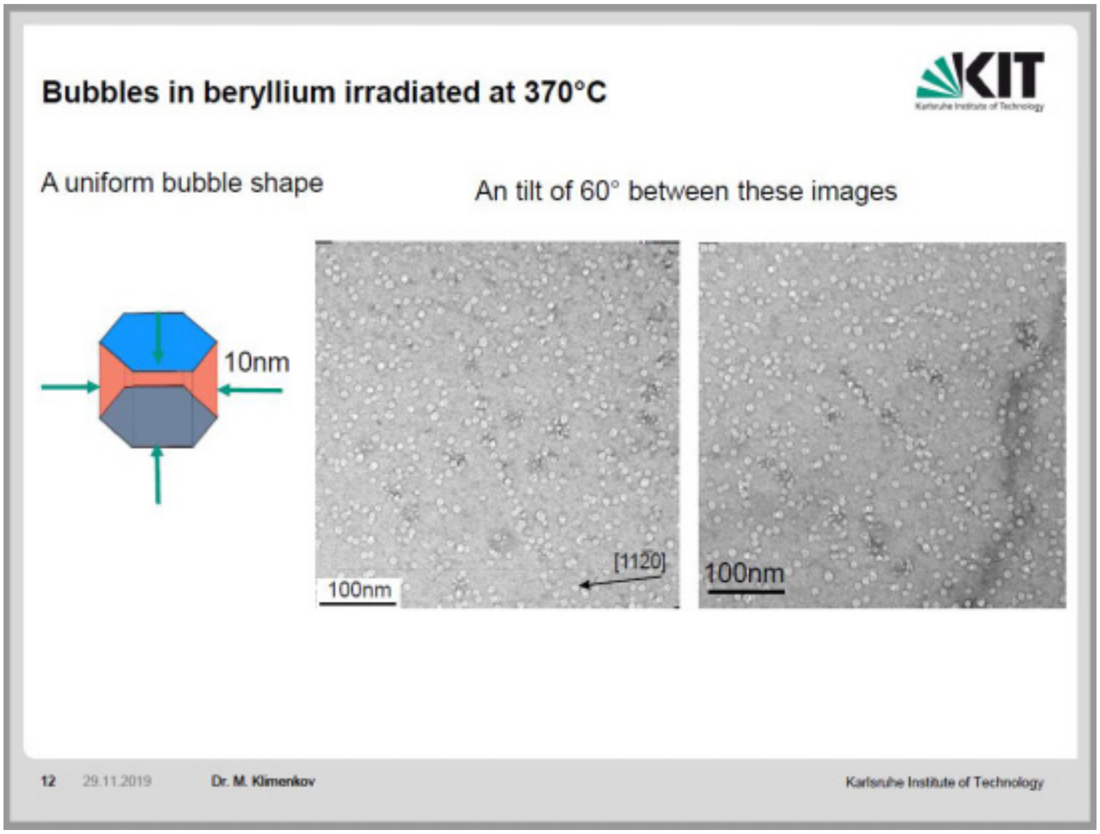
Bubbles in beryllium irradiated at 370°C
A uniform bubble shape
An tilt of 60° between these images
10nm
100nm
100nm
12
29.11.2019
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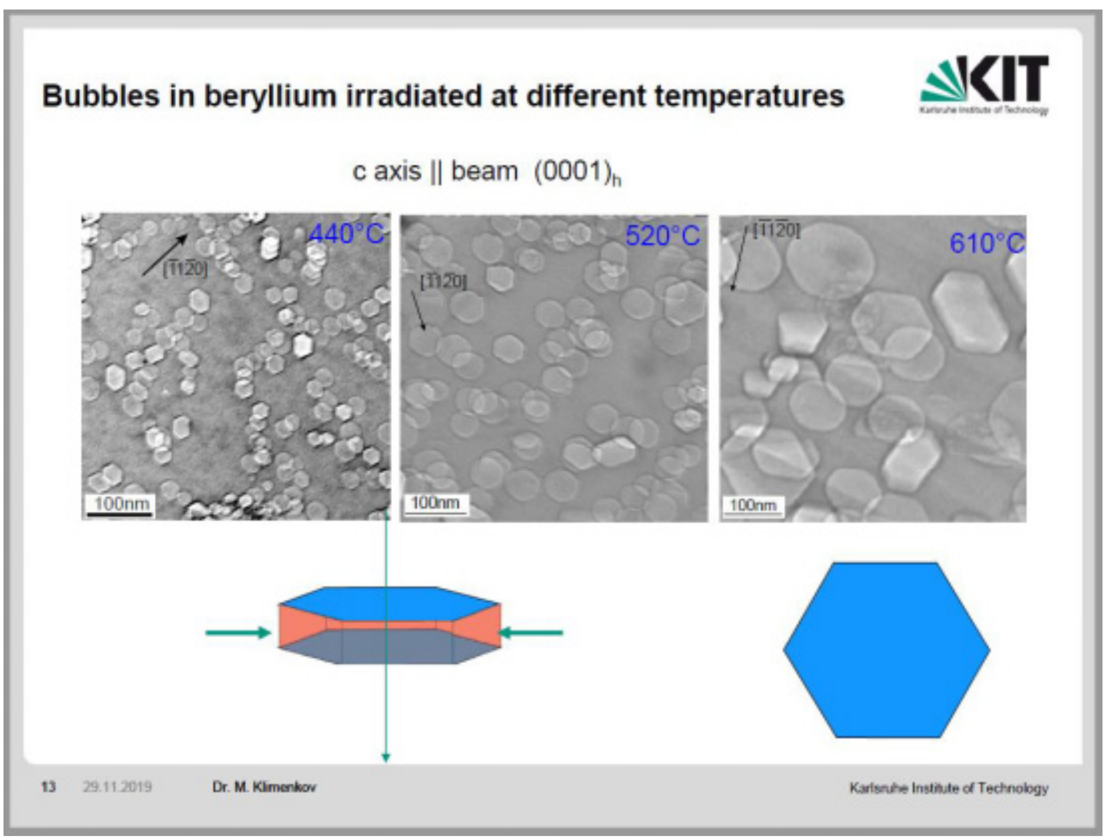
Bubbles in beryllium irradiated at different temperatures
c axis || beam (0001)h
440°C
520°C
610°C
100nm
100nm
100nm
13
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Bubbles in beryllium irradiated at different temperatures

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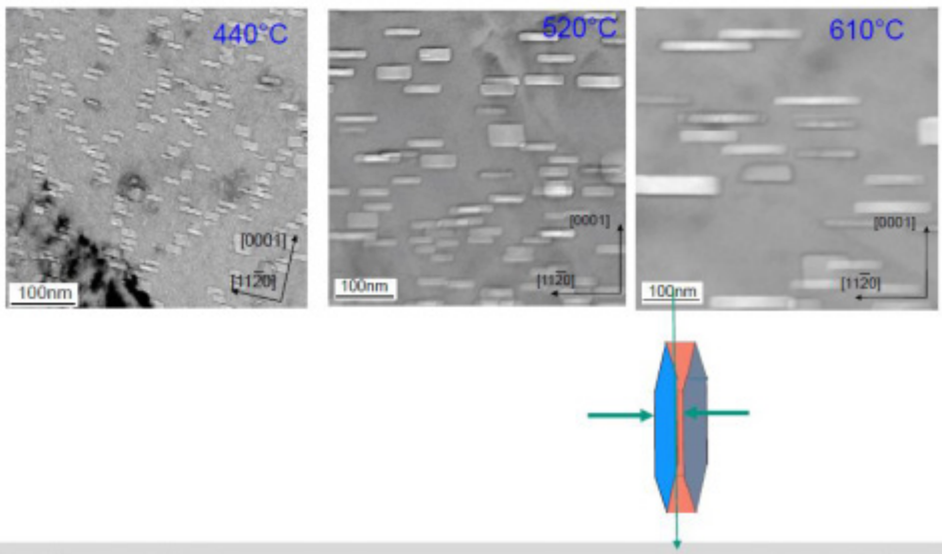
Images of the bubbles in the prismatic orientations



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Quantitative evaluation

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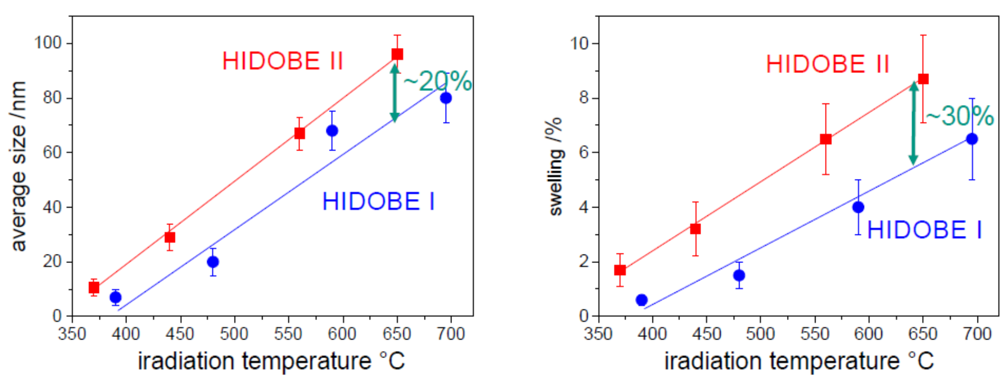
Effect of radiation dose on the microstructure of irradiated beryllium

Bubble diameter

Swelling



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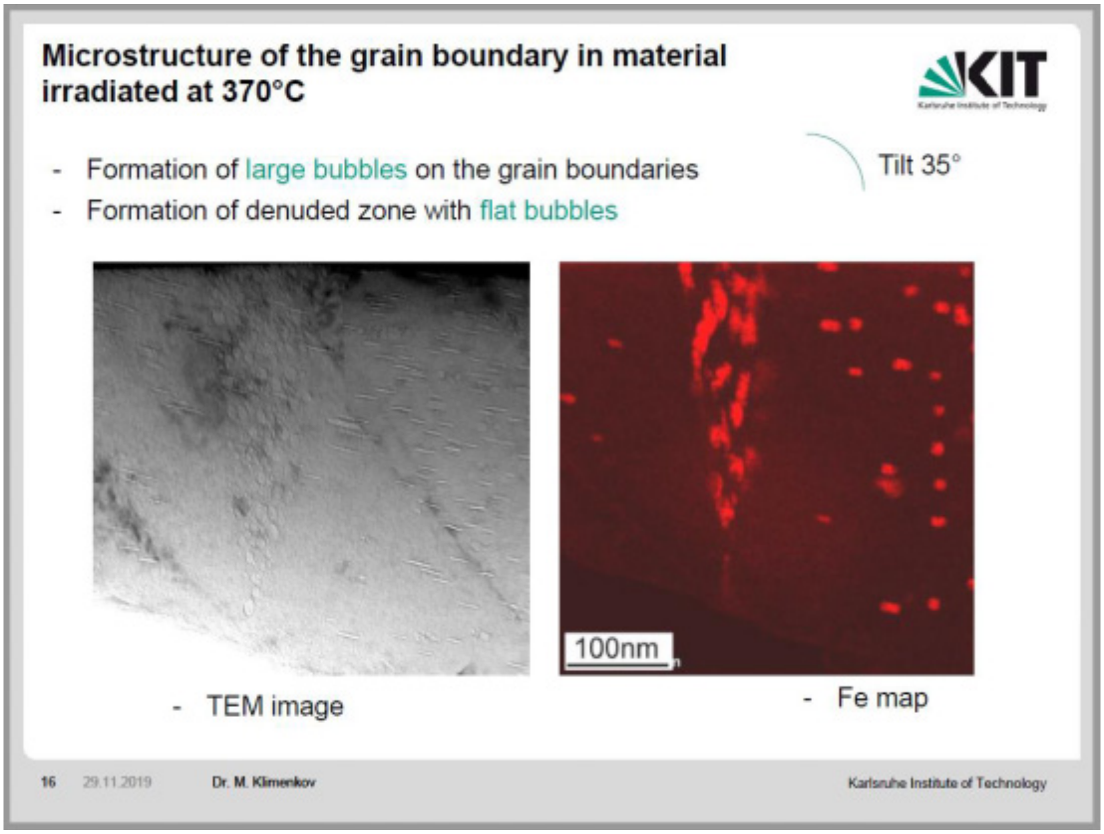
Microstructure of the grain boundary in material irradiated at 370°C
KIT
Karlsruhe Institute of Technology
- Formation of large bubbles on the grain boundaries
- Formation of denuded zone with flat bubbles
Tilt 35°
100nm
- TEM image
- Fe map
16
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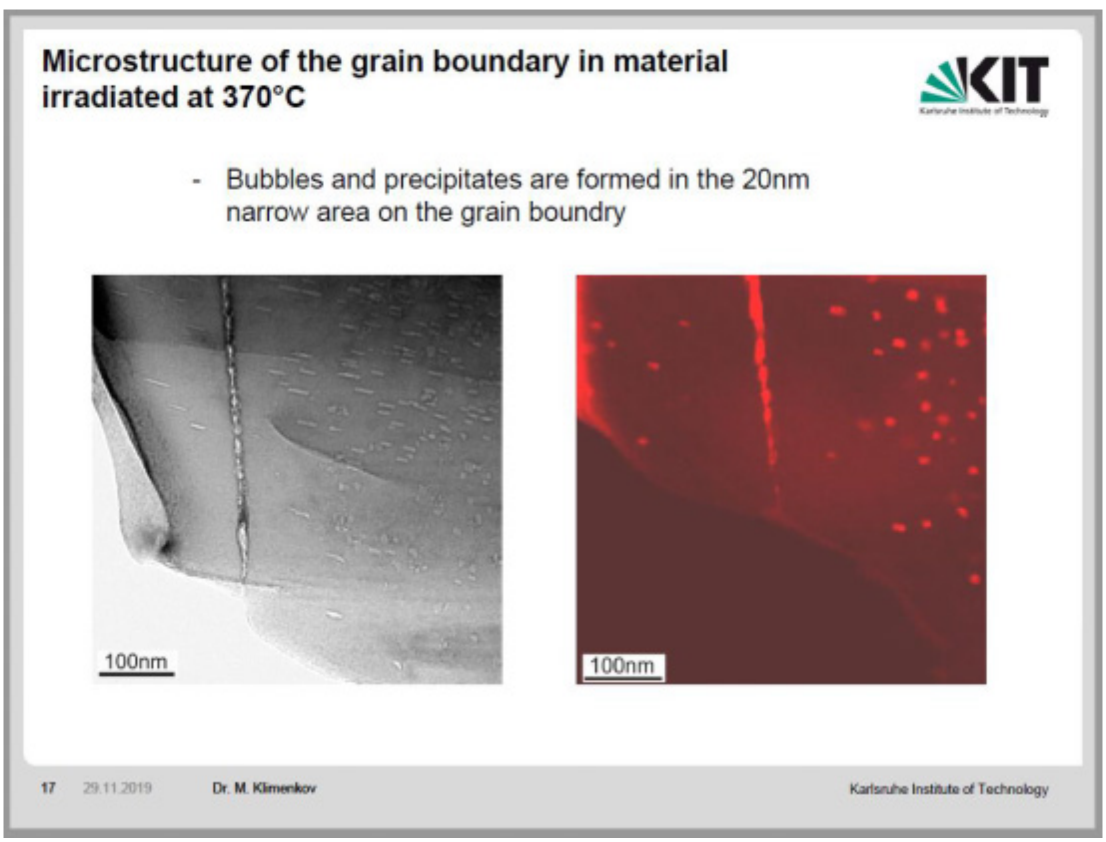
Karlsruhe Institute of Technology


Microstructure of the grain boundary in material irradiated at 370°C
KIT
Karlsruhe Institute of Technology
- Bubbles and precipitates are formed in the 20nm narrow area on the grain boundry
100nm
100nm
17
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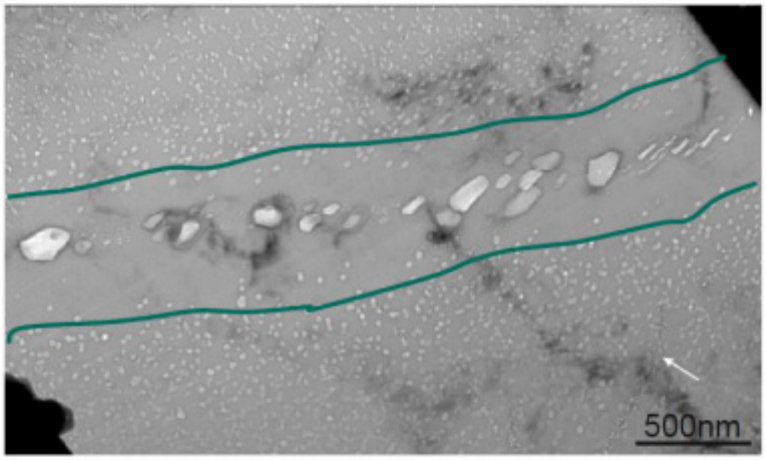
Microstructure of the grain boundary 440°C

- Formation of large bubbles on the grain boundaries
- Formation of bubble denuded zone



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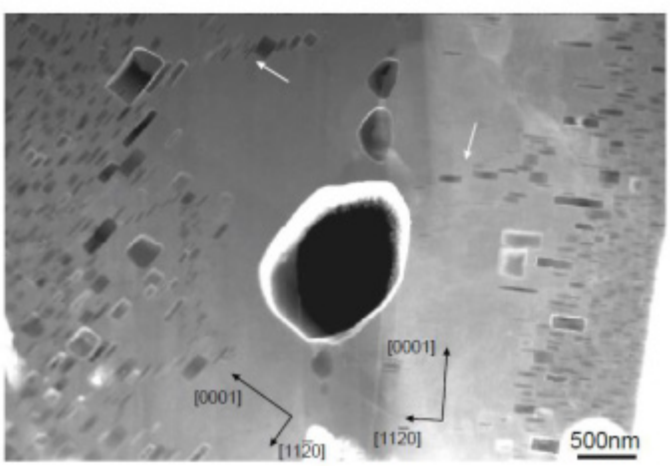
Microstructure of the grain boundary 650°C

- Formation of bubbles with up to 1µm size on the grain boundaries
- Formation of bubble denuded zone



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Detection of helium and hydrogen inside individual bubble in neutron irradiated beryllium

Transmission Electron Microscope
Talos F200X G2 / 200 kV FEG

TEM point resolution/information limit: 0.25 nm/ 0.12 nm, STEM resolution: 0.16 nm.

Equipment:

Ceta 16M camera with high speed option

HAADF, DF2, DF4 and BF STEM detectors.

Super-X EDS: high sensitivity and fast EDS mapping.

EELS: Gatan's Enfinium ER (977) spectrometer which includes UltraFast DualEELS spectrum imaging.

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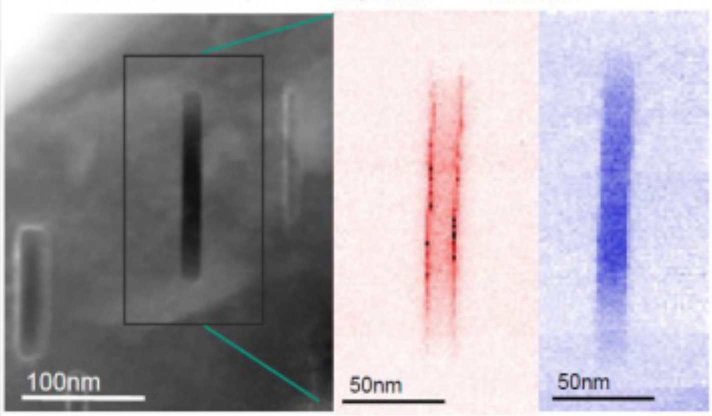
Detection of helium and hydrogen inside individual bubble in neutron irradiated beryllium

TEM dark field image $^3H_2$ signal He signal



Formation of beryllium hydride BeH_2 layers on internal walls He detection inside bubbles

Calculation of the gas pressure inside the individual bubbles

$$n_x = \frac{I_x}{I_z \cdot d \cdot \sigma_x}$$

n_x the number density of the gas

I_z and I_x are integrated intensities from the spectra

$\sigma_p(\Delta,\beta)$ is the partial inelastic cross-section for He/H

d is the bubble thickness in the direction of electron beam.

n_{He} =4.2±1.0 at/nm^3

n_{H2} =3.5±0.8 at/nm^3

$p_{(He+H2)}$=290±75 bar

M. Klimenkov Micron 127 (2019) 102754

Visit presentation of N. Zimber for more details

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Summary:

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- Microstructural changes induced by neutron irradiation in beryllium were studied by TEM.
- The bubbles inside grains have a shape like **thin disks** and they are located in basal planes of hexagonal beryllium
- The large bubbles and precipitates are formed on the grain boundaries
- The bubbles are filed with **helium - tritium gas** mixture,
- Tritium is mostly absorbed on the bubble internal walls

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Experimental Investigation of Radiation Damage Effects in Beryllium: Updates on Recent Results obtained on Proton, Neutron, and He-ions Irradiated Samples
V. Kuksenko (UKAEA)

Experimental investigation of radiation damage effects in beryllium: updates on recent results obtained on proton, neutron and He-ions irradiated samples

Viacheslav Kuksenko

United Kingdom Atomic Energy Authority, Culham Science Centre, Abingdon, OX14 3DB, United Kingdom

Beryllium is an essential material for reflectors and moderators in material testing nuclear reactors, plasma-facing material (JET, ITER) and potential neutron multiplier (DEMO) for fusion reactor designs, target components material in the currently running (NuMI) and near-future multi-megawatt accelerator particle sources (LBNF), and it is under extensive investigation by fission, fusion reactor, and proton accelerator facilities communities.

The paper gives an overview of the recent results obtained in the Materials Research Facility of the UKAEA on beryllium samples:

- exposed to high energy protons in the NuMI beamline at 50ºC (maximum dose 0.5 dpa, 2000appm of helium transmutant), and investigated within the international RaDIATE collaboration;
- after helium implantation at 50ºC and 200ºC (0.1dpa, 2000appm of helium);
- after neutron irradiation at different DEMO relevant conditions during HIDOBE-2 campaign in the HFR and investigated within the collaboration with Karlsruhe Institute of Technology (Germany).

The main part of the paper will be devoted to the micromechanical tests results obtained using nanoindentation hardness measurements and microcantilevers fracture tests. The local properties data will be analyzed in combination with observed microstructural changes. The paper will also give an overview of future plans.

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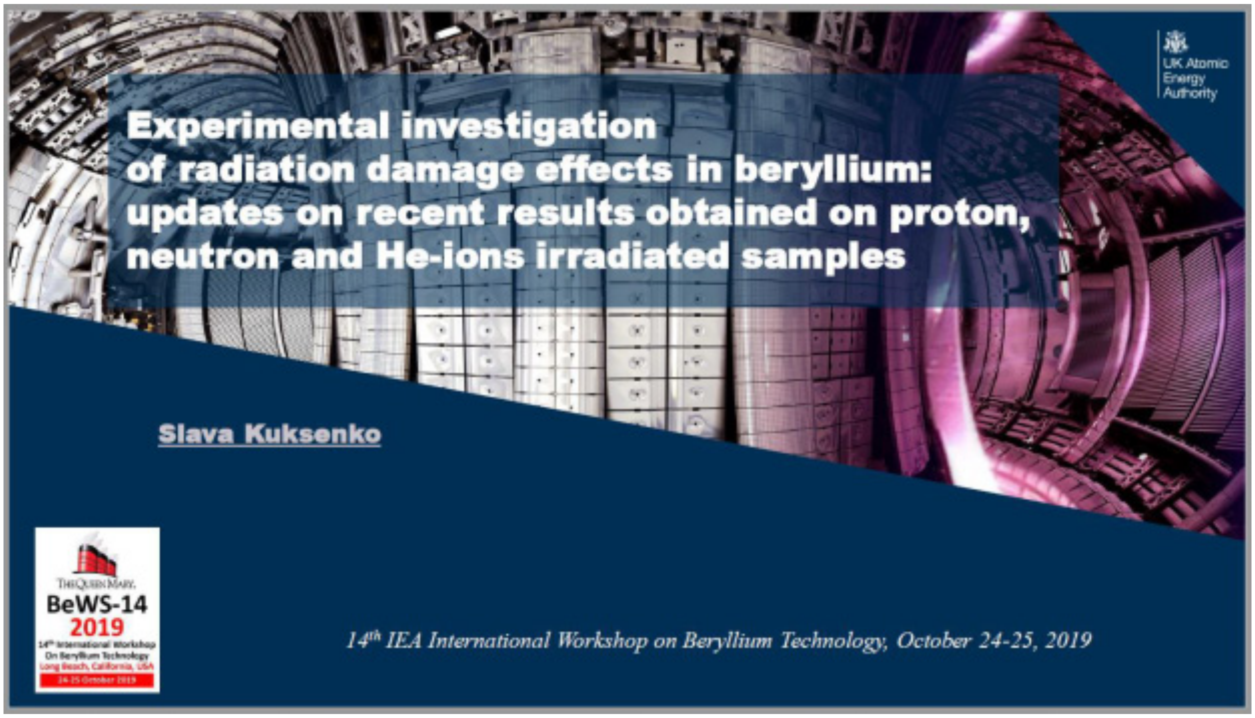
Abingdon, OX14 3DB
UNITED KINGDOM



Be
Beryllium

Beryllium has a unique combination of mechanical and physical properties:

- High strength
- Low density
- Low nuclear interaction cross-section (so low heat load)
- High heat capacity
- Thermal conductivity
- High melting point


First Wall of the JET tokamak


Primary beam window of the NuMI neutrino source

Beryllium is of particular importance for nuclear application:

- first wall material in JET experiment and the currently constructed International Thermonuclear Experimental Reactor (ITER)
- neutron multiplier candidate material for tritium breeding blanket of the future DEMO fusion reactor
- Reflector and moderation in research reactors and spallation neutron sources
- as beam windows and target components material in the next generation of neutrino sources


Be reflector in the ISIS Neutron Source (TS2)

2 |

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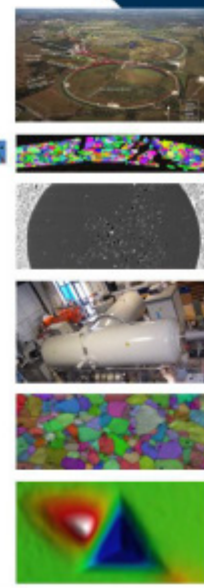
Outline

- Effects of the high energy protons in the NuMI beamline at 50°C;
- Effects of helium implantation at 50°C and 200°C;
- Effects of neutron irradiation at different DEMO relevant conditions during HIDOBE-2 campaign in the HFR
- Thermal shock effects is beryllium
- Investigation of correlation between neutronic performance of beryllium and its microstructure and chemical composition



3 |

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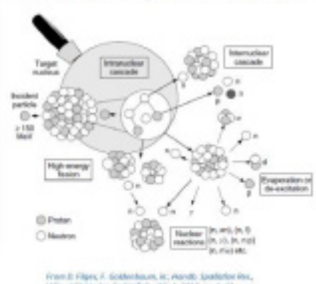
Be Beryllium

Beryllium is among very few candidate materials for beam windows and target components in proton accelerator driven particle sources



The NuMI Facility
"Neutrinos (ν – Nu) at the Main Injector":
Intense muon-neutrino beam directed towards Minnesota

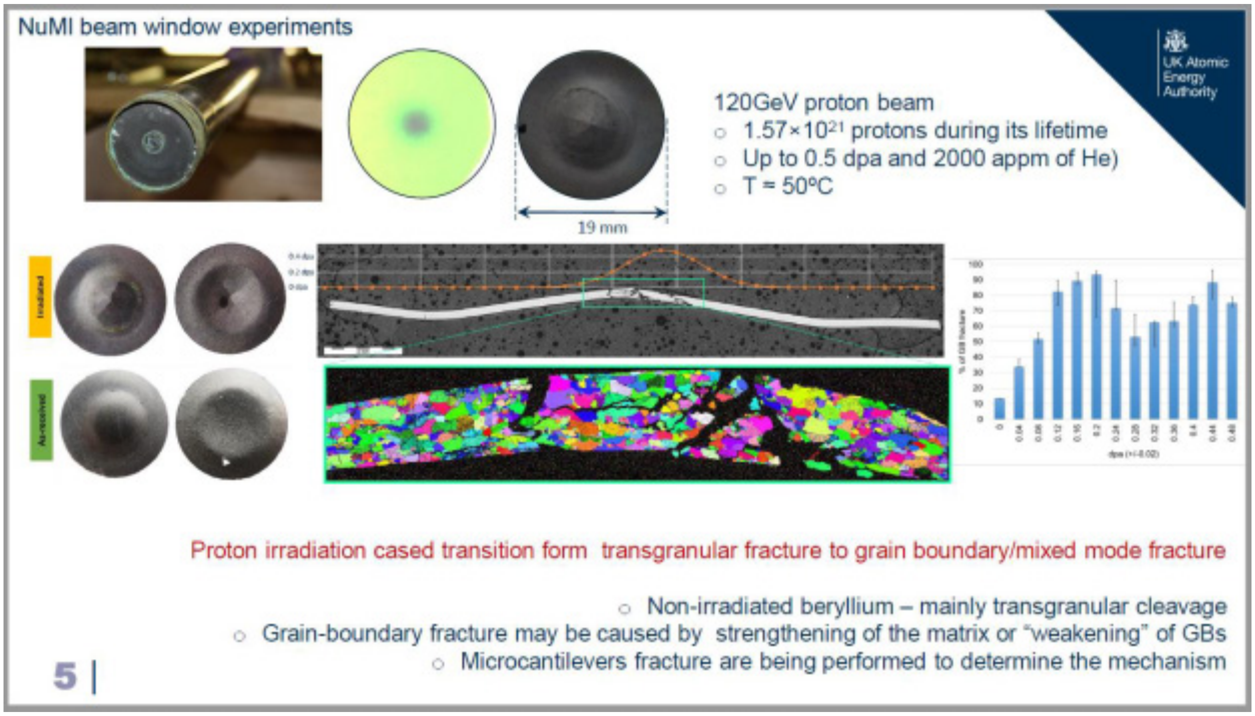
	Approximate He production in beryllium, appm/dpa
SM-3 high-flux reactor (Russia)	330
BOR-60 reactor (Russia)	280
HFR, HIDOBE-01 irradiation campaign (Petten, Netherlands)	160
Beryllium reflectors in the ISIS neutron source (RAL, UK)	220 (TS1)
	110 (TS2)
DEMO fusion reactor	700
NuMI beam window (FNAL, USA)	4000

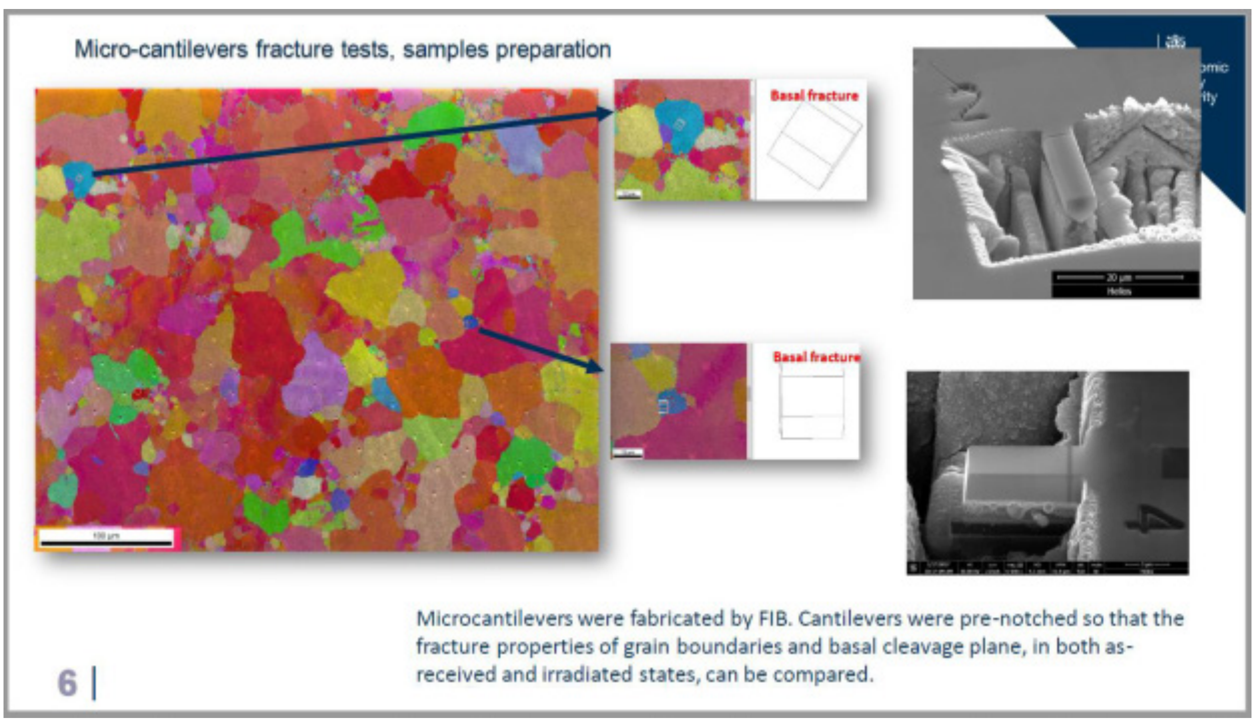
Application	Operating conditions			
	Avg. T (°C)	Peak T(°C)	Total DPA	He appm
Beam window NuMI	50	<100	~ 0.08/yr	~ 320/yr
Beam window LBNF	200	300	~ 0.23/yr	~ 920/yr
Target LBNF	375	450	~ 0.23/yr	~ 920/yr

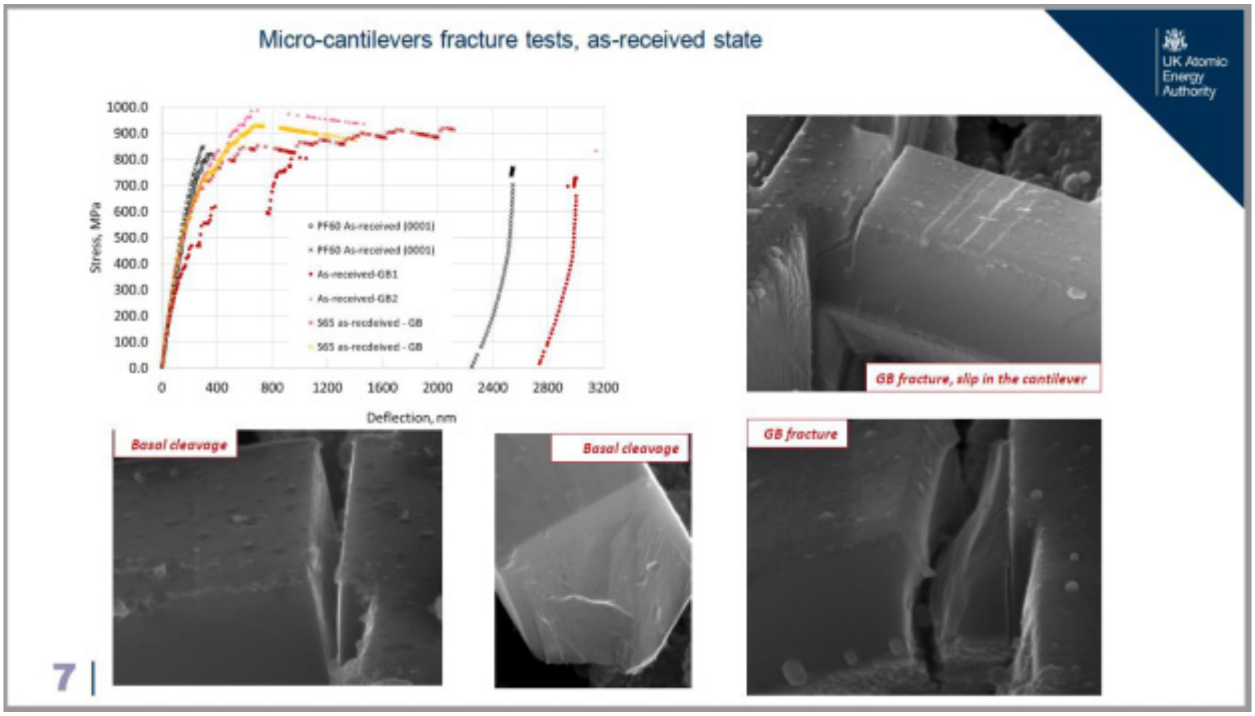
RaDIATE

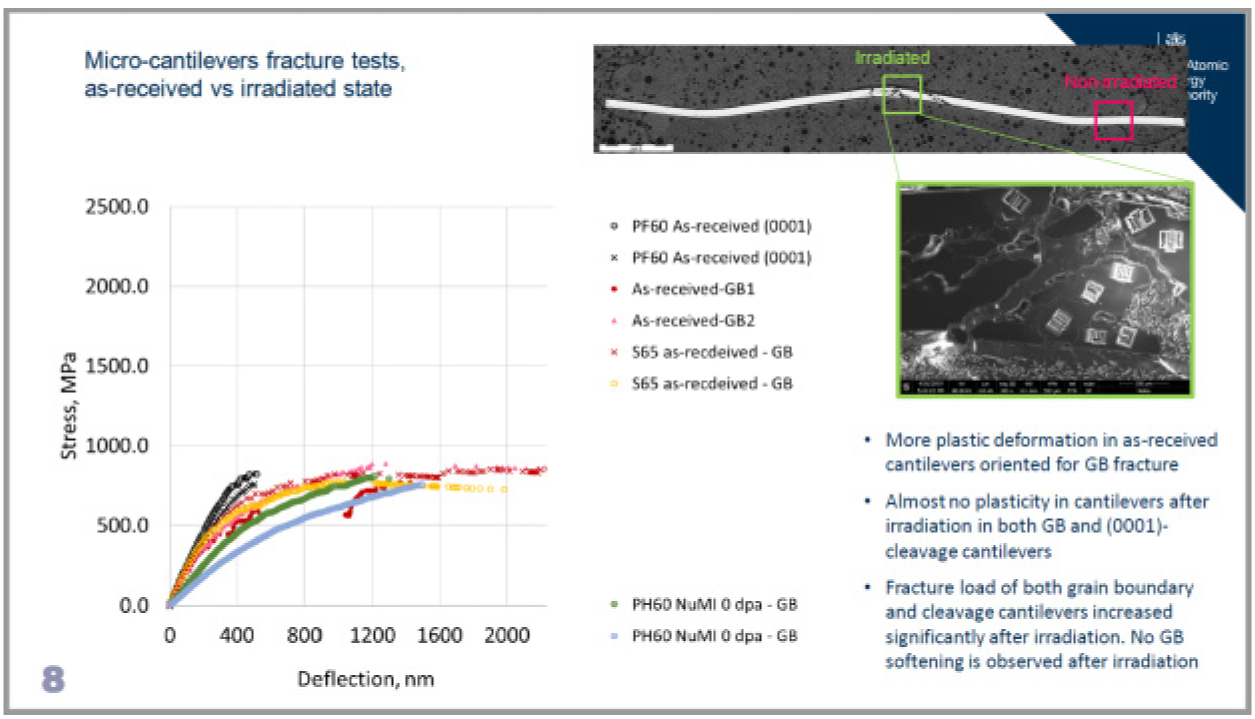
To limit the risk of components failure originating from radiation-induced changes of the material, scientifically based lifetime limits should be developed for existing target systems and future facilities.

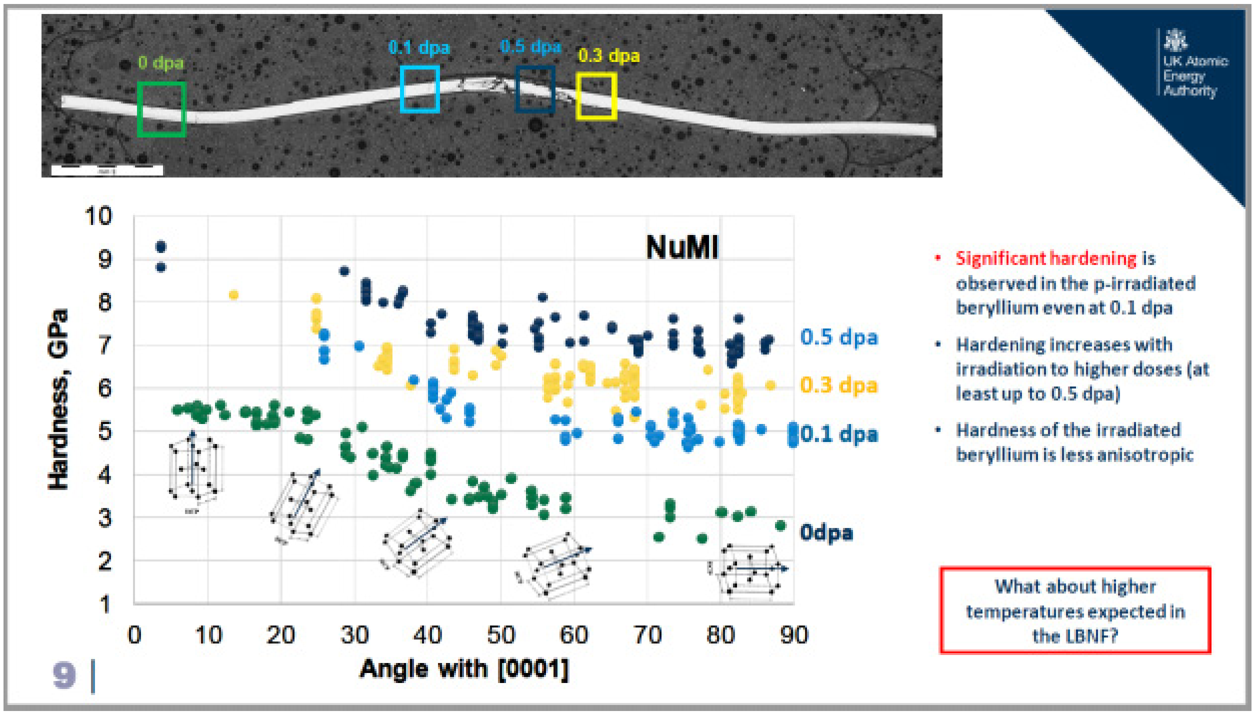
4 |

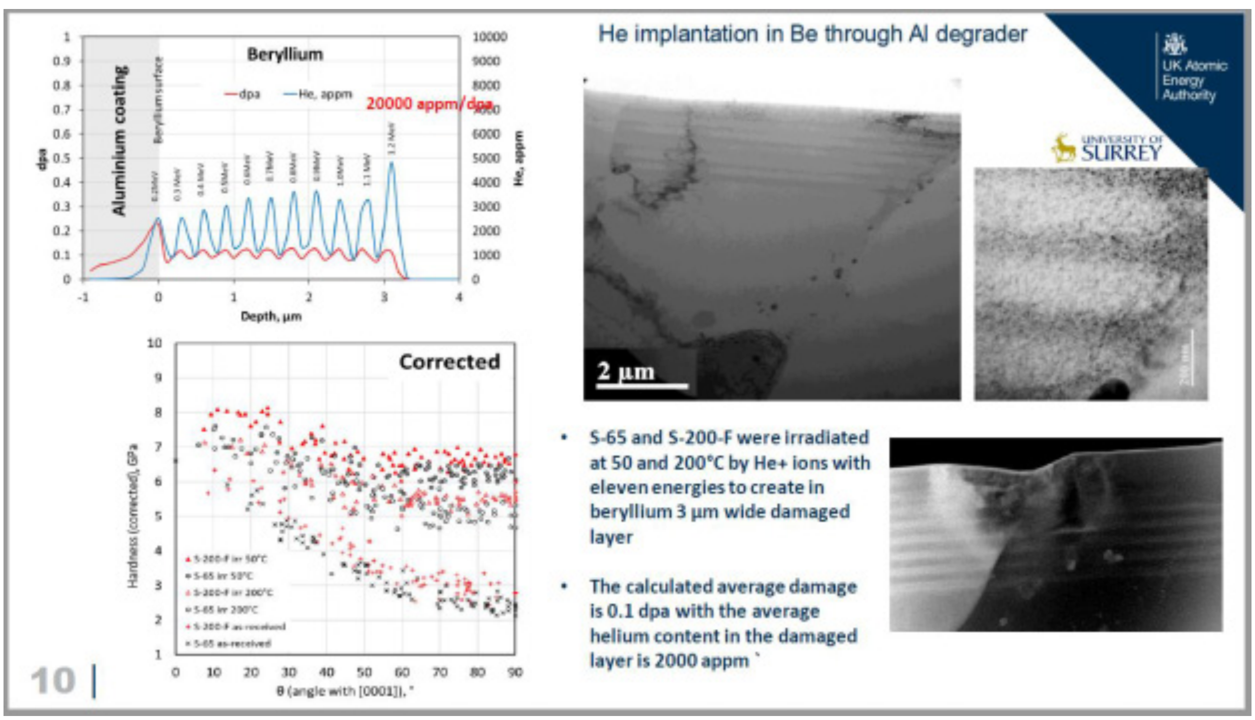

NuMI beam window experiments
UK Atomic Energy Authority
120GeV proton beam
1.57×10²¹ protons during its lifetime
Up to 0.5 dpa and 2000 appm of He)
T = 50ºC
19 mm
Irradiated
As-received
% of GB fracture
dpa (+/-0.02)
Proton irradiation cased transition form transgranular fracture to grain boundary/mixed mode fracture
Non-irradiated beryllium – mainly transgranular cleavage
Grain-boundary fracture may be caused by strengthening of the matrix or "weakening" of GBs
Microcantilevers fracture are being performed to determine the mechanism
5 |

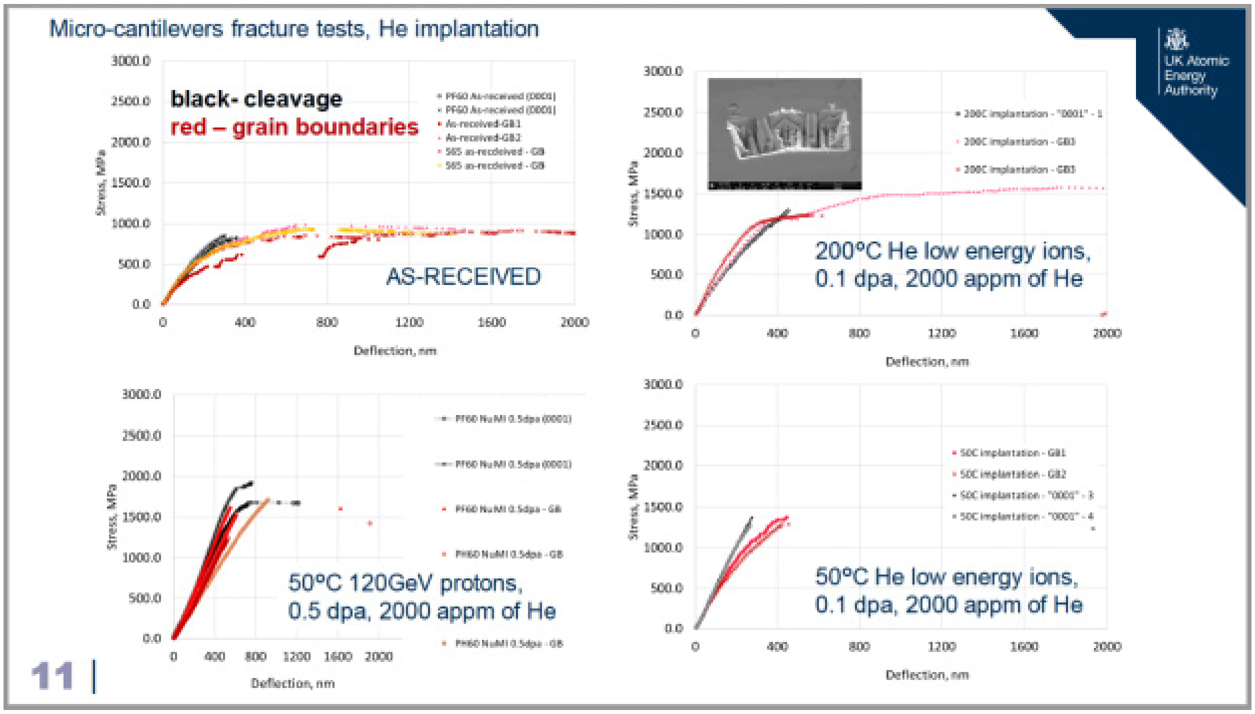

Micro-cantilevers fracture tests, samples preparation
Basal fracture
Basal fracture
20 µm
Helios
100 µm
Microcantilevers were fabricated by FIB. Cantilevers were pre-notched so that the fracture properties of grain boundaries and basal cleavage plane, in both as-received and irradiated states, can be compared.
6 |


Micro-cantilevers fracture tests, as-received state
UK Atomic Energy Authority
Stress, MPa
Deflection, nm
PF60 As-received (0001)
PF60 As-received (0001)
As-received-GB1
As-received-GB2
S65 as-recdeived - GB
S65 as-recdeived - GB
GB fracture, slip in the cantilever
Basal cleavage
Basal cleavage
GB fracture
7


Micro-cantilevers fracture tests,
as-received vs irradiated state
Irradiated
Non-irradiated
Stress, MPa
Deflection, nm
PF60 As-received (0001)
PF60 As-received (0001)
As-received-GB1
As-received-GB2
S65 as-recdeived - GB
S65 as-recdeived - GB
PH60 NuMI 0 dpa - GB
PH60 NuMI 0 dpa - GB
• More plastic deformation in as-received cantilevers oriented for GB fracture
• Almost no plasticity in cantilevers after irradiation in both GB and (0001)-cleavage cantilevers
• Fracture load of both grain boundary and cleavage cantilevers increased significantly after irradiation. No GB softening is observed after irradiation
8


0 dpa
0.1 dpa
0.5 dpa
0.3 dpa
UK Atomic Energy Authority
NuMI
Hardness, GPa
Angle with [0001]
0.5 dpa
0.3 dpa
0.1 dpa
0dpa
• Significant hardening is observed in the p-irradiated beryllium even at 0.1 dpa
• Hardening increases with irradiation to higher doses (at least up to 0.5 dpa)
• Hardness of the irradiated beryllium is less anisotropic
What about higher temperatures expected in the LBNF?
9


Beryllium
Aluminium coating
Beryllium surface
dpa
He, appm
20000 appm/dpa
Depth, μm
Corrected
Hardness (corrected), GPa
θ (angle with [0001]), °
He implantation in Be through Al degrader
UK Atomic Energy Authority
UNIVERSITY OF SURREY
2 μm
• S-65 and S-200-F were irradiated at 50 and 200°C by He+ ions with eleven energies to create in beryllium 3 μm wide damaged layer
• The calculated average damage is 0.1 dpa with the average helium content in the damaged layer is 2000 appm
10



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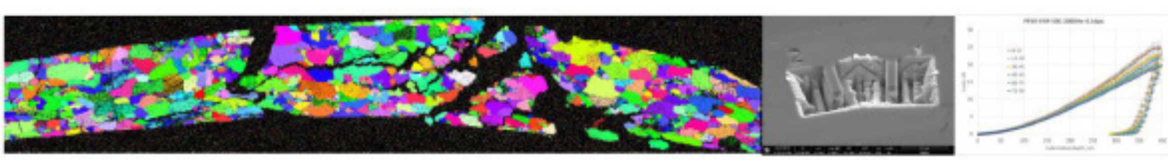
Conclusions

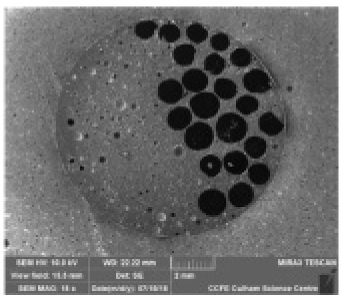
1. Radiation induces significant hardening and fracture mechanism change of beryllium even at 0.1 dpa
2. Irradiation at 200°C leads to lower hardening.
3. 50°C irradiation (both with protons and helium ions) leads to complete loss of ductility pre-notched cantilevers. Some ductility before fracture is retained in the cantilevers from the beryllium irradiated at 200°C



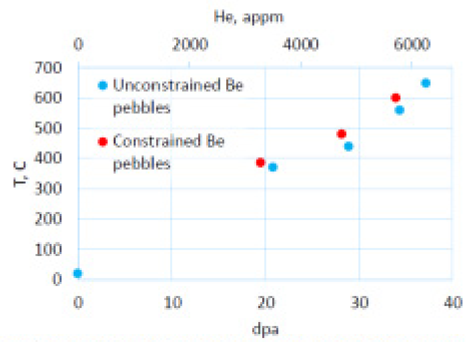
12 |

Irradiation parameters for HIDOBE-02

Name	T irr, C	dpa	He, appm	T, appm
6bPC2	600	34	5557	600
6mPC1	480	28.2	4788	508
5mPC1	387	19.5	3638	369
49sPC	370	20.8	3632	367
44sPC	440	28.9	4751	502
8sPC	560	34.4	5524	596
7sPC	650	37.2	5925	644
1473-10	20	-	-	-

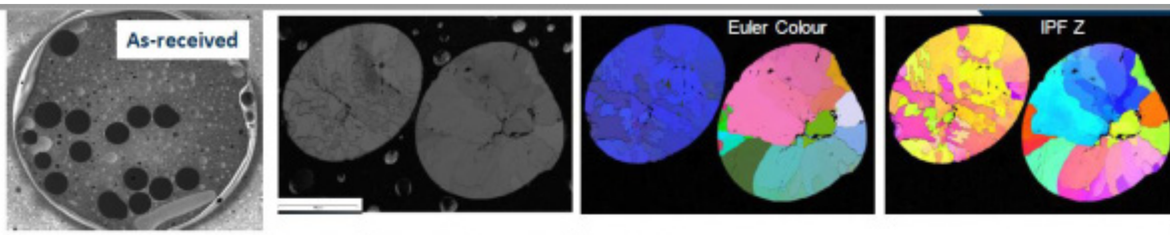


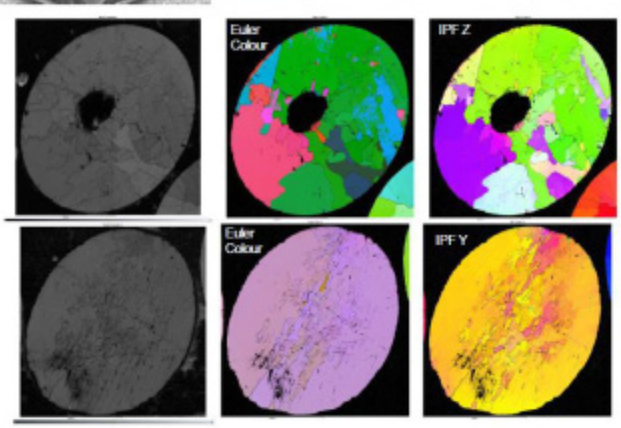
Collaboration with
P. Vladimirov, V. Chakin, N. Zimbler, M. Klimenkov, R. Gaslin, KIT Hot lab team and others in KIT



- 7 samples irradiated samples from the HIDOBE-2 campaign were received from KIT.
- Every sample has from 8 to 23 pebbles cross-sections polished in KIT to "EBSD quality"
- Lift-outs were made for detailed TEM analysis at KIT (see presentation of Nikolai Zimber)

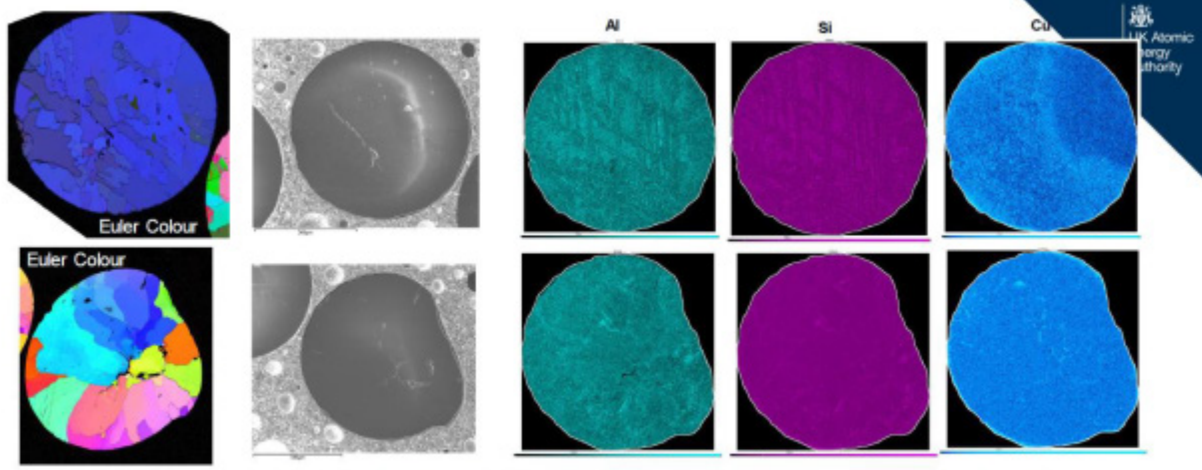
13 |





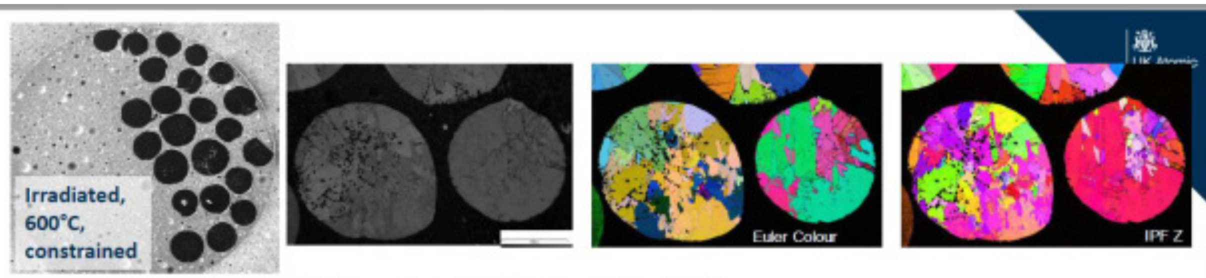
- **All pebbles have pores, some have cracks**
- **Some of the pebbles have dominant shrinkage pore**
- **Some of the pebbles are polycrystalline, some of them are "almost" single grains (at least in the investigated cross-section) and have only low-angle grain-boundaries**

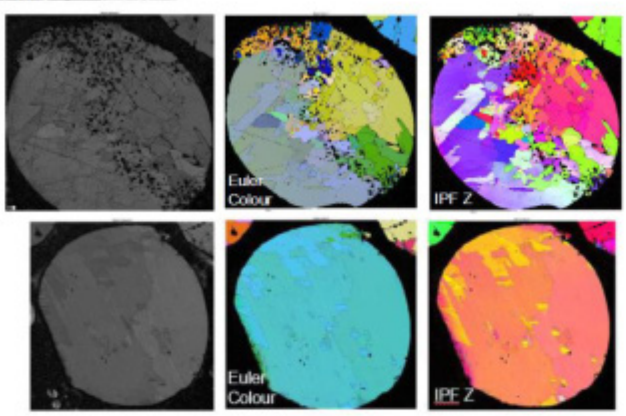
14 |



- O, C, Al, Si, P, Fe, Mg, Mn, Ni, Cu, Ti, Ca, Cr were identified. Majority of the impurities are non-homogeneously distributed
- In "polycrystalline" pebbles Al and Si have lineal curved segregation including at GB, in "single grain" pebbles these species have "needle-like" distribution
- Samples have coper-rich areas. Coper also precipitates

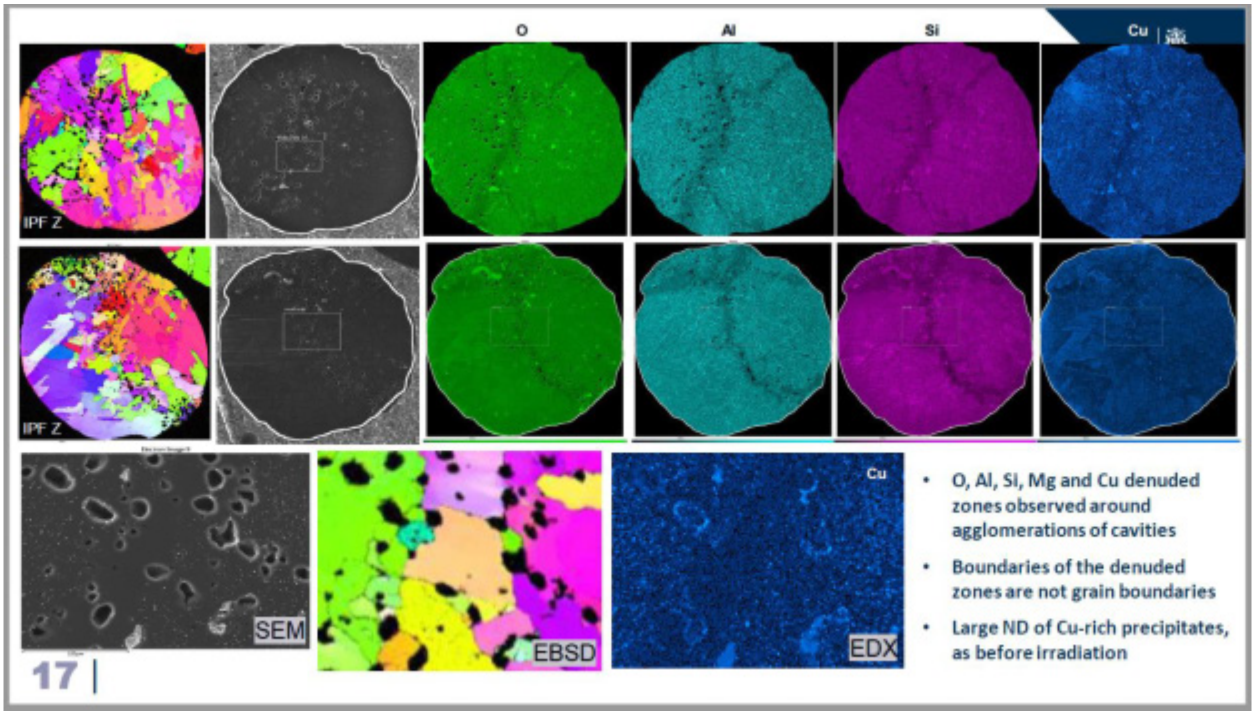
15 |





- Some pebbles have large number of non-homogeneously distributed cavities – this was not observed before irradiation
- Agglomeration of cavities is around high angle grain boundaries
- Some pebbles are still almost free of cavities – as many pebbles before irradiation

16 |


O
Al
Si
Cu
IPF Z
IPF Z
SEM
EBSD
Cu
EDX
• O, Al, Si, Mg and Cu denuded zones observed around agglomerations of cavities
• Boundaries of the denuded zones are not grain boundaries
• Large ND of Cu-rich precipitates, as before irradiation
17 |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No conclusions yet
Thank you!
18 |

Investigation of high-dose irradiated beryllium microstructure including STEM-EELS analysis of Helium/Tritium bubbles

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Beryllium is considered as a plasma-facing material as well as an effective neutron multiplier material for the Helium-Cooled Pebble-Bed (HCPB) design concept of the tritium-breeding blanket being developed at the Karlsruhe Institute of Technology (KIT). Large amounts of helium and tritium will be generated by neutron-induced transmutation within beryllium. Accumulation of tritium within 300 tons of beryllium pebbles required for the demonstration fusion reactor DEMO could imply severe safety and waste processing issues.

In order to assess the tritium inventory, a fundamental understating of the microstructure developing during neutron irradiation is necessary. This work presents detailed analytical transmission electron microscopy (TEM) studies of the microstructural changes in beryllium after the high-dose irradiation program (HIDOBE-02). The investigated beryllium samples were constrained into a containment and irradiated up to a displacement damage of 34dpa, a helium concentration of 5500appm and a tritium content of 600appm at 387°C, 480°C, and 600°C.

The microstructural investigations revealed the formation of zones denuded from bubbles along grain boundaries (GB), whereby their width is increasing with increasing temperature up to a size of several microns at 600°C. In addition, chains of bubbles were found within the denuded zones at various locations. We suggest alignment of these chains along dislocation lines, which are providing thermal vacancies for bubble growth. Otherwise, this observation would contradict the general assumption, that the vacancy-concentration in the direct vicinity of GBs is not sufficient for the formation of bubbles.

For the first time, the co-existence of helium and tritium inside several gas bubbles located in the grain interior as well as at GBs was confirmed using electron energy-loss spectroscopy (EELS). In addition, we have shown that in hexagonal bubbles tritium predominantly occupies the basal plane, which is in good accordance with theoretical considerations. These observations underline the assumption that tritium is trapped directly inside the bubbles located within single grains and at their grain boundaries even at elevated irradiation temperatures. The results are used to quantify the amount of tritium within bubbles, i.e. to determine the He/^{3}H-ratio, which is crucial in order to understand the interaction mechanisms between beryllium and tritium.

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Tritium Release and Retention Behavior of Beryllium and Titanium Beryllide Irradiated up to High-Neutron Doses
V. Chakin (KIT, Germany) et al.

Tritium release and retention behavior of beryllium and titanium beryllide irradiated up to high neutron doses

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Beryllium pebbles with 1mm diameter will be used as neutron multiplier in the Helium-Cooled Pebble Bed (HCPB) breeding blanket of DEMO. Accumulation of helium in beryllium causes formation of helium bubbles that are the structural traps for tritium. Beta-radioactive tritium complicates handling of radioactive beryllium waste. Tritium and helium release and retention behavior of irradiated beryllium is an essential point on the safety reason. High-dose neutron irradiation tests in nuclear material testing reactors at relevant irradiation parameters allow to predict to some extent the tritium behavior in beryllium in the DEMO blanket conditions.

The HIDOBE-2 experiment in the High Flux Reactor (HFR) included irradiation of beryllium pebbles with a diameter of 1mm fabricated by the rotating-electrode method (REM) at temperatures of 370, 440, 560, 650°C and up to 367, 502, 596, 644appm tritium and 3632, 4751, 5524, 5925 helium productions, corresponding to 21, 29, 34, 37dpa damage dose, as well as pellets with a diameter of 8mm and thickness of 2mm from beryllium and titanium beryllide at temperatures of 440, 525, 664, 768°C and up to 430, 550, 625, 653appm tritium and 4144, 5142, 5757, 5992appm helium productions, corresponding to 23, 31, 36, 38dpa damage doses, respectively. The post-irradiation examination (PIE) included the temperature-programmed desorption (TPD) tests using a flow-through setup with a quadrupole mass-spectrometer (QMS) and an ionization chamber (IC). The gas mixture of Ar+0.1 vol. % H_2 was used as a purge gas to transport the species released under the permanent heating modes having constant rates of 1 and 7°C/min. The heating of pebbles and pellets was performed from room temperature up to 1100°C with a final exposure at the maximum temperature for 3h.

As a rule, the tritium and helium release curves have only one release peak. The helium release peak is slightly shifted to higher testing temperatures relative to that of tritium peak. At the higher heating rate of 7°C/min, the tritium and helium release peaks are always manifested at higher temperatures as for 1°C/min. In any case, all tritium and helium release peaks cover the temperature region of 850-1050°C. The total tritium release, called in our case as tritium retention, decreases on increasing irradiation temperature. Tritium retention in titanium beryllide at three highest irradiation temperatures is significantly lower than that in beryllium. In particular, at temperatures of 664 and 768°C, the tritium retention in titanium beryllide is close to zero, while in beryllium this reaches up to 3000MBq/(g-s).

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Tritium release and retention behavior of beryllium and titanium beryllide irradiated up to high neutron doses

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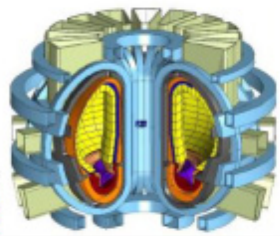


Outline

- Helium Coold Pebble Bed (HCPB) blanket of DEMO
- Materials, experimental
- Thermal-programmed desorption (TPD) of tritium/helium from irradiated Be pebbles and Be7Ti pellets
- Microstructure of irradiated Be and Be7Ti pellets
- Conclusions

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4th IEA International Workshop on Beryllium Technology for Fusion, September 15-17, 1999, Karlsruhe, Germany
1999-2019
BeWS-4 BeWS-14
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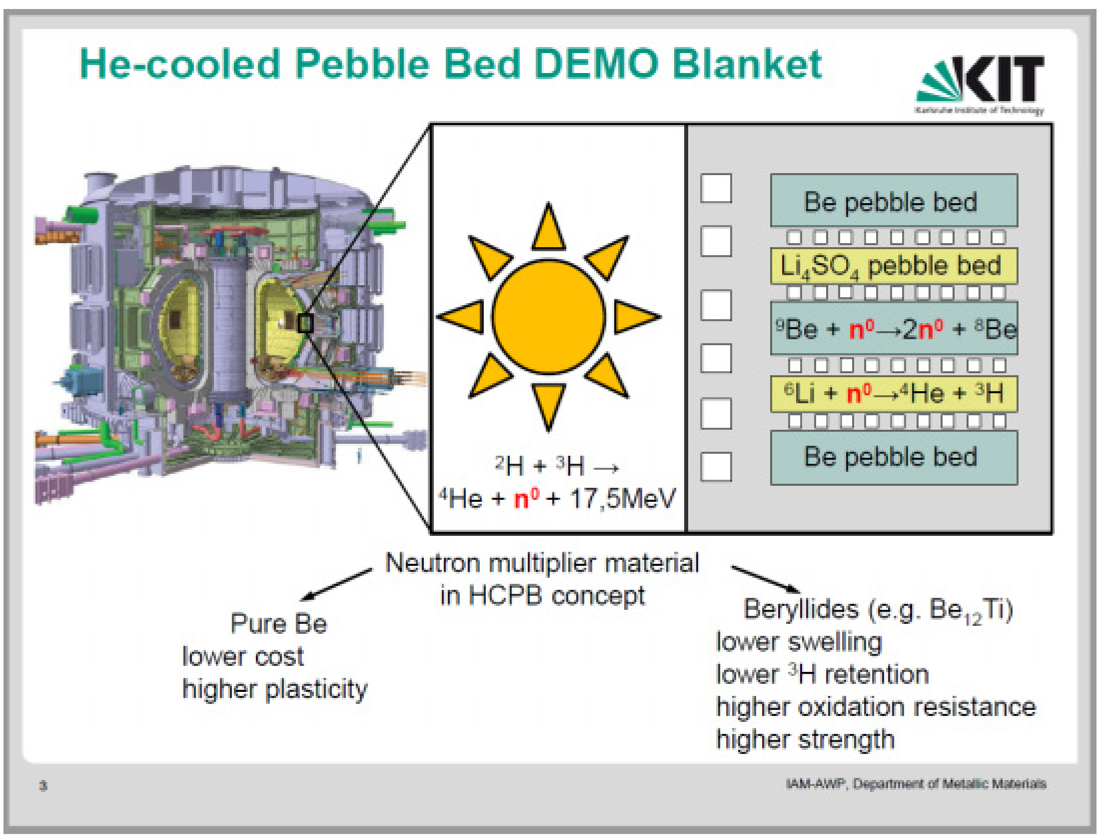
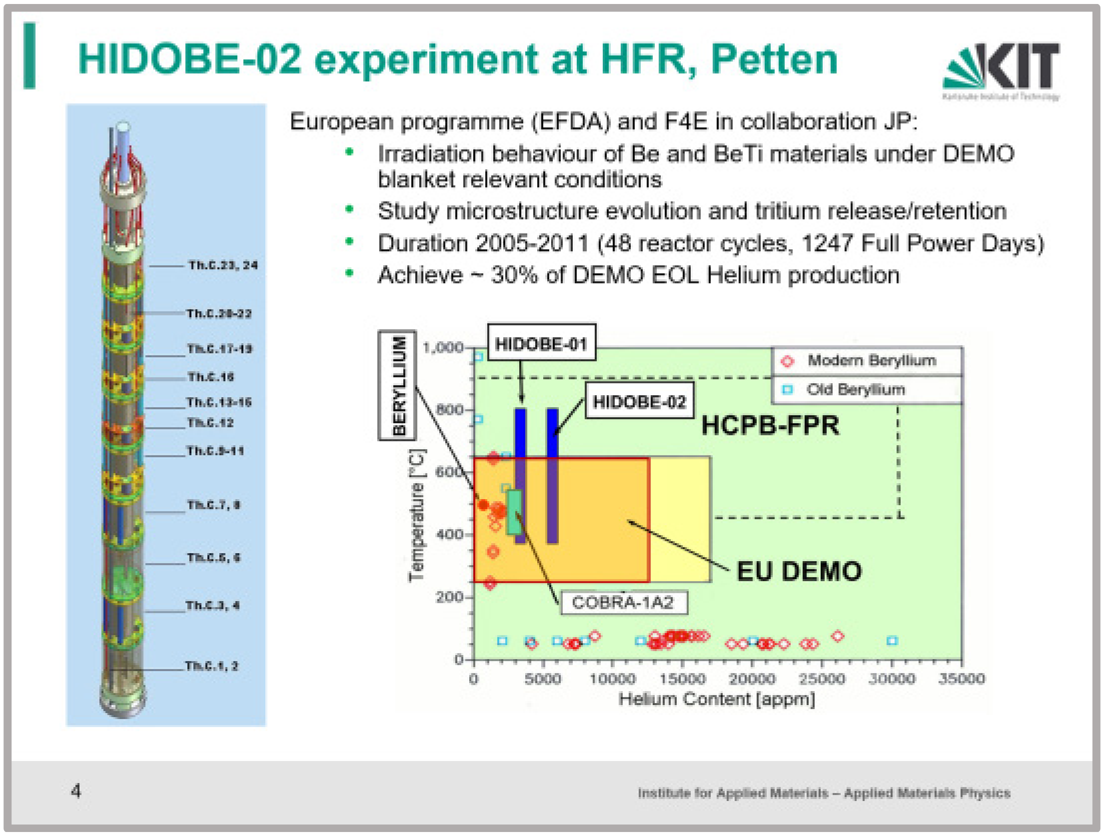

He-cooled Pebble Bed DEMO Blanket
Be pebble bed
Li_4SO_4 pebble bed
$^{9}Be + n^{0} \rightarrow 2n^{0} + {}^{8}Be$
$^{6}Li + n^{0} \rightarrow {}^{4}He + {}^{3}H$
Be pebble bed
$^{2}H + {}^{3}H \rightarrow$
$^{4}He + n^{0} + 17{,}5MeV$
Neutron multiplier material in HCPB concept
Pure Be
lower cost
higher plasticity
Beryllides (e.g. $Be_{12}Ti$)
lower swelling
lower ^{3}H retention
higher oxidation resistance
higher strength
3
IAM-AWP, Department of Metallic Materials


HIDOBE-02 experiment at HFR, Petten
KIT
European programme (EFDA) and F4E in collaboration JP:
Irradiation behaviour of Be and BeTi materials under DEMO blanket relevant conditions
Study microstructure evolution and tritium release/retention
Duration 2005-2011 (48 reactor cycles, 1247 Full Power Days)
Achieve ~ 30% of DEMO EOL Helium production
Th.C.23, 24
Th.C.20-22
Th.C.17-19
Th.C.16
Th.C.13-15
Th.C.12
Th.C.9-11
Th.C.7, 8
Th.C.5, 6
Th.C.3, 4
Th.C.1, 2
BERYLLIUM
HIDOBE-01
HIDOBE-02
Modern Beryllium
Old Beryllium
HCPB-FPR
EU DEMO
COBRA-1A2
Temperature [°C]
Helium Content [appm]
0
200
400
600
800
1,000
5000
10000
15000
20000
25000
30000
35000
4
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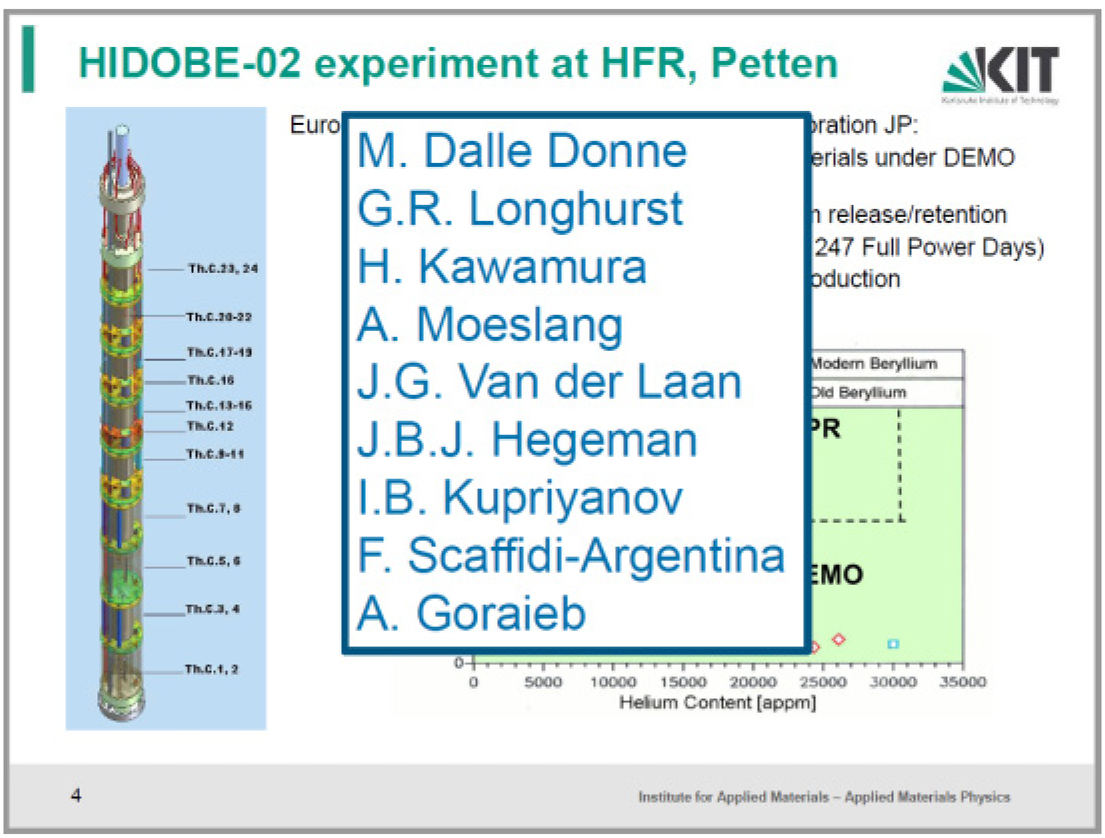
HIDOBE-02 experiment at HFR, Petten
KIT
M. Dalle Donne
G.R. Longhurst
H. Kawamura
A. Moeslang
J.G. Van der Laan
J.B.J. Hegeman
I.B. Kupriyanov
F. Scaffidi-Argentina
A. Goraieb
Helium Content [appm]
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Irradiation parameters of Be and Be7Ti pellets in HIDOBE-02 at HFR, Petten

Sample	T_{irr}, K	F, $\times 10^{26}$ m^{-2}, E>1 MeV	D, dpa	^4He, appm	^3H, appm
Be Be7Ti	710	1.16	23	4144	430
	800	1.51	31	5142	550
	940	1.73	36	5757	625
	1040	1.82	38	5992	653

Be and Be7Ti pellets with sizes of Ø8×2 mm manufactured by vacuum casting were irradiated up to unique (!) damage doses and helium/tritium productions within HIDOBE-02 experiment.

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Irradiation parameters of Be pebbles from HIDOBE-02

Be pebble	T_{irr} , K	F, $\times 10^{26}$, m^{-2}, E>1MeV	D, dpa	^4He, appm	^3H, appm
Ø0.5 mm	643	1.06	21	3632	367
	723	1.43	29	4751	502
	833	1.68	34	5524	596
	923	1.81	37	5925	644
Ø1 mm	643	1.06	21	3632	367
	723	1.43	29	4751	502
	833	1.68	34	5524	596
	923	1.81	37	5925	644

Be pebbles produced by REM were irradiated at irradiation temperatures 643-923 K, damage doses 21-37 dpa, up to 6000 appm ^4He and 640 appm ^3H productions, respectively.

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Chemical composition of Be pebbles and pellets, and Be7.7at.%Ti pellets

Pellets Ø 8×2 mm

Sample	Be	BeO	Ti	Fe	Al	Mg	Si	Cr	Co	Ni	Mn	U
Be	99.5	0.02	-	0.1	0.07	0.05	0.03	<0.01	0.0003	0.01	0.007	0.0069
Be7Ti	71.2	0.27	28.5	0.031	0.06	<0.001	0.033	0.005	<0.001	0.003	0.007	0.0041

Pebbles Ø0.5 mm and Ø1 mm

Element	Content, wt.%
Be	99.5
BeO	0.36
Fe	0.094
Al	0.048
Mg	0.024
Si	0.029
U	<0.01

Be7at.%Ti pellets with sizes of Ø8×2 mm contain 28.5wt.% Ti as well as 0.041 wt.% U.

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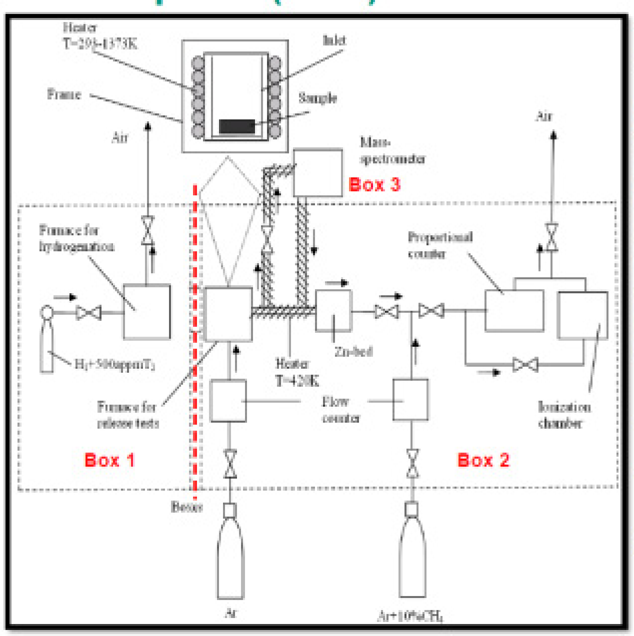
Set-up for Temperature-Programmed Desorption (TPD) tests



The set-up for TPD tests is located in three glove boxes filled by pure nitrogen:

Box 1: loading system (furnace with crucible for testing sample, gas balloon with H_2+500appm T_2 loading gas)
Box 2: TPD facility (furnace with crucible for testing sample, pipelines, Zn-bed, proportional (PC) and ionization (IC) chambers
Box 3: quadrupole mass-spectrometer (QMS) with two heads (1-6AMU, 1-100AMU)

Testing parameters for TPD tests:

- heating rate 1K/min (0.0167 K/s) and 7K/min (0.1167 K/s) ;
- temperature increase up to T_{max}=1100°C with exposure at T_{max} for 3 h;
- purge gas Ar+0.1%H_2;
- synchronized measurements of tritium and helium release by IC and QMS, respectively.

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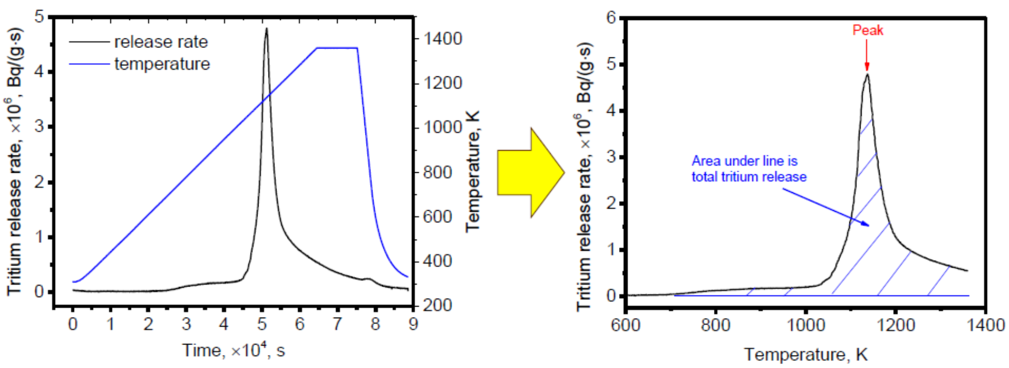
Be pebbles

TPD tests of irradiated Be pebbles: ^3H release

Be pebbles with Ø1 mm irradiated at T_{irr} = 923 K and tested at 1 K/min (0.0167 K/s)



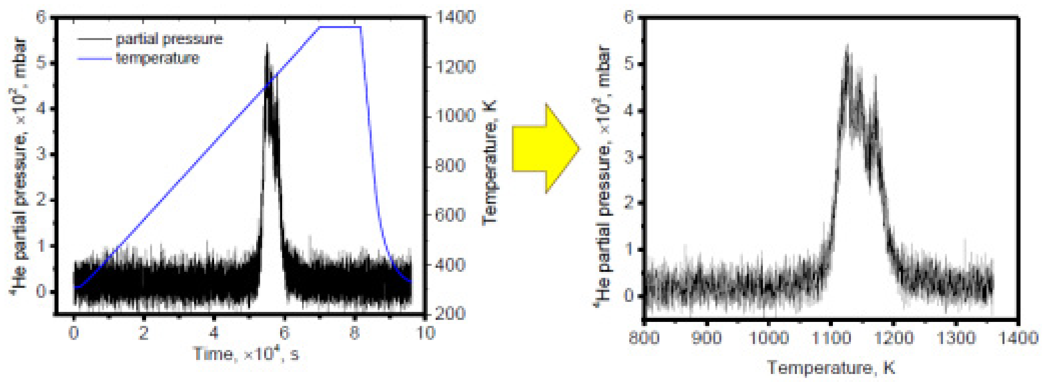
The released tritium activity rate is measured by IC in Bq/(g·s).

Only one peak was detected during each TPD test for both tritium and helium release.

It is more suitable to use TPD spectra with zwo axes, tritium release rate and testing temperature.

TPD tests of irradiated Be pebbles: ^{4}He release

Be pebbles with Ø1 mm irradiated at T_{irr} = 923 K and tested at 1 K/min (0.0167 K/s)



^{4}He partial pressure is measured during TPD tests by QMS.

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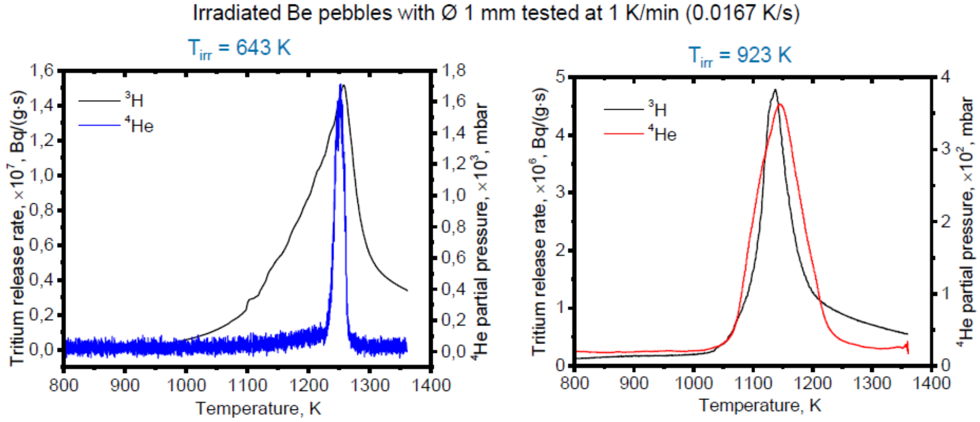
Mutual location of ^{3}H/^{4}He release peaks

Irradiated Be pebbles with Ø 1 mm tested at 1 K/min (0.0167 K/s)



The ^{3}H and ^{4}He peaks are mainly located close each to other on testing temperature scale.

The tritium release starts at comparatively lower temperatures for lower irradiation temperatures.

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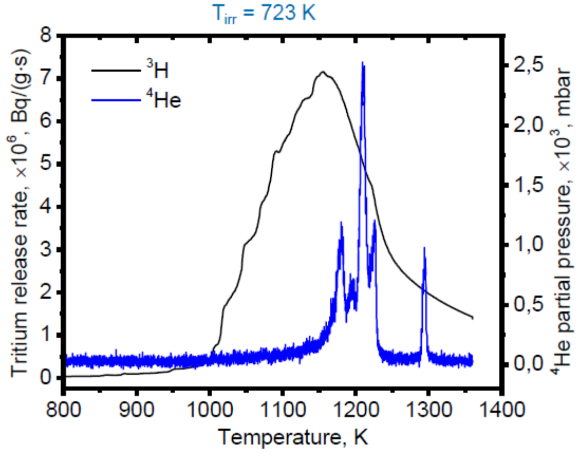
Mutual location of $^3H/^4He$ release peaks

Irradiated Be pebbles with Ø 0.5 mm tested at 1 K/min (0.0167 K/s)



During some TPD tests, the ^4He peaks are located at higher temperatures than the ^3H peaks.

This behaviour of tritium and helium atoms can be caused due to the comparatively higher diffusion mobility of helium atoms in the matrix during TPD tests.

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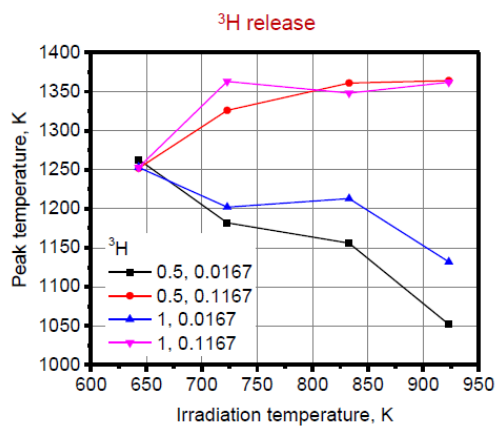
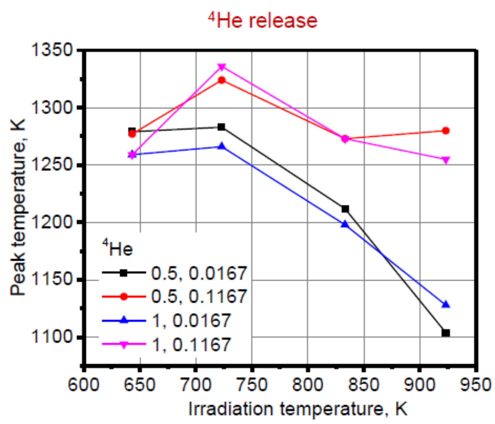
$^3H/^4He$ release peaks





For TPD tests with lower heating rate of 0.0167 K/s, the peak temperatures for both tritium and helium release decrease on increasing irradiation temperature.

For higher heating rate of 0.1167 K/s, this behaviour is unregular.

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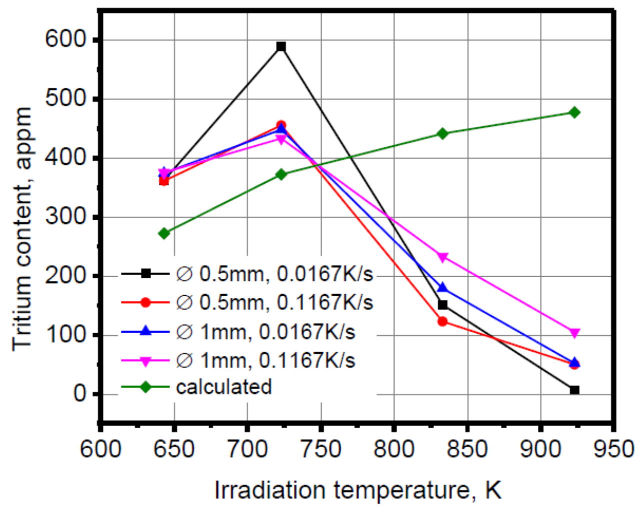
Total tritium release from irradiated Be pebbles



- Tritium remains completely in pebbles at T_{irr} = 643 K and 723 K. At T_{irr} = 833 K and 923 K significant amount of tritium leaves pebbles.
- The difference between the calculated and experimental values is explained by the inaccuracy of neutron-physical calculations.
- There are no fundamental differences in the tritium content depending on the pebble diameter and heating rate during TPD tests.

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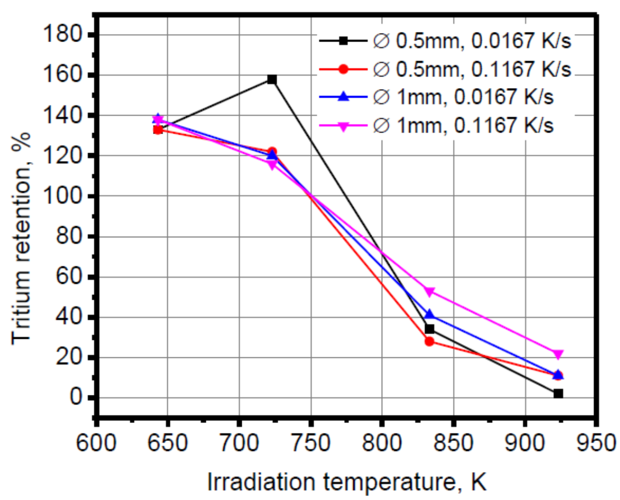
Tritium retention in Be pebbles during irradiation



- The similar behavior of tritium retention on increasing irradiation temperature occurs independent on the pebble diameter and the heating rate.
- The retention values are close each to other for different pebble diameters and the heating rates at each irradiation temperature.

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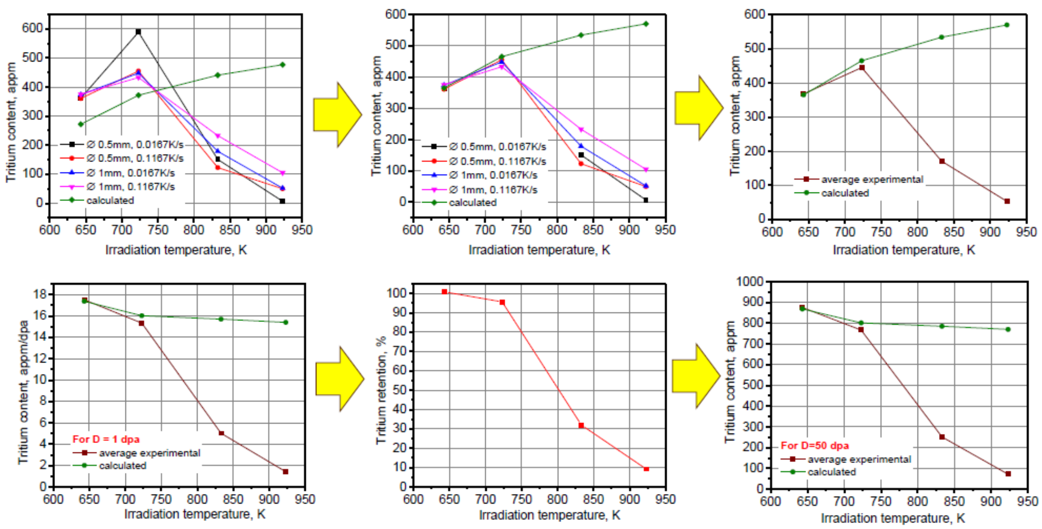
Tritium retention in Be pebbles during irradiation by approximation



It is possible to perform a rough estimation an average tritium inventory in Be pebbles after neutron irradiation in material testing reactors up to high neutron damage doses.

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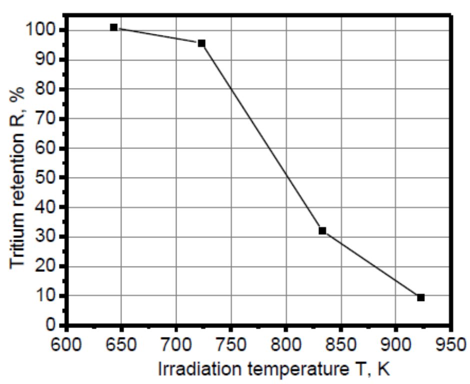
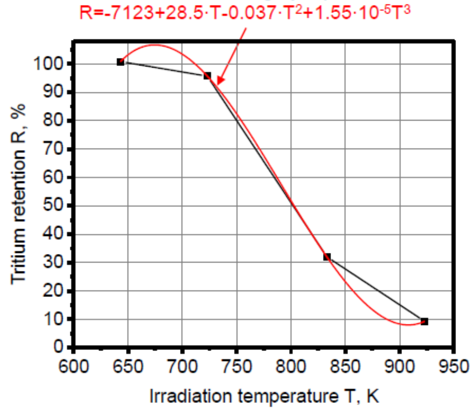
Fitting of tritium retention



Tritium retention fitting by equation
$R=-7123+28.5\cdot T-0.037\cdot T^2+1.55\cdot 10^{-5}T^3$



It is possible to estimate tritium inventory L in Be sample at an irradiation temperature T, if a maximum value of tritium production Q in the sample is known, by using equation:

$$L = (-7123+28.5\cdot T-0.037\cdot T^2+1.55\cdot 10^{-5}T^3)\times Q$$

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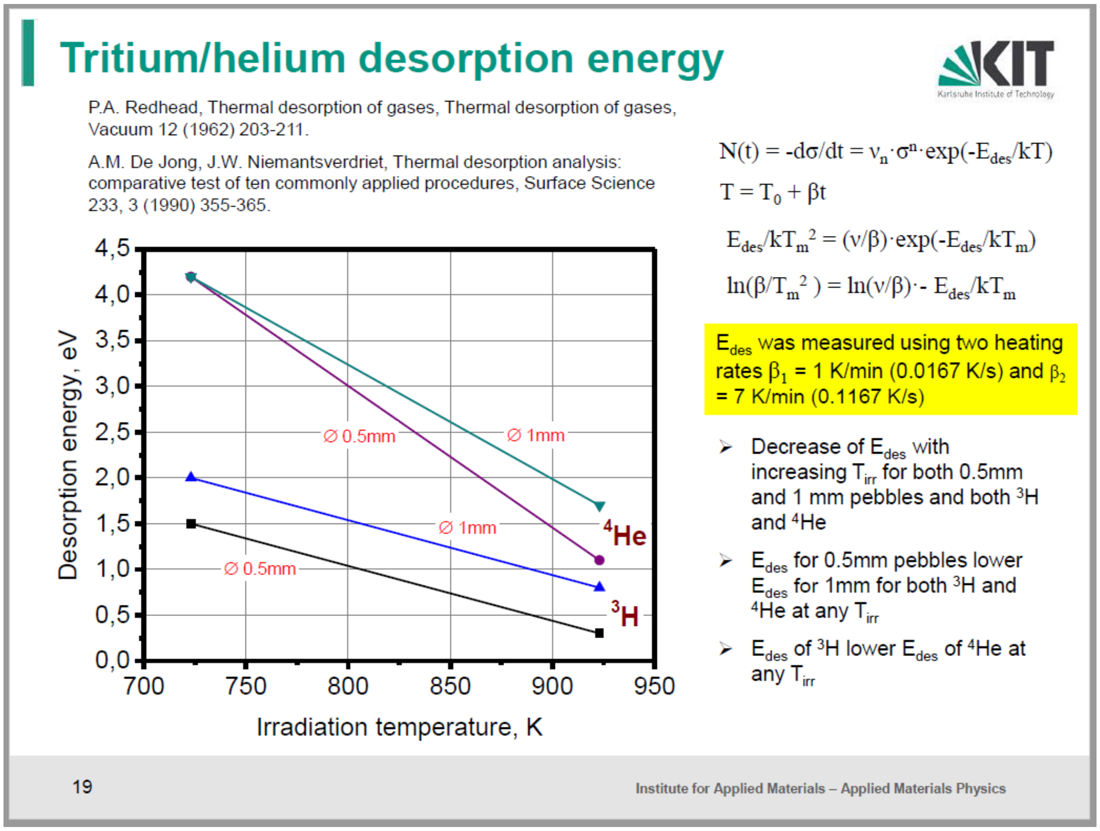
Tritium/helium desorption energy
KIT
Karlsruhe Institute of Technology
P.A. Redhead, Thermal desorption of gases, Thermal desorption of gases, Vacuum 12 (1962) 203-211.
A.M. De Jong, J.W. Niemantsverdriet, Thermal desorption analysis: comparative test of ten commonly applied procedures, Surface Science 233, 3 (1990) 355-365.
$N(t) = -d\sigma/dt = \nu_n \cdot \sigma^n \cdot \exp(-E_{des}/kT)$
$T = T_0 + \beta t$
$E_{des}/kT_m^2 = (\nu/\beta) \cdot \exp(-E_{des}/kT_m)$
$\ln(\beta/T_m^2) = \ln(\nu/\beta) - E_{des}/kT_m$
E_{des} was measured using two heating rates β_1 = 1 K/min (0.0167 K/s) and β_2 = 7 K/min (0.1167 K/s)
Decrease of E_{des} with increasing T_{irr} for both 0.5mm and 1 mm pebbles and both ^3H and ^4He
E_{des} for 0.5mm pebbles lower E_{des} for 1mm for both ^3H and ^4He at any T_{irr}
E_{des} of ^3H lower E_{des} of ^4He at any T_{irr}
Desorption energy, eV
Irradiation temperature, K
4,5
4,0
3,5
3,0
2,5
2,0
1,5
1,0
0,5
0,0
700
750
800
850
900
950
∅ 0.5mm
∅ 1mm
∅ 1mm
∅ 0.5mm
^4He
^3H
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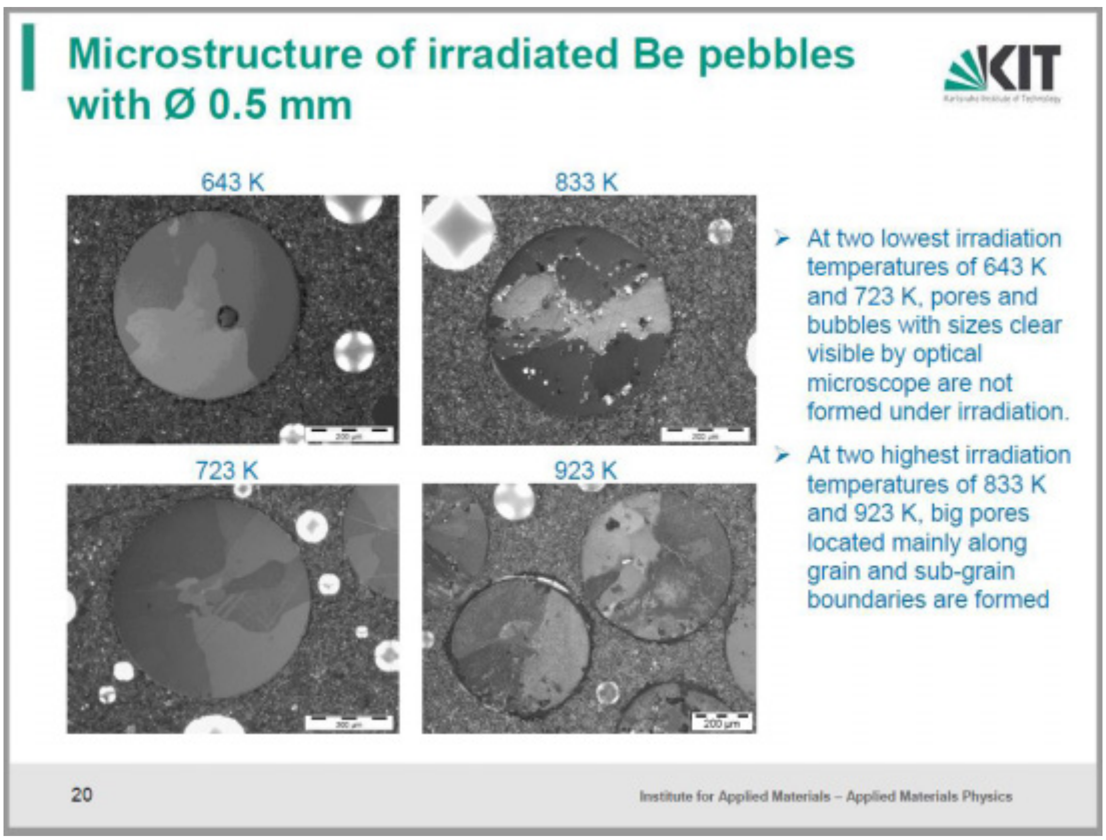
Microstructure of irradiated Be pebbles with Ø 0.5 mm
KIT
643 K
833 K
723 K
923 K
200 µm
At two lowest irradiation temperatures of 643 K and 723 K, pores and bubbles with sizes clear visible by optical microscope are not formed under irradiation.
At two highest irradiation temperatures of 833 K and 923 K, big pores located mainly along grain and sub-grain boundaries are formed
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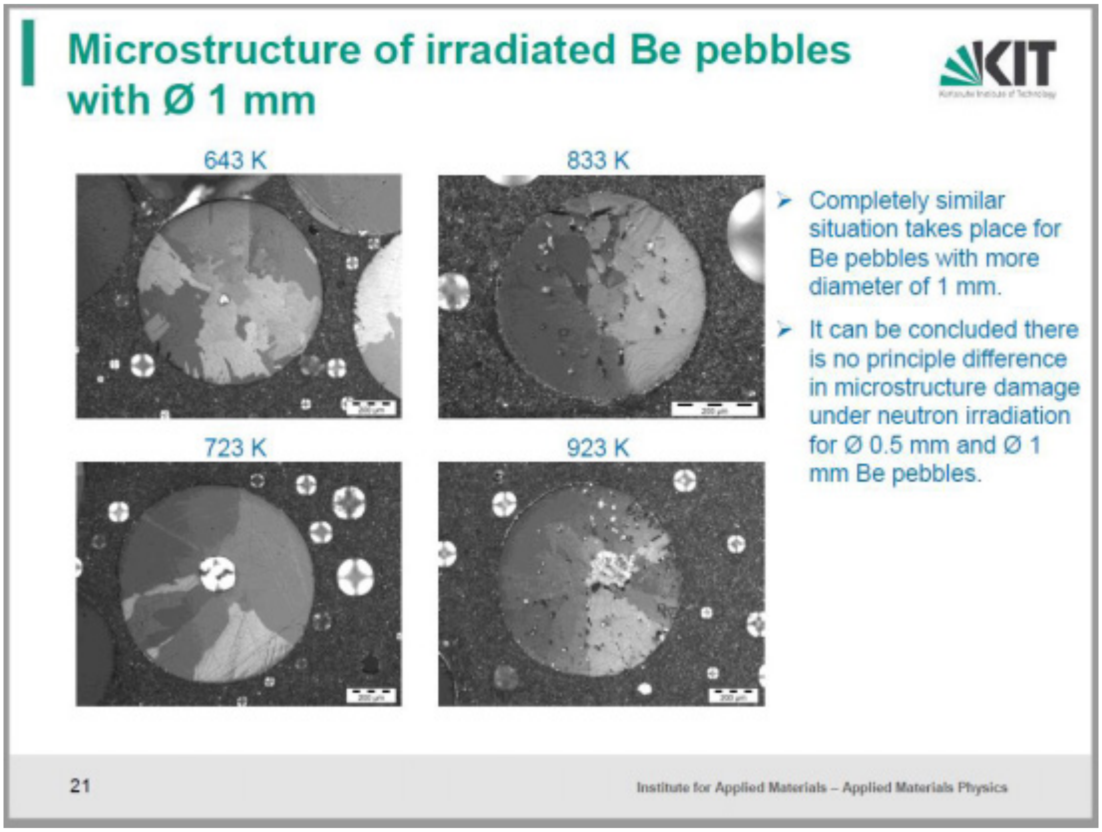
Microstructure of irradiated Be pebbles with Ø 1 mm
KIT
643 K
833 K
723 K
923 K
Completely similar situation takes place for Be pebbles with more diameter of 1 mm.
It can be concluded there is no principle difference in microstructure damage under neutron irradiation for Ø 0.5 mm and Ø 1 mm Be pebbles.
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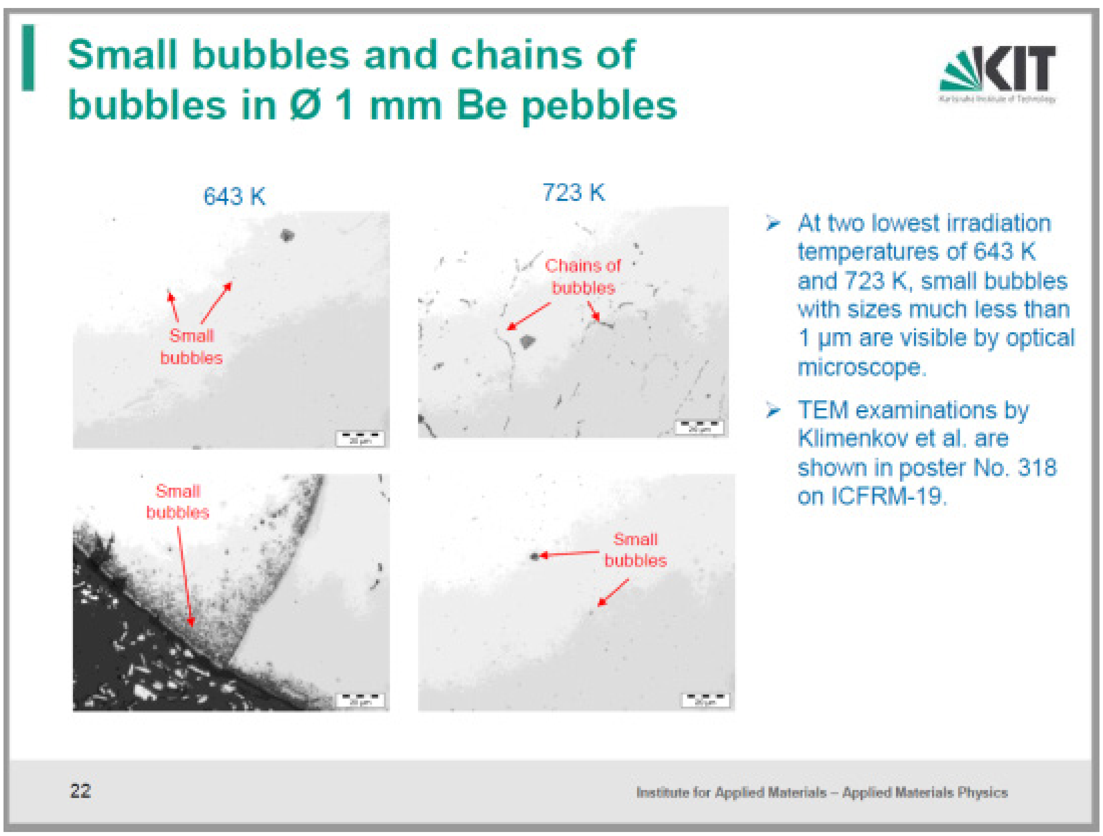
Small bubbles and chains of bubbles in Ø 1 mm Be pebbles
KIT
643 K
723 K
Small bubbles
Chains of bubbles
Small bubbles
Small bubbles
At two lowest irradiation temperatures of 643 K and 723 K, small bubbles with sizes much less than 1 µm are visible by optical microscope.
TEM examinations by Klimenkov et al. are shown in poster No. 318 on ICFRM-19.
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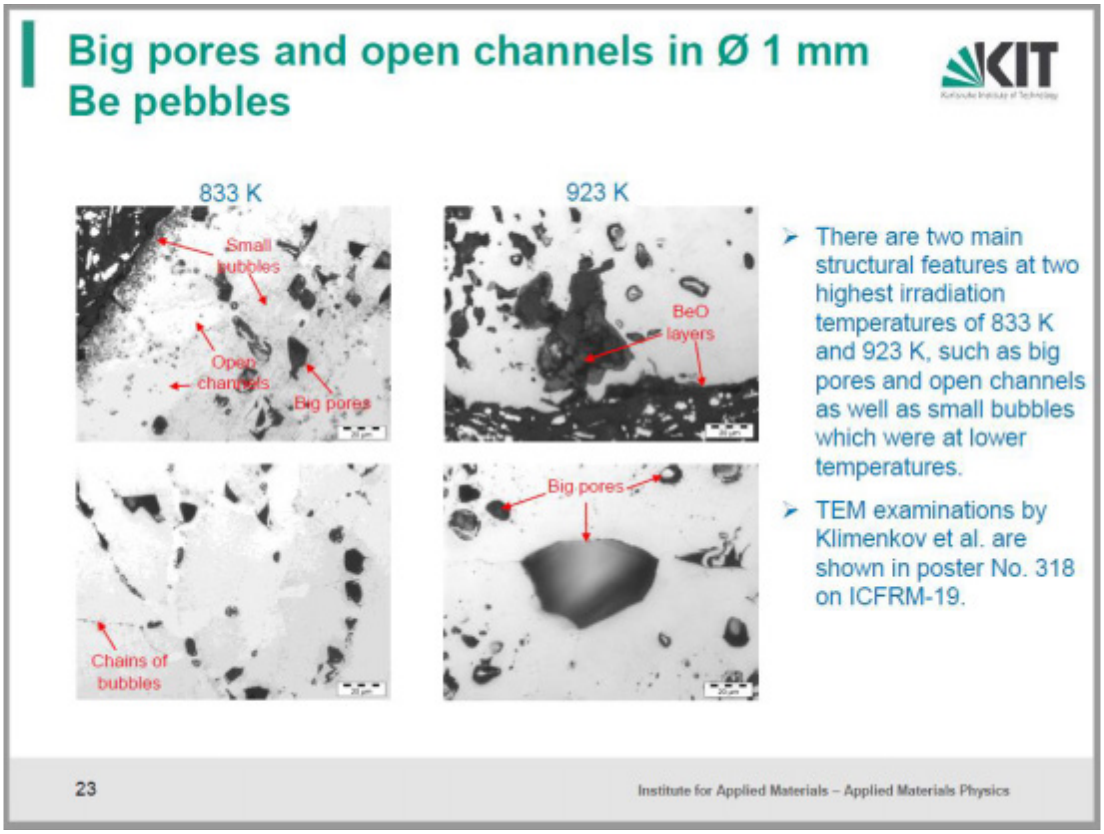
Big pores and open channels in Ø 1 mm Be pebbles
833 K
923 K
Small bubbles
Open channels
Big pores
BeO layers
Chains of bubbles
Big pores
There are two main structural features at two highest irradiation temperatures of 833 K and 923 K, such as big pores and open channels as well as small bubbles which were at lower temperatures.
TEM examinations by Klimenkov et al. are shown in poster No. 318 on ICFRM-19.
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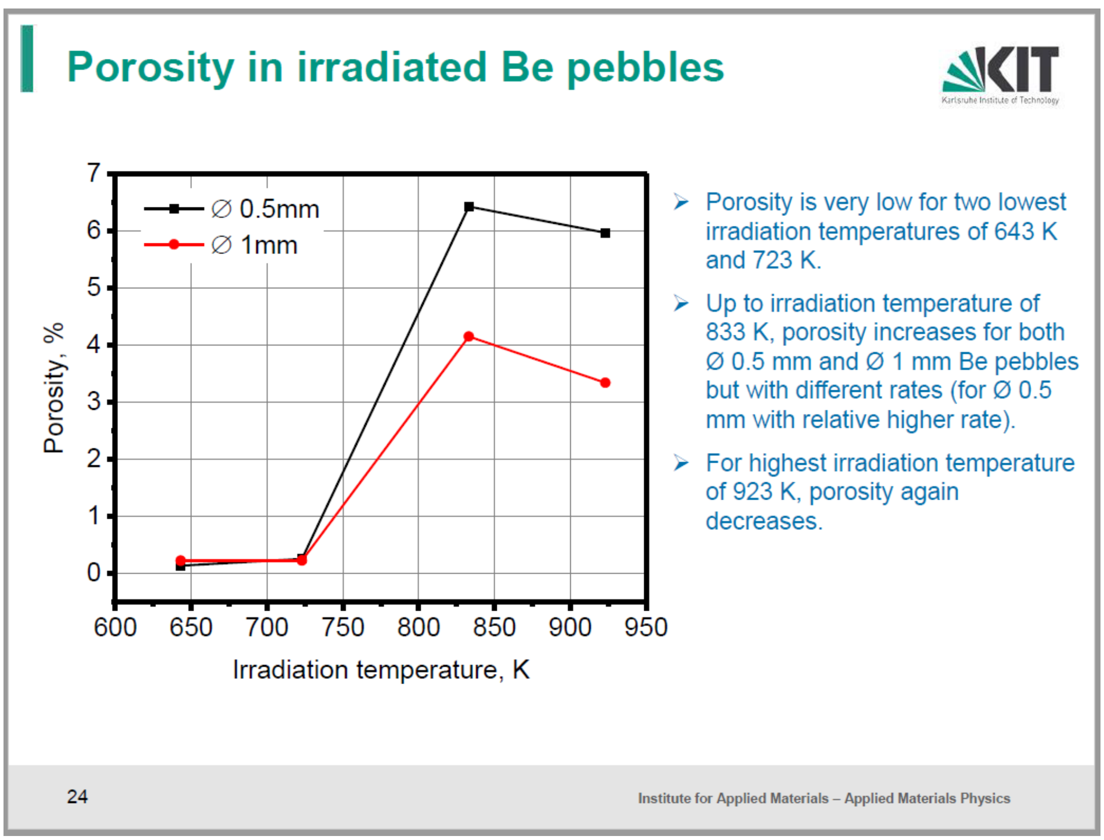
Porosity in irradiated Be pebbles
∅ 0.5mm
∅ 1mm
Porosity, %
Irradiation temperature, K
0
1
2
3
4
5
6
7
600
650
700
750
800
850
900
950
Porosity is very low for two lowest irradiation temperatures of 643 K and 723 K.
Up to irradiation temperature of 833 K, porosity increases for both Ø 0.5 mm and Ø 1 mm Be pebbles but with different rates (for Ø 0.5 mm with relative higher rate).
For highest irradiation temperature of 923 K, porosity again decreases.
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Be and Be-7at.%Ti pellets

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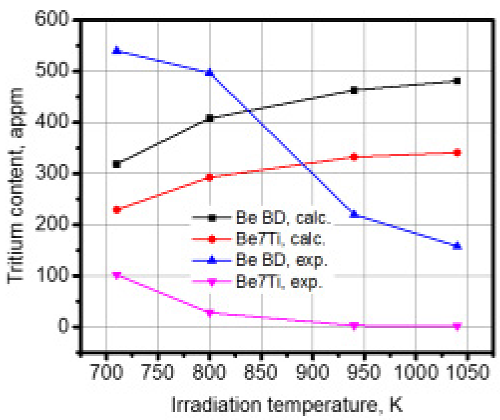
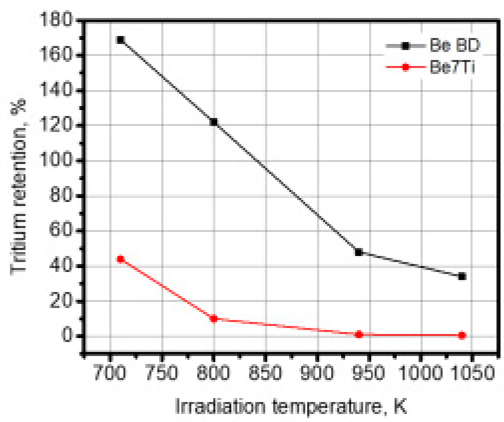
Tritium retention in Be and Be-7at.%Ti pellets





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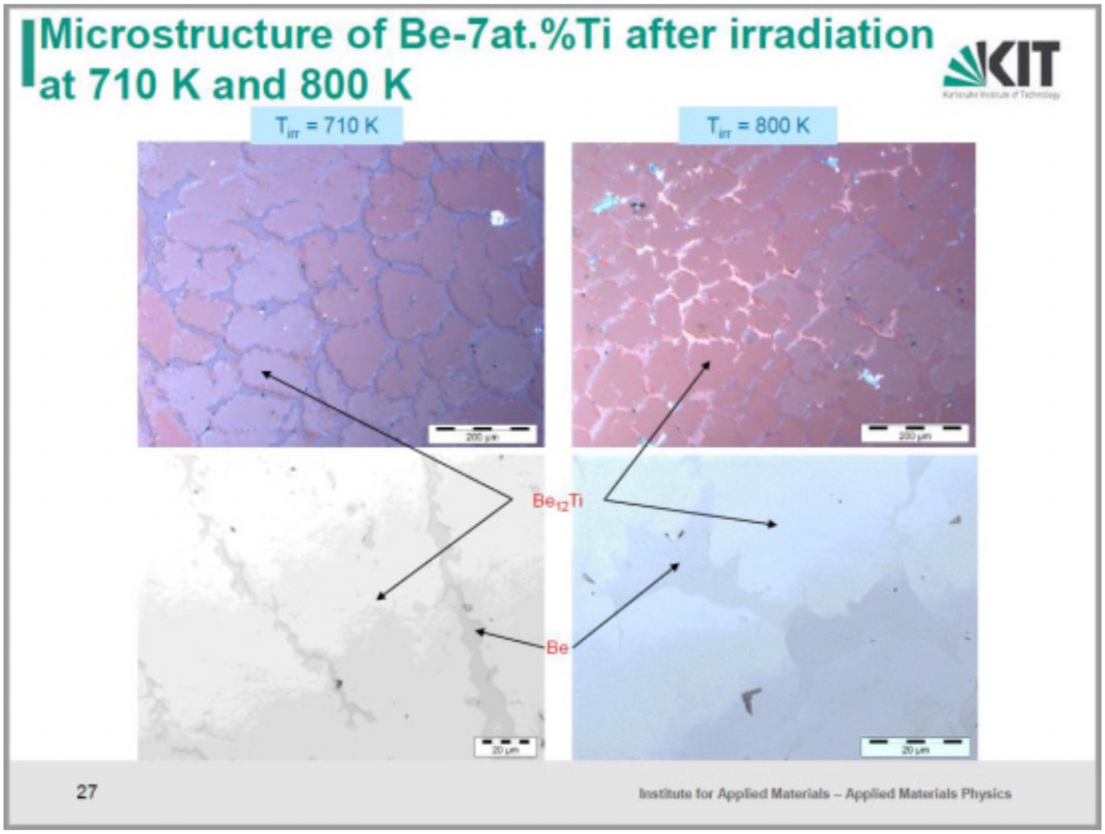
Microstructure of Be-7at.%Ti after irradiation at 710 K and 800 K
KIT
T_{irr} = 710 K
T_{irr} = 800 K
$Be_{12}Ti$
Be
20 µm
27
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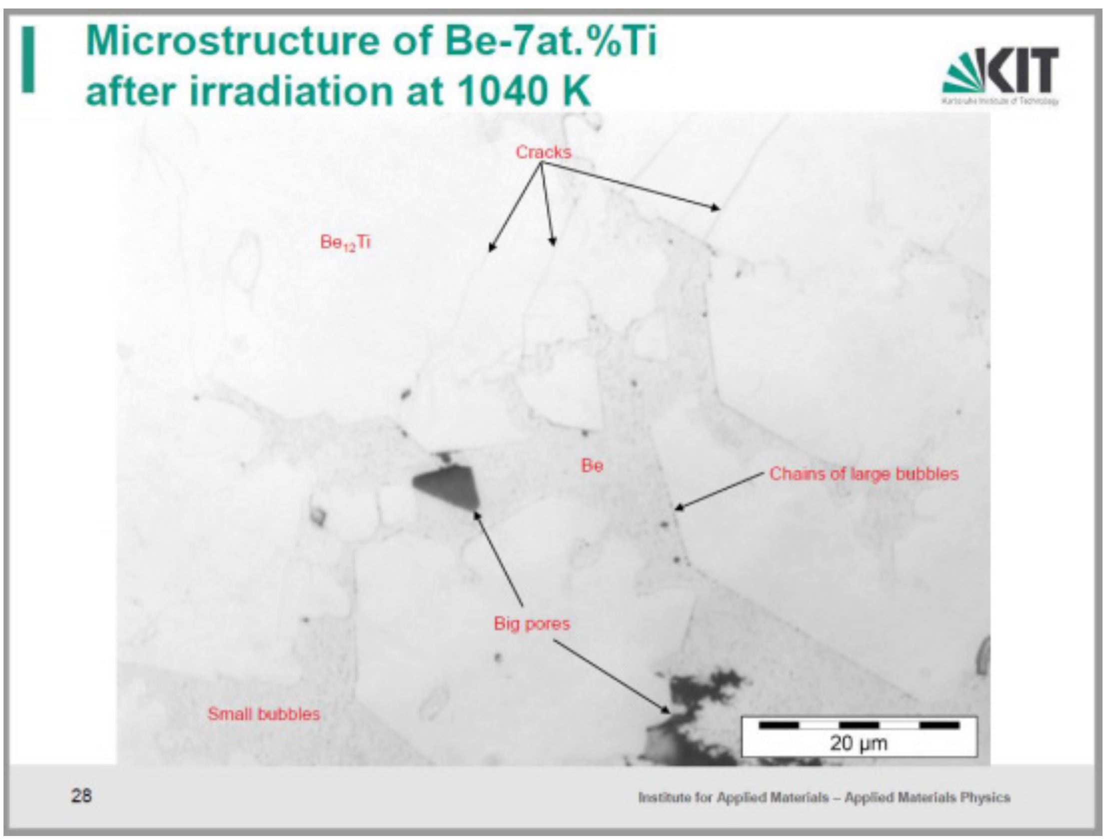
Microstructure of Be-7at.%Ti after irradiation at 1040 K
KIT
Cracks
$Be_{12}Ti$
Be
Chains of large bubbles
Big pores
Small bubbles
20 µm
28
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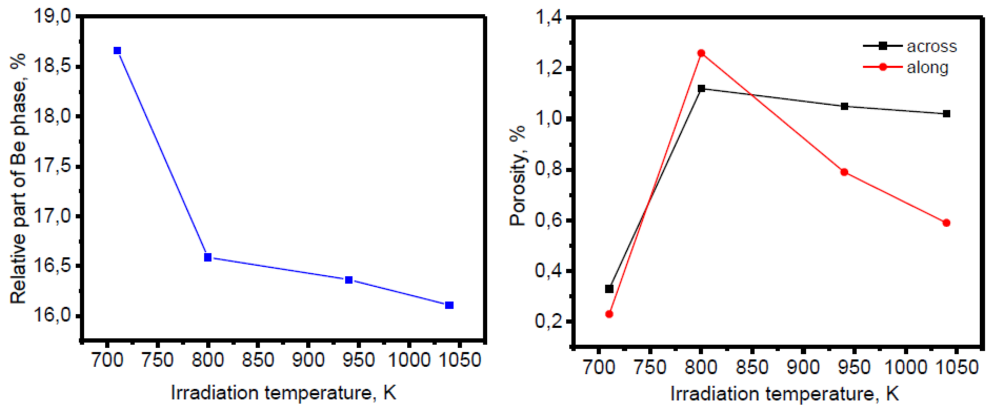
Porosity in Be-7at.%Ti



Porosity has an irregular behavior on increasing irradiation temperature because the big poros were formed in the pellets already before irradiation during production what is typically for arc-melting process.

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Conclusions

1. Be pebbles were exposed under unique irradiation parameters up to 38 dpa damage dose, 6000 appm helium, 640 appm tritium at temperatures of 643-1040 K which are relevant to the HCPB DEMO blanket conditions. This HIDOBE-02 irradiation experiment was in the HFR, Petten, the Netherlands, on 2005-2011, i.e. within 6 years.

2. Tritium escaped Be pebbles and pellets already during irradiation since irradiation temperature of 750-800 K. The most significant amount of tritium leaved beryllium pebbles at temperatures of higher than 800 K when tritium retention was 3-20 % depending on pebble size and heating rate.

3. During irradiation, tritium is trapped by the structural traps such as radiation-induced gas pores and bubbles. The efficiency of traps decreases on increasing irradiation temperature. In addition, formation of open structural channels at highest irradiation temperatures contributes to the accelerated tritium release from beryllium pebbles and pellets.

4.Titanium beryllide Be-7at.%Ti has much lower tritium retention compared to that in Be pellets and even in Be pebbles! This allows to consider titanium beryllide as a potential replacement for pure Be in the HCPB concept of DEMO blanket.

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Thank you so much for your attention!
See you again in 2021 on next BeWS-15!
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Deuterium retention on Be-C-O films: in-situ versus ion implantation loading

R. Mateus[1], C. Porosnicu[2], C.P. Lungu[2], Z. Siketić[3], I. Bogdanovic Radovic[3], M. Kumar[4], C. Pardanaud[4], A. Hakolla[5], and E. Alves[1]

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Beryllium tiles will be a major component of the next fusion reactor chambers. Tiles will cover nearly all the chamber walls and will be exposed to extreme operating conditions where erosion, radiation damage and fuel trapping are the most relevant. One way to study the retention processes rely on the production in laboratory of different kinds of Be coatings. In this study, we report results obtained on 400nm Be-C-O-^{2}H films deposited on both Si and W plates and compared with deuterium implanted films. It is compared the efficiency of the two methods to incorporate ^{2}H in the films and the role of the interlayers between the films and substrates in the retained contents.

Implanted films were prepared at room temperature by using 15keV $^{2}H^{+}$ ion beams. The fluence was limited to 2 x 10^{17} ion/cm^{2} in order to avoid major morphological changes that may enhance a gas release.

The samples were analysed with ion beam techniques namely, elastic backscattering spectroscopy (EBS), Rutherford backscattering spectroscopy (RBS) and nuclear reaction analysis (NRA) making use of 1600keV $^{1}H^{+}$, 2000keV $^{4}He^{+}$ and 1000keV $^{3}He^{+}$ incident beams, respectively. Some months after implantation, aged coatings were analysed by time-of-flight elastic recoil detection (TOF-ERDA) to evaluate ^{2}H release.

Results point to ion implantation as alternative to in-situ load for deuterium incorporation into thin layers and for the used ion fluences retained contents close to 3 at. % of ^{2}H are easily obtained. Be, C, O and ^{2}H depth profiles of thin layers evaluated by EBS/RBS/NRA or by TOF-ERDA are compatible. As predicted and due to the mirror quality of Si plates, the use of Si substrates led to slightly lower retained contents. Also, the release of ^{2}H in aged samples agrees with previous data.

For the case of thick coatings (5-10μm) the limit of 2 x 10^{17} ion/cm^{2} hinders the competitive role of ion implantation, while the retained amounts achieved by co-deposition of ^{2}H deeply depends of the density of defects and C incorporation, as observed by Raman spectroscopy, and easily increase by a factor of 10.

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Deuterium retention on Be-O films: in-situ versus ion implantation loading

Rodrigo Mateus[1], Norberto Catarino[1], Corneliu Porosnicu[2], Cristian P. Lungu[2], Zdravko Siketić[3], Iva Bogdanovic Radovic[3], Eduardo Alves[1]

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14th International Workshop on Beryllium Technology, 24th to 25th October 2019, Long Beach, USA

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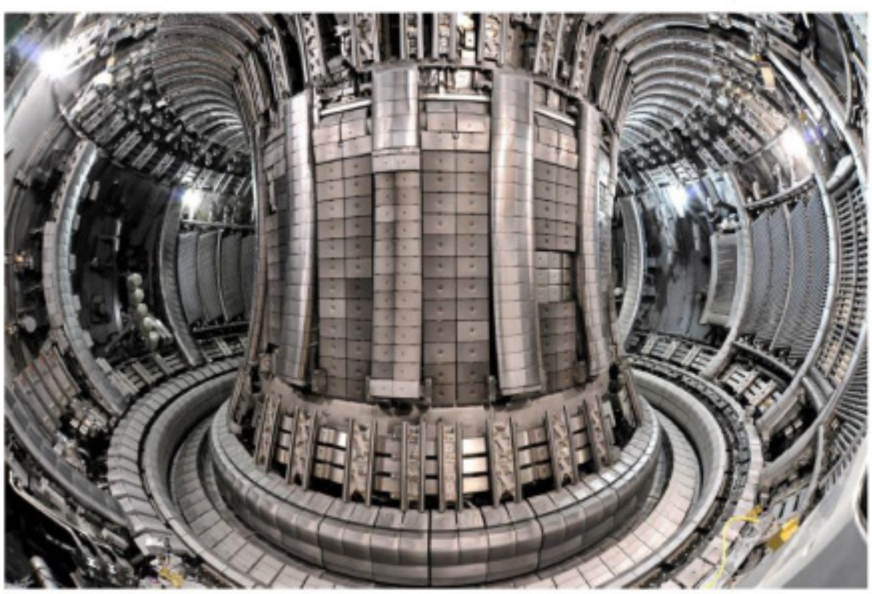
Beryllium will be major element in the next Fusion Reactors

The ITER-like Wall in JET 2012



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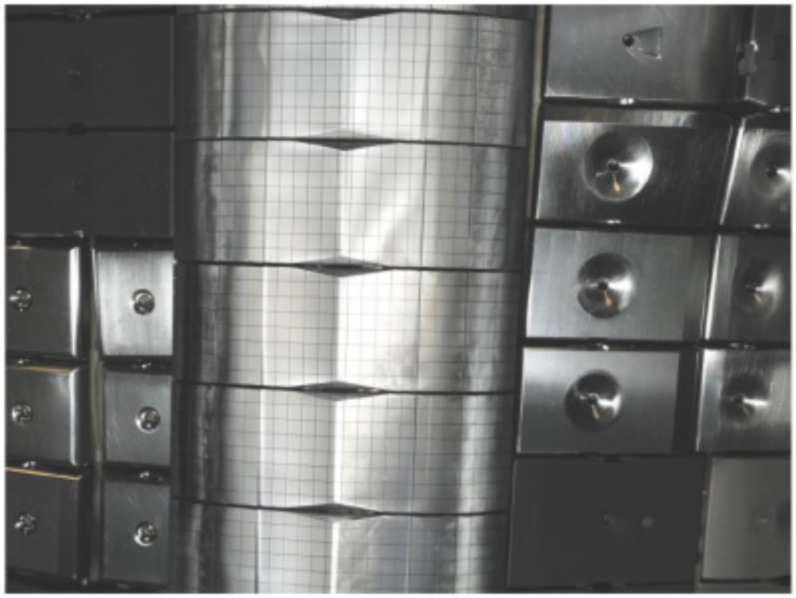
JET Be limiter tiles



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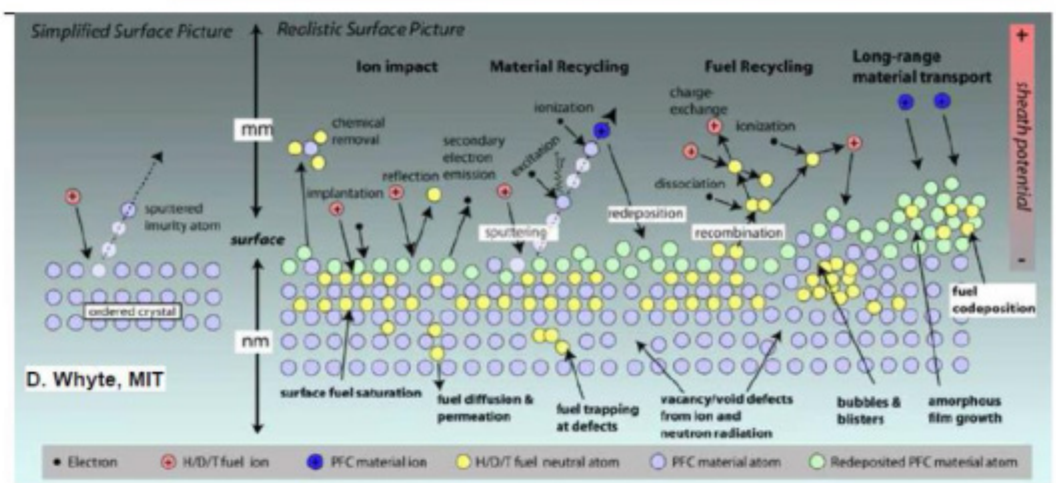
Particle-Wall interaction is a complex dynamic non- equilibrium process



(Courtesy Dennis Whyte, MIT)

Surface is continuously evolving by erosion, deposition, mixing, chemical reactions, implantation……at different rates.

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Ion Beam Measurements

Elements	Techniques
➢ ^{1}H and ^{2}H	➢ ERDA, NRA with ^{3}He 0.8 – 2.4 MeV
➢ ^{12}C and ^{13}C	➢ RBS, EBS ≠ ^{1}H energies, NRA
➢ Be, B	➢ EBS, NRA, SIMS
➢ W, Ni (+ Cr, Fe, Cu)	➢ RBS, PIXE, SIMS
➢ Complex depth profiles	➢ NDF – simultaneous analysis

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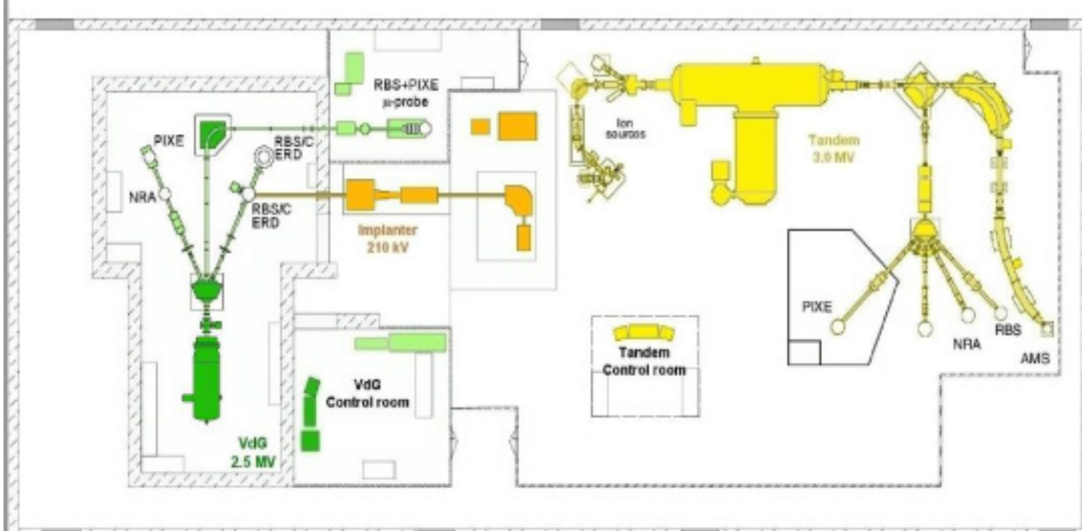
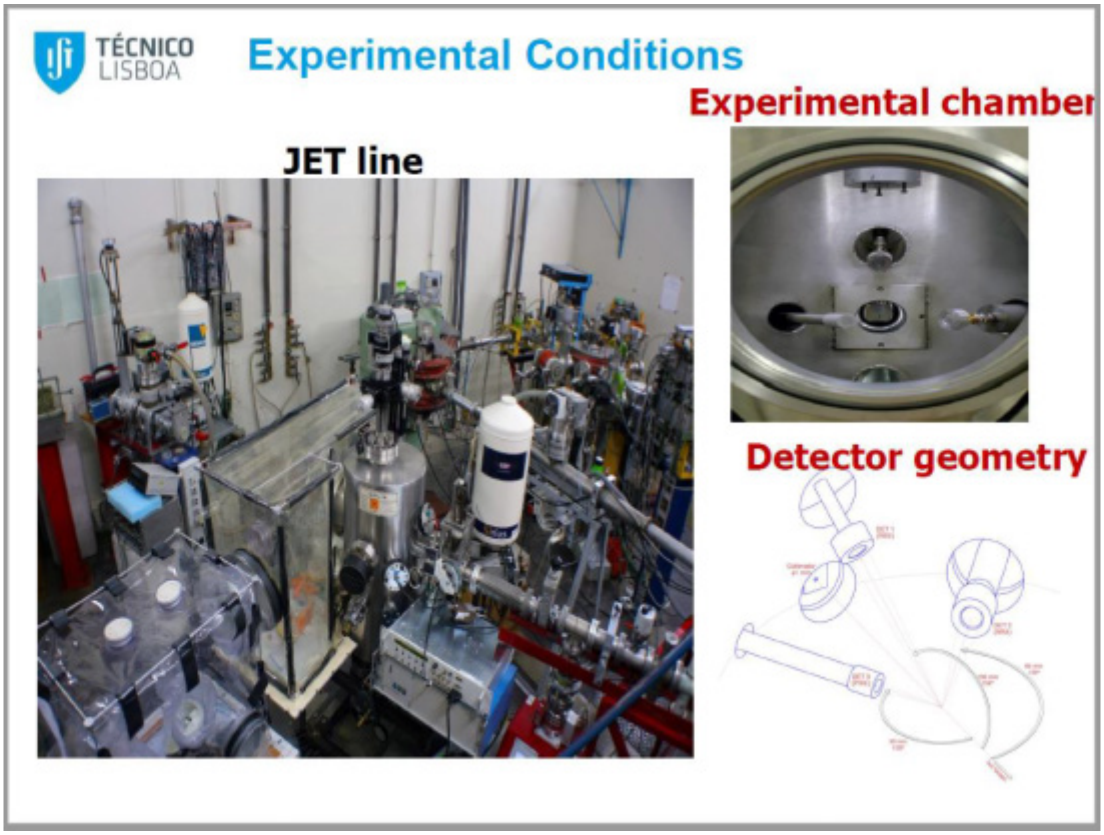
Ion Beam Laboratory CTN-IST

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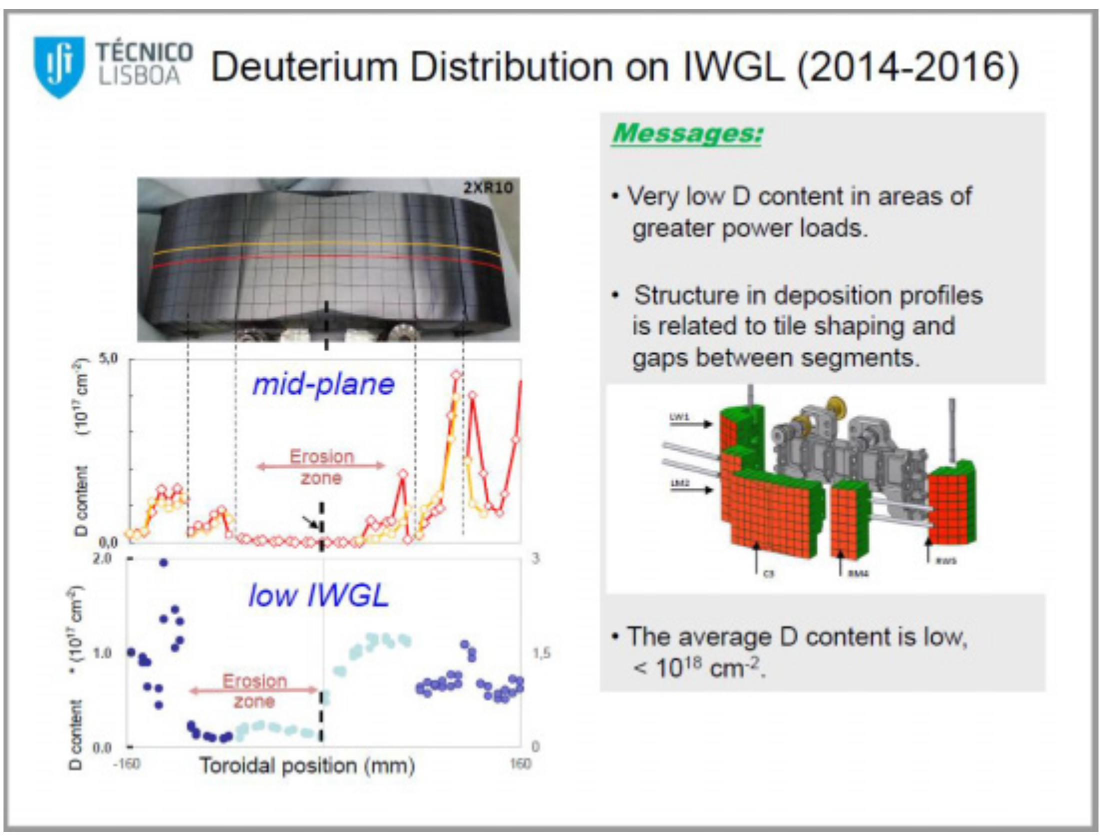
Experimental Conditions
Experimental chamber
JET line
Detector geometry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Deuterium Distribution on IWGL (2014-2016)
2XR10
mid-plane
Erosion zone
D content (10^17 cm^-2)
5,0
0,0
low IWGL
Erosion zone
D content * (10^17 cm^-2)
2.0
1.0
0.0
3
1,5
0
-160
Toroidal position (mm)
160
Messages:
• Very low D content in areas of greater power loads.
• Structure in deposition profiles is related to tile shaping and gaps between segments.
• The average D content is low, < 10^18 cm^-2.

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Motivation

- Gas retention in thin films and coatings for fusion applications
- Production of Be-based materials (Be, Be-O, Be-C-O) for fusion research by implantation of $^{2}H^{+}$ ions with fluences of 2 x 10^{17} ion/cm^{2}
- Implantation of energetic 3-10 keV $^{2}H^{+}$ ions in pure Be at RT

  2 x 10^{17} ion/cm^{2} ⇒ fluence limit for the saturated of ^{2}H in Be
  blistering, nano-cavities towards the surface
  gas release mechanism initialized at 3 x 10^{16} at/cm

  (V.N. Chernikov et al., J. Nucl. Mater. 233-237 (1996) 860–864)
- Similar fluence limit and structural effects for implanted 3-15 keV He^{+} ions

  Threshold limit at 2 x 10^{17} ion/cm^{2}

  (V.N. Chernikov et al., J. Nucl. Mater. 258-263 (1998) 694–699)

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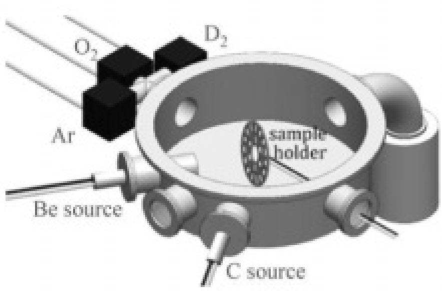
Deposition of thin Be-based coatings by HiPIMS

- High-power impulse magnetron sputtering (HiPIMS) at INFLPR, Romania

  (P. Dinca et al. Surf. Coat. Technol. 321 (2017) 397–402



HiPIMS deposition chamber:

Ar as background gas

O_2 $^{2}H_2$ gas - O and ^{2}H sources

Be, graphite rods - Be and C sources

Table 1
Nominal compositions of Be coatings deposited by HiPIMS.

Nominal composition	Nominal stoichiometry	Nominal composition	Nominal stoichiometry
Be on Si	-	Be+D on Si	(100:5)
Be+O on Si	(95:5)	Be+O on Si	(95:5:5)
Be+O on Si	(90:10)	Be+O+D on Si	(90:10:5)
Be+O+C on Si	(90:5:5)	Be+O+C+D on Si	(90:5:5:5)
Be+O+C on Si	(84:8:8)	Be+O+C+D on Si	(84:8:8:5)
Be on W	-	Be+D on W	(100:5)
Be+O on W	(95:5)	Be+O+D on W	(95:5:5)
Be+O on W	(90:10)	Be+O+D on W	(90:10:5)
Be+O+C on W	(90:5:5)	Be+O+C+D on W	(90:5:5:5)
Be+O+C on W	(84:8:8)	Be+O+C+D on W	(84:8:8:5)

- Be, Be-O, Be-C-O thin films

  400 nm nominal thickness

  Si and W plates as substrates

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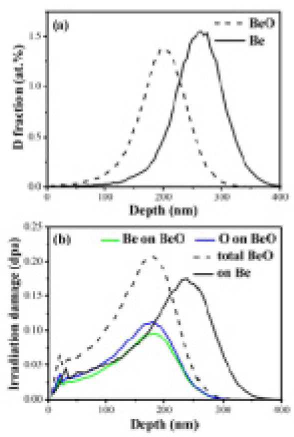
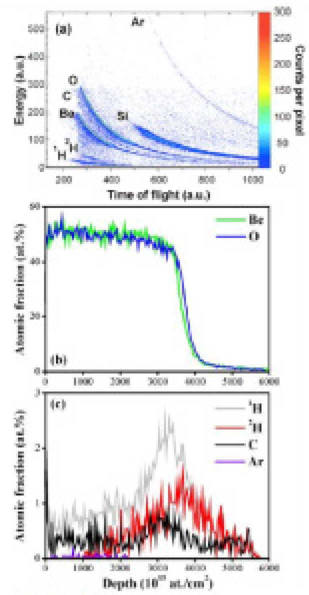
Results: SRIM calculations / Ion Beam Analysis as-implanted Be-O (90:10) film on Si



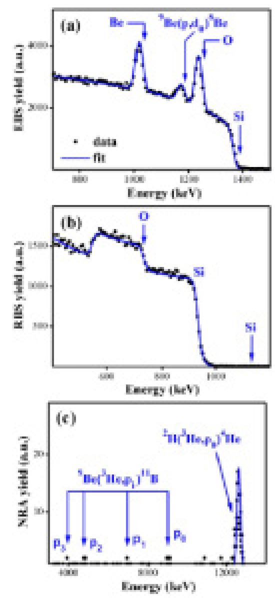
SRIM simulations:

15 keV ^{2}H$^+$ ions
fluence of 2 x 10^{17} ion/cm^2:

(a) ^{2}H depth profiles on Be and on BeO
(b) corresponding irradiation damage



TOF-ERDA:
(a) 2D coincident maps
(b) depth profiles for Be and O
(c) and for ^{1}H, ^{2}H, C and Ar



(a) EBS with 1600 keV ^{1}H$^+$
(b) RBS with 2000 keV ^{4}He$^+$
(c) NRA with 1000 keV ^{3}He$^+$

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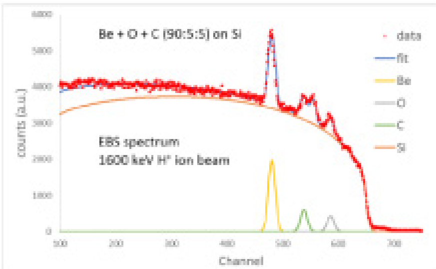
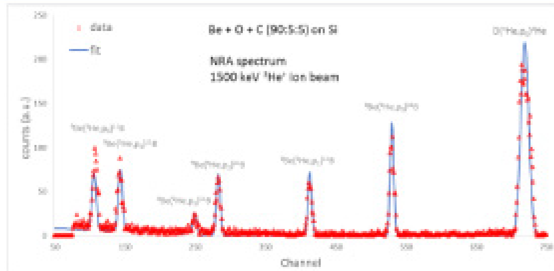
Results: retained ^{2}H contents Ion implantation vs. in-situ loading

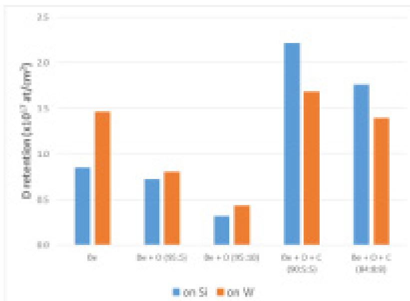
Table 2
Thicknesses and depth contents of ^{2}H in Be-based coatings loaded in-situ by HiPIMS or by ion implantation with ^{2}H.

Coating's description		In-situ loading of ^{2}H		Ion implantation of ^{2}H			
		EBS/RBS/NRA (as deposited)		EBS/RBS/NRA (as-implanted)		ToF-ERDA (3 month aged)	
		Thickness (10^{17} at/cm^2)	^{2}H content (10^{17} at/cm^2)	Thickness (10^{17} at/cm^2)	^{2}H content (10^{17} at/cm^2)	Thickness (10^{17} at/cm^2)	^{2}H content (10^{17} at/cm^2)
Be on Si	-	57.5	1.89	33.0	0.82	32.4	1.10
Be+O on Si	(95:5)	46.0	0.48	51.0	0.96	53.7	0.67
Be+O on Si	(90:10)	35.0	0.62	40.5	0.62	37.5	0.20
Be+O+C on Si	(90:5:5)	27.5	1.02	26.5	1.87	25.0	1.70
Be+O+C on Si	(84:8:8)	25.8	0.80	25.0	1.91	17.5	1.90
Be on W		-	2.21	-	1.16	33.3	1.00
Be+O on W	(95:5)	-	0.76	-	1.23	-	0.92
Be+O on W	(90:10)	-	0.52	-	0.89	39.0	0.78
Be+O+C on W	(90:5:5)	-	1.10	-	1.80	25.0	1.90
Be+O+C on W	(84:8:8)	-	1.21	-	0.63	22.0	0.55

Results of retained ^{2}H: Ageing effect implanted Be-O-C (90:5:5) film on Si







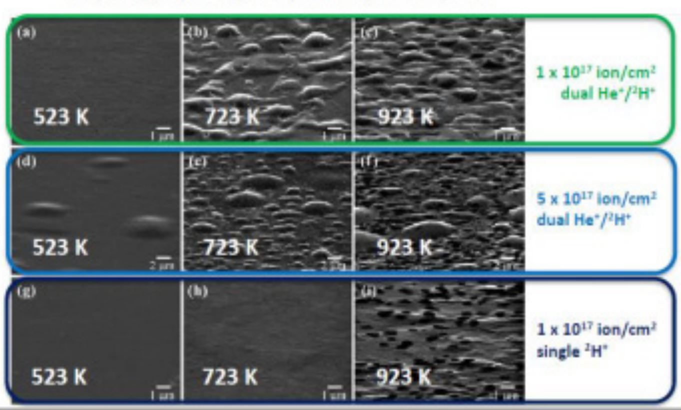
- **Absence of a ^{2}H release, 2 or 3 years after the implantation route (differences close to ~5 at.% between ^{2}H quantified in as-irradiated and in aged samples)**

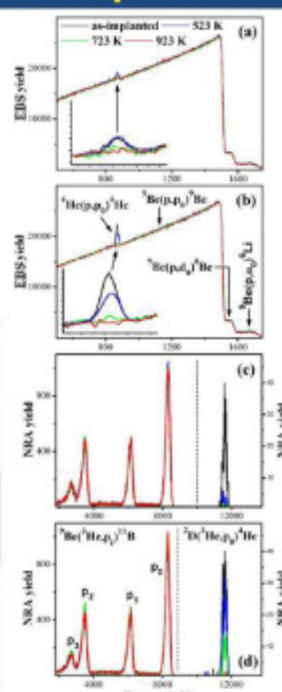
Retention behaviour of ^{2}H and He in Be under dual He$^+$/^{2}H$^+$ and single ^{2}H$^+$ exposure

Table 5
Retained He and D contents in the implanted zone

Fluences	Ion implantation	He/D contents (1e15 at/cm^2) at RT	at 523 K	at 723 K	at 923 K
1e17 at/cm^2	dual ^{4}He$^+$/D$^+$	100/33.0	100/ 4.1	15/ 2.1	0/0
1e17 at/cm^2	single D$^+$	-/21.2	-/18.9	-/16.5	0/0
5e17 at/cm^2	dual ^{4}He$^+$/D$^+$	300/34.2	195/24.8	27/11.8	0/0
5e17 at/cm^2	single D$^+$	-/63.1	-/48.1	-/ 5.9	0/0

Morphology depending on implantation/annealing route





Deuterium retention in Be-W films (Be, Be:W, W) with distinct roughness profiles

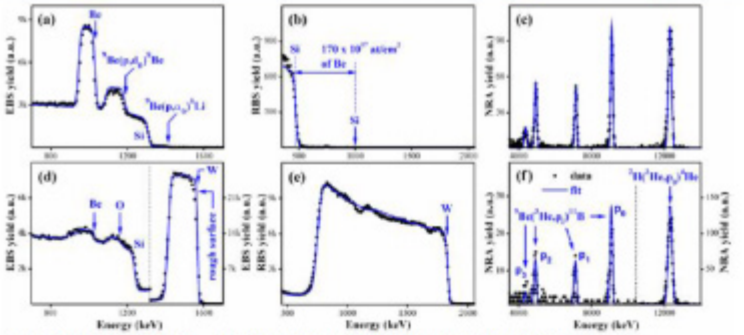


Figure: As-deposited Be / -700 V: (a) EBS, (b) RBS and (c) NRA spectra;
As-deposited Be:W / -700 V: (a) EBS, (b) RBS and (c) NRA spectra;

Table 3
Averaged roughness (Ra), maximum peak-to-peak amplitude (Rp-p), thickness and Be, O and W contents in the as-deposited films and D contents in the as-implanted films. The errors for the elemental analysis are 5% and 10% for the EBS/RBS and NRA results, respectively.

as-deposited coatings	Ra (nm)	Rp-p (nm)	Thickness (10^{17} at/cm^2)	Be (at.%)	O (at.%)	W (at.%)	D after 1e17 ion/cm^2 (10^{17} at/cm^2) / (at.%)		D after 5e17 ion/cm^2 (10^{17} at/cm^2) / (at.%)	
							1.0 MeV $^3He^+$	1.5 MeV $^3He^+$	1.0 MeV $^3He^+$	1.5 MeV $^3He^+$
Be/-700 V	52	424	170	100	~0	-	0.78 / 1.5	0.78 / 1.5	4.43 / 8.7	4.00 / 7.8
Be/0 V	56	386	175	100	~0	-	0.77 / 1.5	0.81 / 1.5	4.68 / 9.1	4.64 / 9.1
Be/+400 V	88	730	160	100	~0	-	0.81 / 1.5	0.76 / 1.4	4.65 / 9.3	4.55 / 9.1
Be:W/-700 V	3.1	34	73	45	~0	55	0.59 / 2.0	0.71 / 2.4	3.62 / 13	3.52 / 13
Be:W/0 V	0.3	2.6	74	40	5	55	0.76 / 2.8	0.81 / 3.0	2.58 / 9.9	2.79 / 11
Be:W/+400 V	3.0	20	69	33	7	60	0.72 / 2.6	0.71 / 2.5	3.97 / 15	3.87 / 15
W/-700 V	5.0	36	76	-	-	100	0.34 / 2.1	-	1.57 / 9.5	-
W/0 V	3.4	32	79	-	-	100	0.37 / 2.3	-	0.98 / 5.9	-
W/+400 V	9.0	57	79	-	-	100	0.35 / 2.1	-	1.17 / 7.1	-

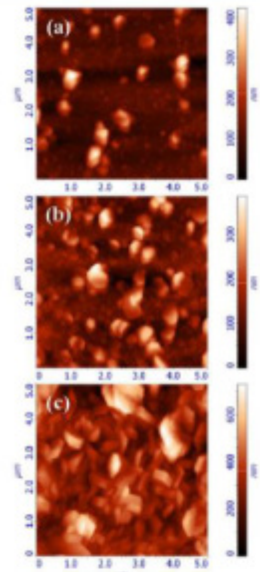


Figure: 2D AFM maps.
(a) as-deposited Be / -700 V
(b) as-deposited Be / 0 V
(c) as-deposited Be / +400 V

Resume

- 400 nm Be-based coatings (Be, Be-O, Be-O-C) were implanted with 15 keV ^2H ions in November 2016 and November 2017;
- Implanted fluences (2 x 10^{17} ion/cm^2) similar to the fluence limit to avoid saturation of ^2H and $^2H_2$ in Be;
- Absence of a ^2H release, 2 or 3 years after the implantation route, as pointed by NRA (differences close to ~5 at.% between ^2H quantified in as-irradiated and in aged samples);
- Carbon enhances 2H retention;
- Studies of He and ^2H retention and release mechanisms from Be-based coatings at IST;
- Dual He/^2H and single ^2H irradiation effects: He "stabilises" 2H in Be.
- Retention vs. Roughness effects under study.

Aknowledgements:

EUROfusion Consortium WP:PFC

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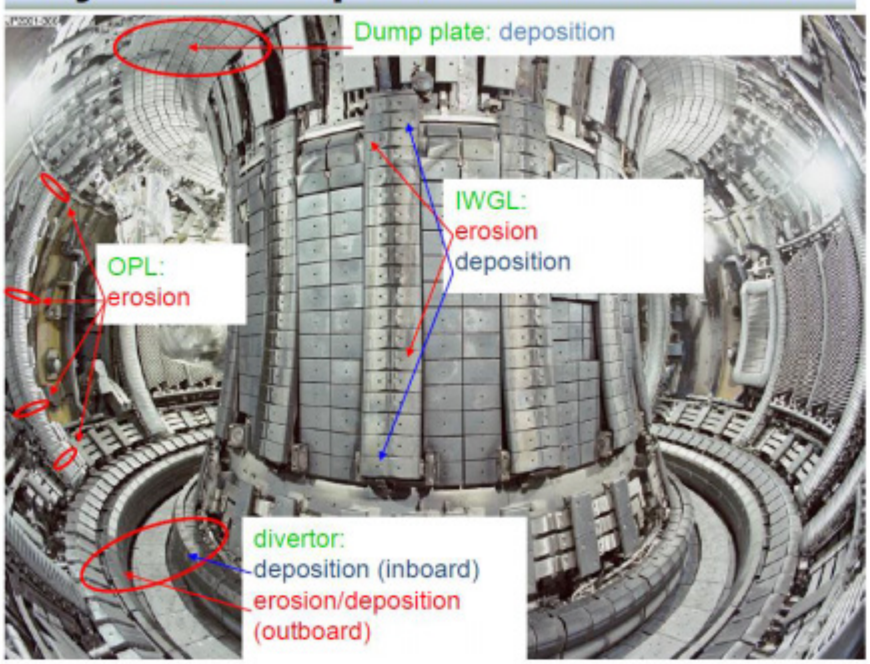
Major areas of plasma-wall interaction



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Analysis of Be-D coatings (IAP) deposited on W plates at different temperatures

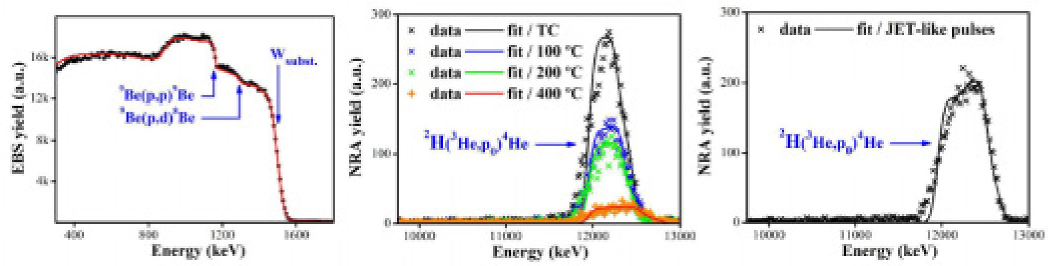
- Be-D coatings deposited at constant temperatures from CT up to 400ºC and under a JET-like pulses regime

Table 1. Be+D coatings co-deposited on W plates and ^{2}H content measured by NRA.

Reference	Nominal compositions	Nominal thickness (μm)	Substrate Temperature (ºC)	^{2}H content by NRA (10^{15} at/cm^{2})
60190516_1_2	Be+D(10%)	5	Chamber Temp. (CT)	262
60190520_1_2	Be+D(5%)	5	Chamber Temp. (CT)	885
60190528_1_2	Be+D(5%)	5	100	489
60190527_1_2	Be+D(10%)	5	200	368
60190523_1_2	Be+D(5%)	5	400	45
60190607_1_2	Be+D(10%)	5	JET-like pulses	690

EBS (1800 keV $^{1}H^{+}$, 2250 keV $^{1}H^{+}$ beams) and NRA (2200 keV $^{3}He^{+}$) analyses

2H retention as function of deposition temperature.



Oxidation of neutron-irradiated Be pebbles

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Beryllium will be used as a neutron-multiplier material to increase the tritium-breeding ratio in nuclear fusion reactors. However, the chemical reactivity of beryllium is a factor of major concern, since the oxidation of beryllium at temperatures above 800°C can become an uncontrollable process.

This work presents a detailed study of chemical composition and reactivity of Be pebbles after exposure to neutron irradiation, up to 3000appm He production and their influence in the oxidation kinetics process. In order to get information on this reaction, samples were oxidized at 700°C under controlled air atmosphere (60% humidity) and with a mixture of 60%O_2 + 40%N_2. The chemical composition of the irradiated samples was studied using ion beam analysis and scanning electron microscopy. The observations revealed the presence of small flakes, which are distributed in the surface, however no modifications in the pebbles shape after longer oxidation were observed. Ion beam elemental maps confirmed the presence of Be oxides distributed on the surface. Moreover, the thickness of the oxide layer increases with the annealing time following a parabolic law.

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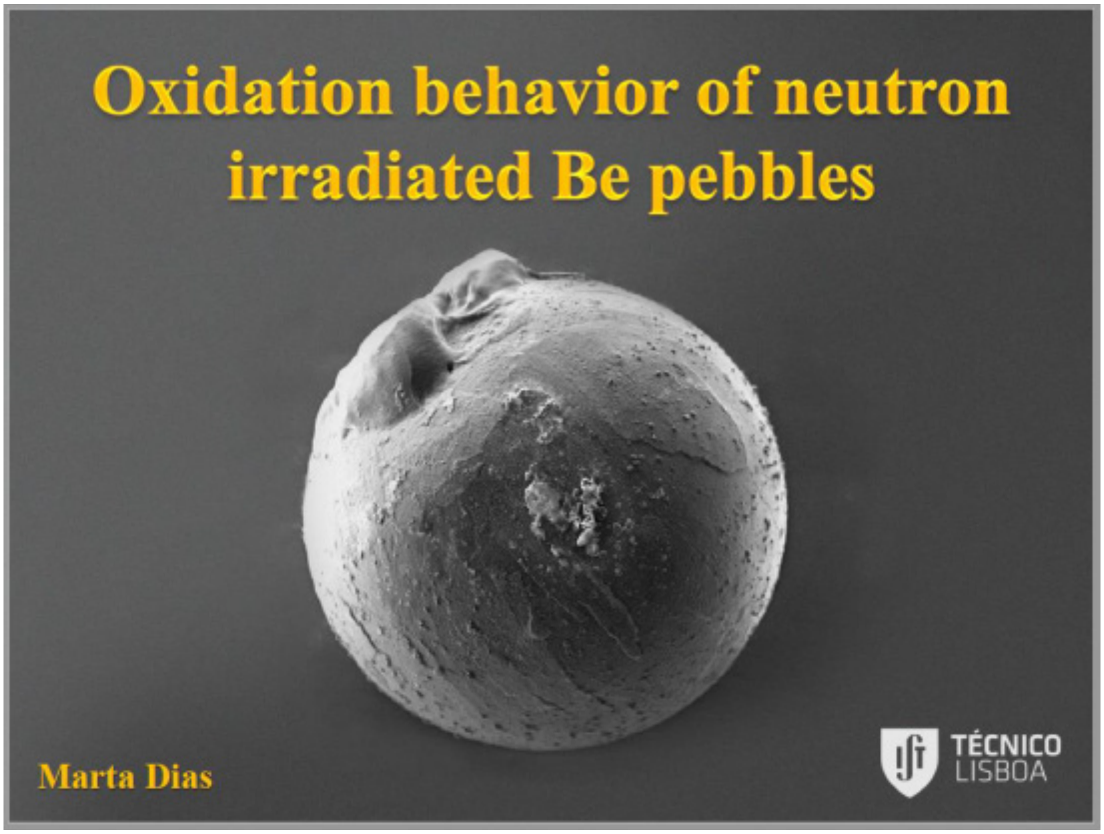
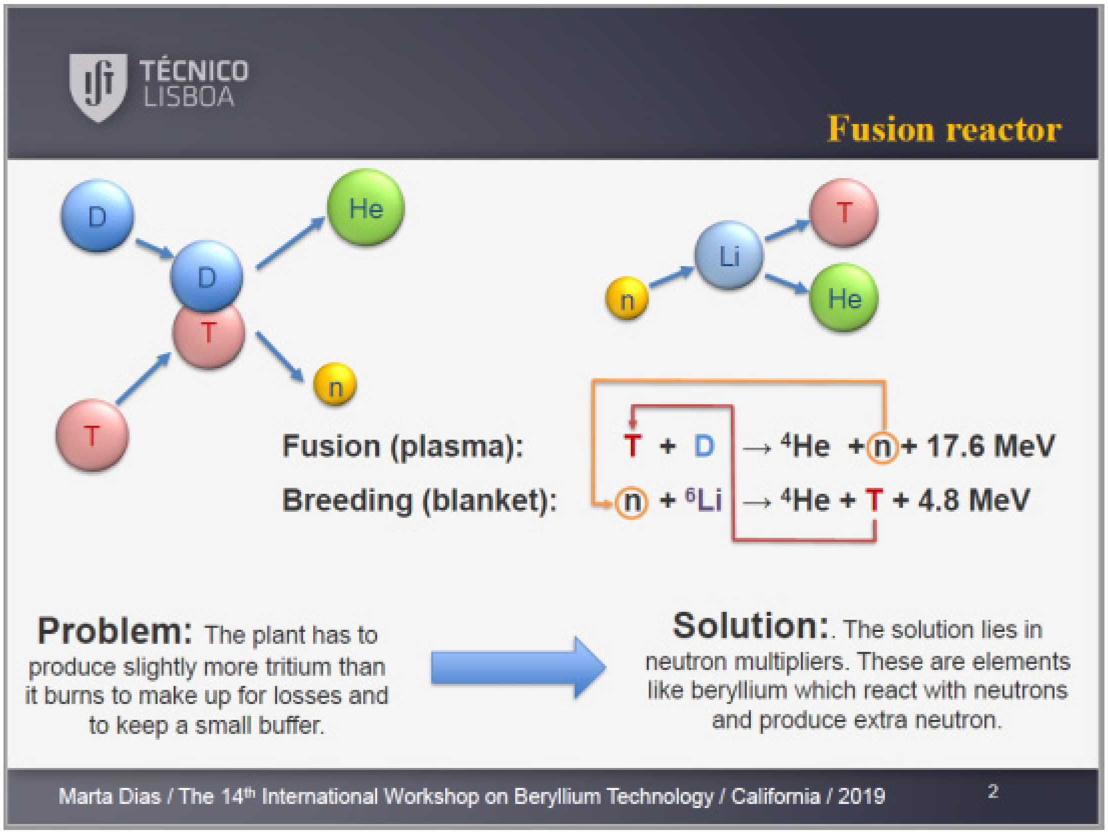

Oxidation behavior of neutron irradiated Be pebbles
Marta Dias
TÉCNICO LISBOA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Fusion reactor
D
D
T
T
He
n
n
Li
T
He
Fusion (plasma): T + D → ⁴He + n + 17.6 MeV
Breeding (blanket): n + ⁶Li → ⁴He + T + 4.8 MeV
Problem: The plant has to produce slightly more tritium than it burns to make up for losses and to keep a small buffer.
Solution:. The solution lies in neutron multipliers. These are elements like beryllium which react with neutrons and produce extra neutron.
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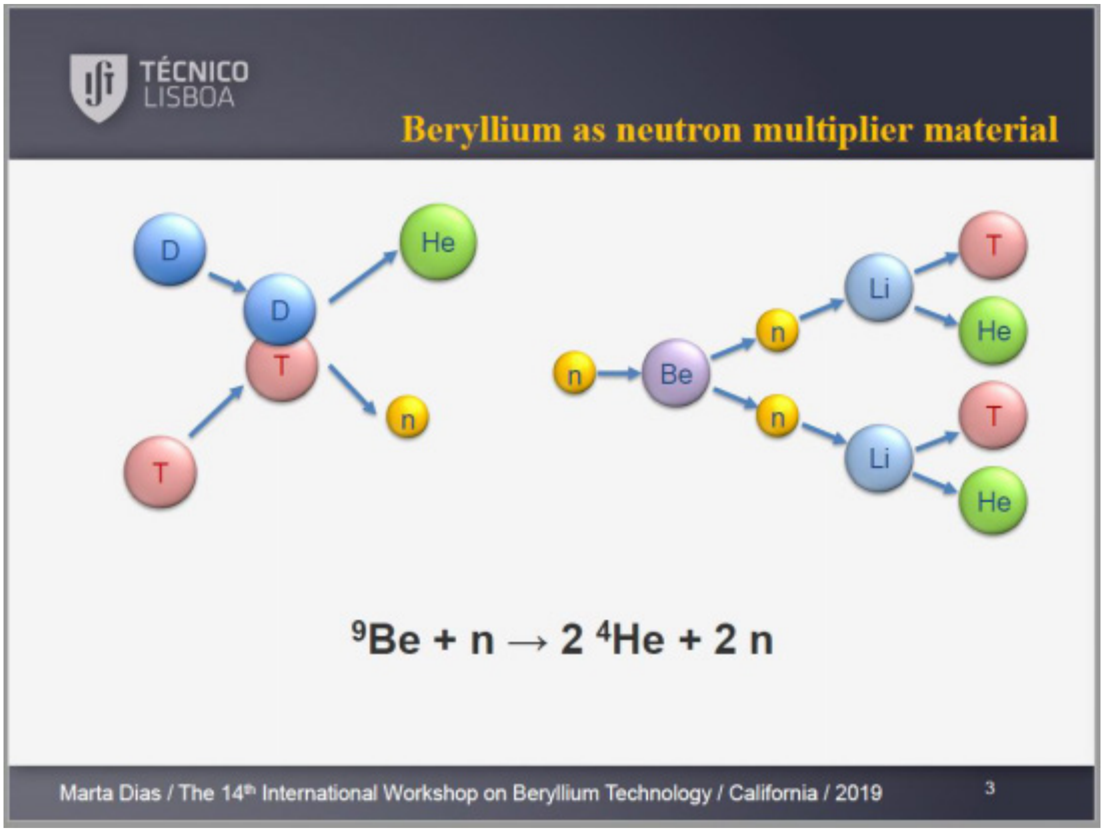
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Beryllium as neutron multiplier material
D
D
T
T
He
n
n
Be
n
n
Li
Li
T
He
T
He
^{9}Be + n → 2 ^{4}He + 2 n
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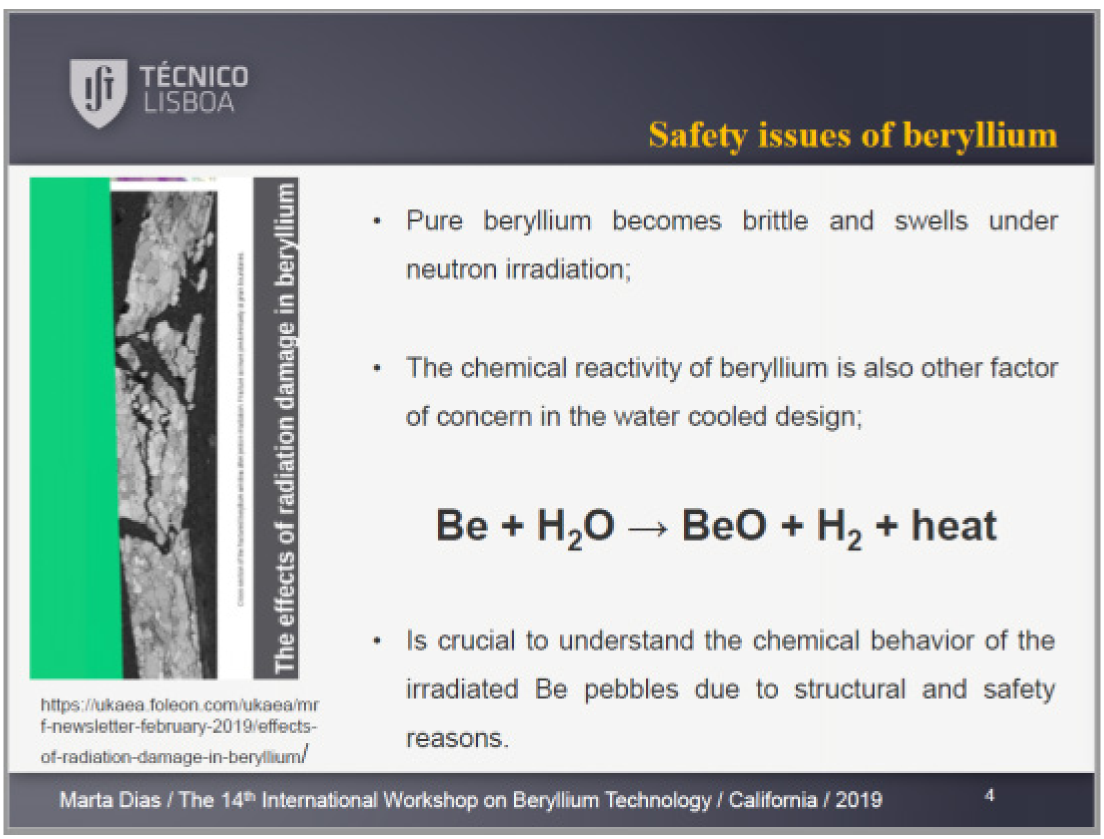
3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Safety issues of beryllium
The effects of radiation damage in beryllium
https://ukaea.foleon.com/ukaea/mrf-newsletter-february-2019/effects-of-radiation-damage-in-beryllium/
• Pure beryllium becomes brittle and swells under neutron irradiation;
• The chemical reactivity of beryllium is also other factor of concern in the water cooled design;
Be + H_2O → BeO + H_2 + heat
• Is crucial to understand the chemical behavior of the irradiated Be pebbles due to structural and safety reasons.
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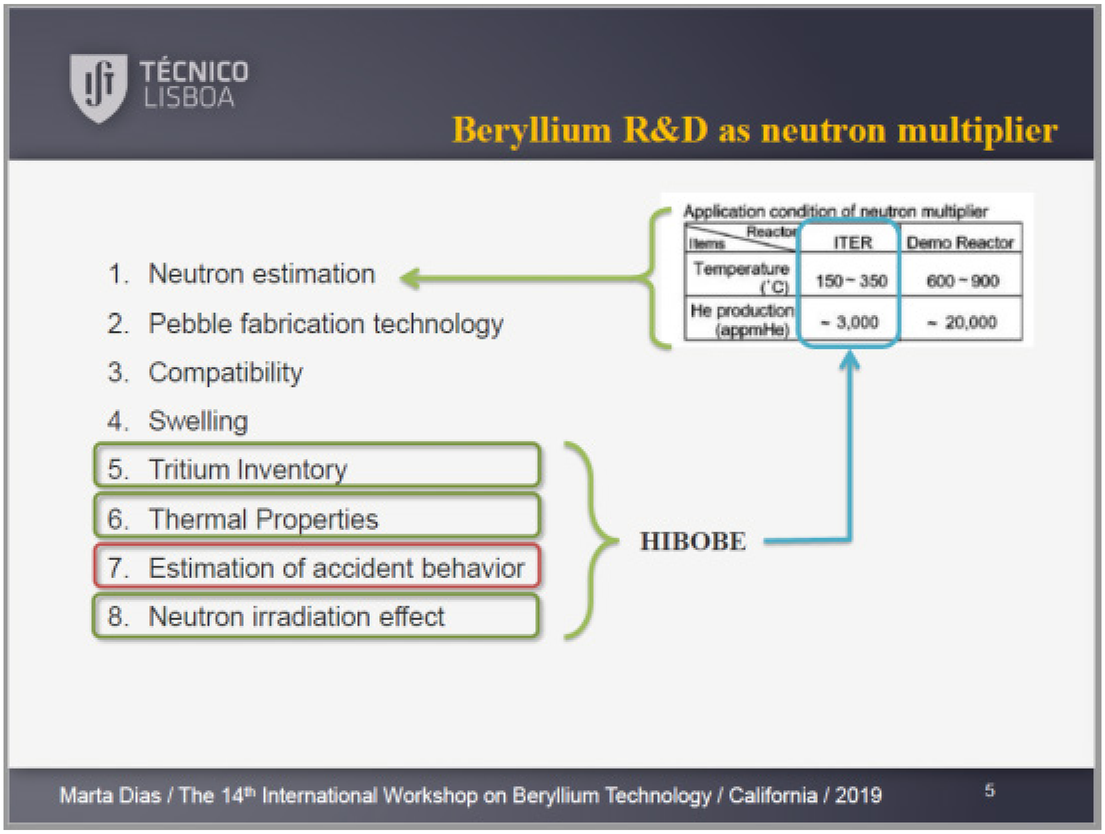
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Beryllium R&D as neutron multiplier
1. Neutron estimation
2. Pebble fabrication technology
3. Compatibility
4. Swelling
5. Tritium Inventory
6. Thermal Properties
7. Estimation of accident behavior
8. Neutron irradiation effect
HIBOBE
Application condition of neutron multiplier
Items / Reactor
ITER
Demo Reactor
Temperature (°C)
150 ~ 350
600 ~ 900
He production (appmHe)
~ 3,000
~ 20,000
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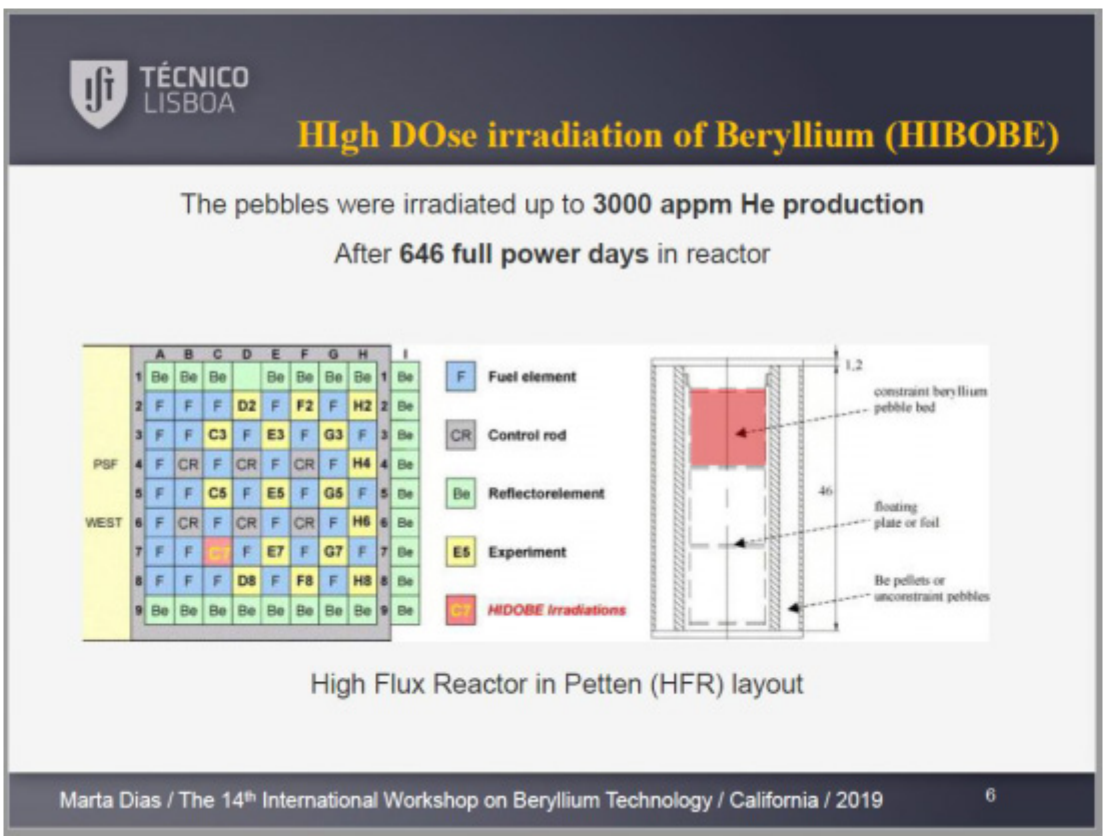
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HIgh DOse irradiation of Beryllium (HIBOBE)
The pebbles were irradiated up to 3000 appm He production
After 646 full power days in reactor
F Fuel element
CR Control rod
Be Reflectorelement
E5 Experiment
C7 HIDOBE Irradiations
PSF
WEST
constraint beryllium pebble bed
floating plate or foil
Be pellets or unconstraint pebbles
High Flux Reactor in Petten (HFR) layout
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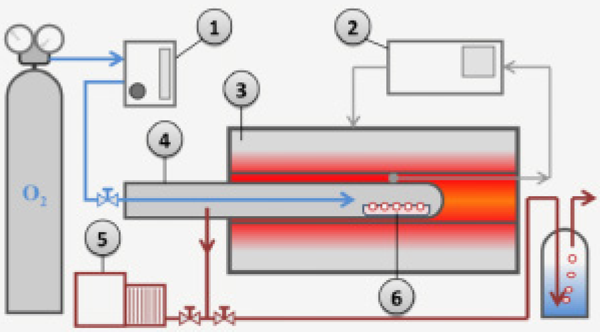
Oxidation Technique

$$Be + H_2O \rightarrow BeO + H_2 + heat$$



(1) Flow controller, (2) temperature controller, (3) tube furnace, (4) quartz tube, (5) vacuum pump, (6) sample.

Conditions:

T=700°C
Under :

(i) **Air atmosphere** with a relative humidity (H_2O water vapor) of 60 %

(ii) **40%N_2 + 60%O_2 mixture**

The annealing time steps, in both cases were : 1h, 3h, 8h, and 18h

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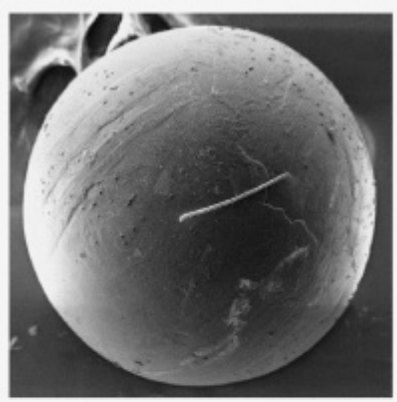
Scanning Electron Microscopy (SEM)



The SEM images are used to confirm the shape before and after oxidation.

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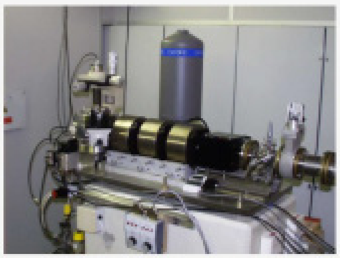
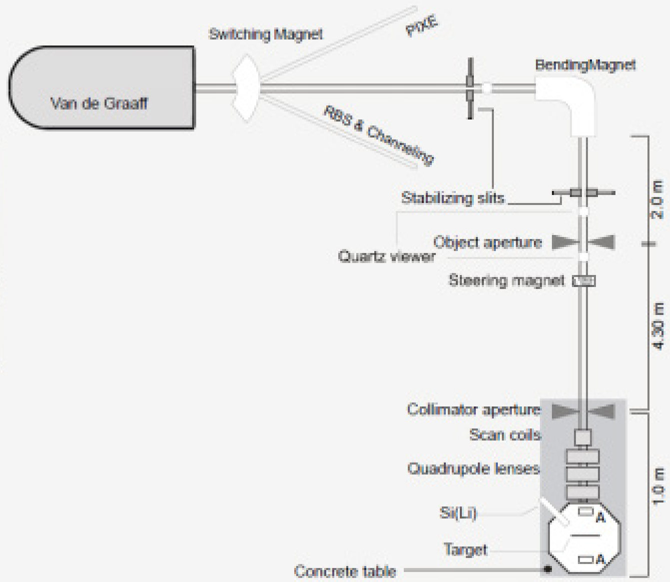
Ion beam Analysis (IBA)





Conditions:

He beam 3x4 μm²
Energy = 2 MeV

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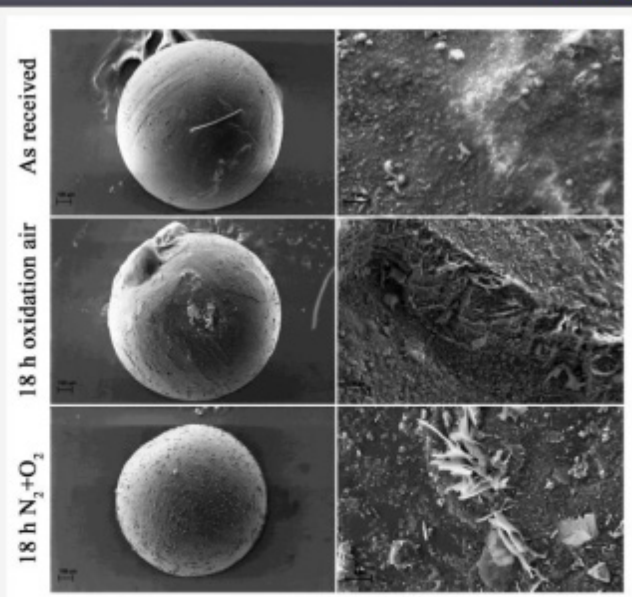
Results: SEM observations



The major features are the presence of small flakes covering the surface in both conditions.

The samples were only observed after the longer time (18h).

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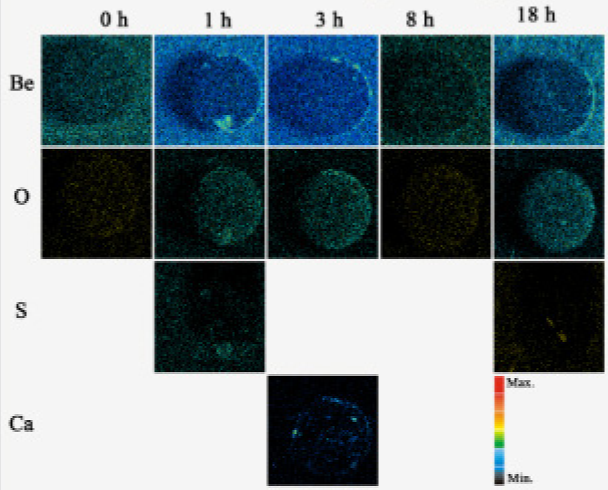
Results: IBA - Elemental Distribution maps

Samples oxidized with 40%N_2 + 60%O_2 mixture



The elemental distribution maps are obtained using PIXE or RBS spectra.

The results evidenced the presence of O and Be elements very homogeneous covering all the surface of the pebbles.

The results for the samples annealed at the air atmosphere are similar.

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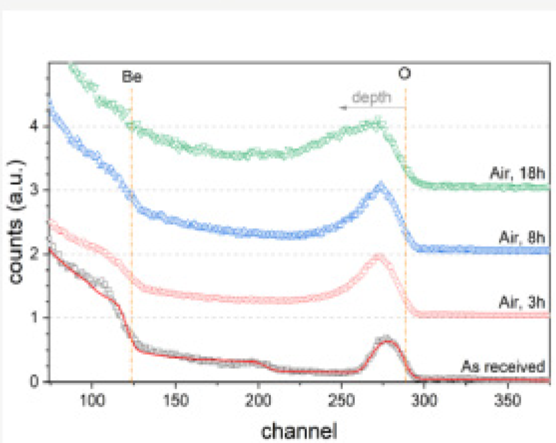
Results: RBS Spectra

Samples oxidized with air atmosphere



The height and width increase of the O peak with the increasing of the annealing time which indicates that the amount of oxygen and width of the oxide layer is increasing.

The behaviour of the curves for the samples oxidized with 60% O_2 + 40% N_2 atmosphere were similar to those evidenced for air oxidation.

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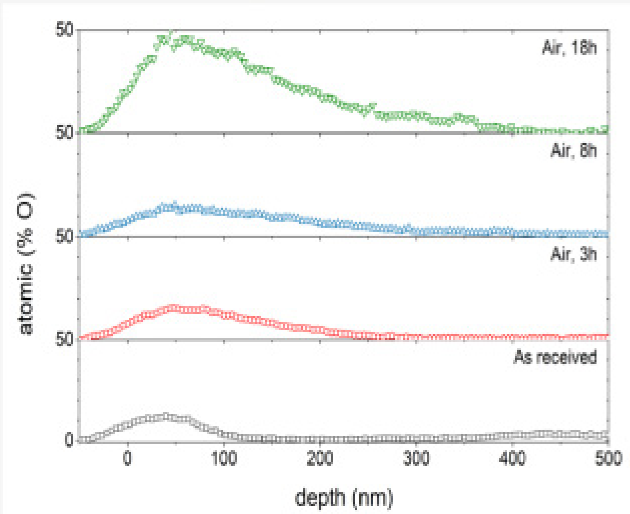
Results: Oxygen profiles results

Samples oxidized with air atmosphere



The results were analysed with the NDF code to extract the thickness of the oxide layer after each annealing step.

It was observed the formation of a stoichiometric BeO scale at the surface.

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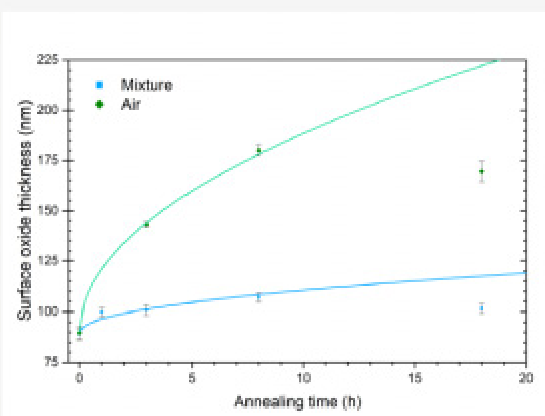
Results: Oxide Growth



Parabolic law $x = x_0 + (k\ t)^{1/2}$ where x_0 is the initial native oxide layer, k oxide rate constant and t the time.

Oxidation rater is notable and higher in the air atmosphere.

Annealing time	Mixture 60% O_2 + 40% N_2	Air
0 h	89 ± 2,7	90 ± 3,2
1 h	100 ± 2,3	-
3 h	101 ± 2,7	143 ± 1,6
8 h	107 ± 2,3	180 ± 2,9
18 h	102 ± 2,5	170 ± 5,3
Equation	$x = x_0 + (kt)^{1/2}$	
x_0	90 ± 1	90 ± 1
k	42 ± 9	970 ± 27

Thickness of the oxygen elemental profile versus the annealed time.

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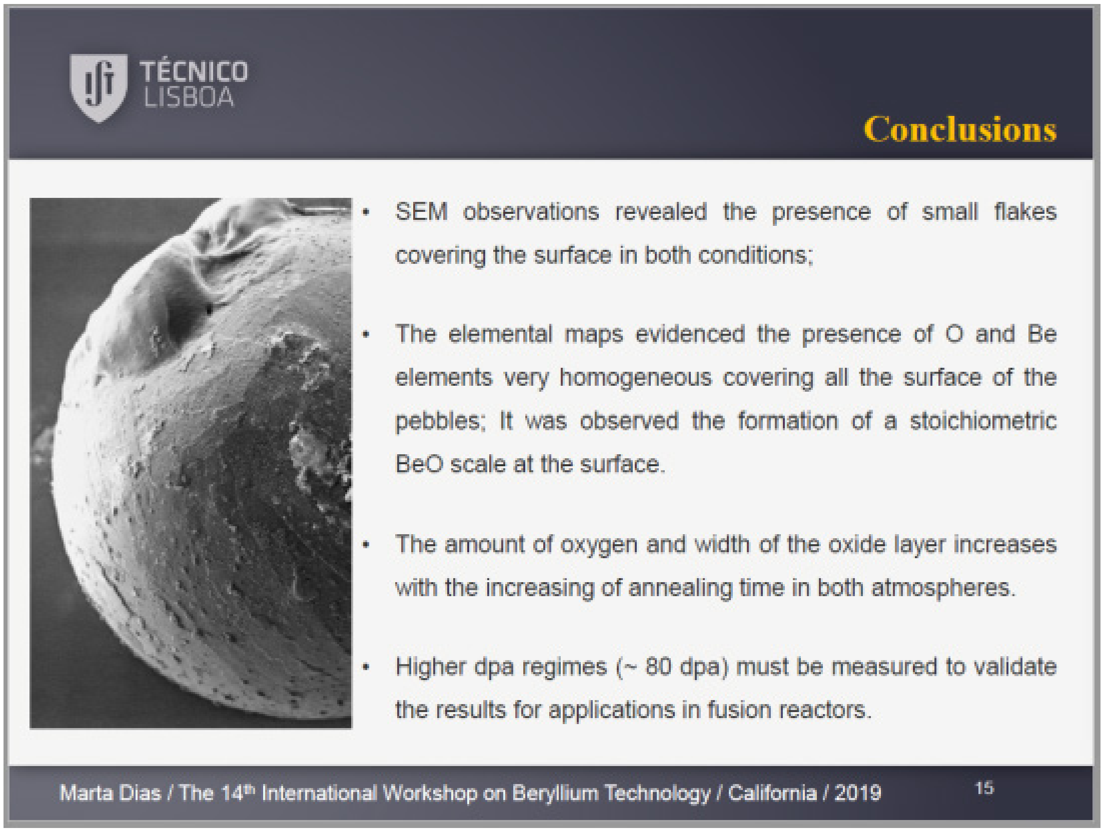
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Conclusions
• SEM observations revealed the presence of small flakes covering the surface in both conditions;
• The elemental maps evidenced the presence of O and Be elements very homogeneous covering all the surface of the pebbles; It was observed the formation of a stoichiometric BeO scale at the surface.
• The amount of oxygen and width of the oxide layer increases with the increasing of annealing time in both atmospheres.
• Higher dpa regimes (~ 80 dpa) must be measured to validate the results for applications in fusion reactors.
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Compatibility of Neutron-Multiplier Materials with Structural Materials
R. Rolli (KIT, Germany) et al.

Compatibility of NMM with structural materials

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Abstract

Solid tritium-breeding blankets with lithium ceramics as breeder and steel as structural material require beryllium as neutron multiplier to increase the Tritium Breeding Ratio (TBR) performance. In the base line HCPB blanket design of ITER, beryllium will be used in the form of a single-size pebble bed, with pebbles of 1mm diameter produced by the Rotating Electrode Method (REM) by NGK, Japan.

Beryllium pebble beds used for neutron multiplication in tritium breeding blanket are placed within boxes made of ferritic-martensitic steel EUROFER, so that a direct contact between boundary pebbles and steel walls is considered.

Even at the maximum working temperature of beryllium pebble beds of 650°C, no significant interaction of pebbles with steel should occur. On the other hand, it is known that interstitial beryllium atoms can quickly diffuse in iron forming various brittle intermetallic phases. Formation of such phases could result in local reduction of steel ductility and increased risk of brittle fracture induced by impact loads.

According to the updated design of breeding blanket for the DEMO fusion reactor, solid $Be_{12}Ti$ blocks are to be used for neutron multiplication. In comparison with pure beryllium, titanium beryllide has higher heat and corrosion resistance. It swells less and retains less tritium under irradiation. In the new design, $Be_{12}Ti$ blocks should not contact directly with EUROFER steel, but it could occur accidently. Considering much higher working temperature of beryllide, at about 900°C the interaction with steel again may cause iron beryllides formation in steel.

Therefore, compatibility tests of beryllium and $Be_{12}Ti$ with EUROFER over long time were performed, and subsequent examinations by optical investigation of the interaction layer were documented. In this study, one will show the results of experiments, which were done in the Fusion Materials Laboratory at KIT.

Keywords: Beryllium; NMM; EUROFER; Beryllide

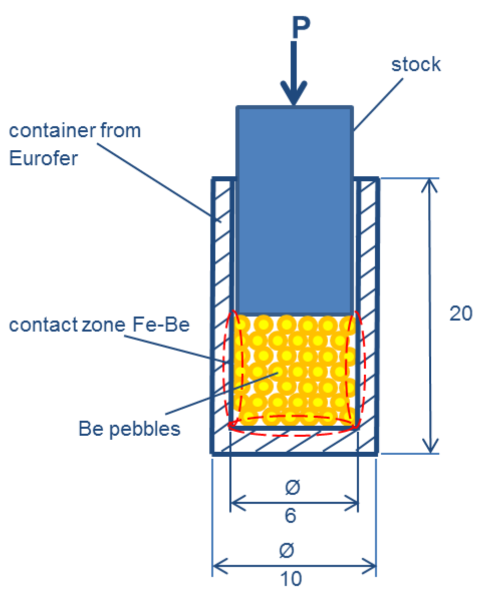
1 Introduction

Due to safety considerations, the experiments on compatibility of Be-containing material with EUROFER97 should be performed within the hot cells in a dedicated facility allowing maximum test temperature of 900°C. Pilot test have shown that very long annealing time is required to reveal possible interactions at 650-700°C. Therefore, it is proposed to perform tests at higher temperatures (800-900°C) and evaluate beryllium diffusion coefficient in EUROFER97 by means of measuring of a thickness of the interaction layer. This approach would allow extrapolation to lower temperature and to longer annealing time. As far as initially uncompressed pebble beds become compressed due to irradiation-induced swelling, the effect of external loading on formation of the interaction layer will be investigated. Various batches of beryllium pebbles as well as beryllides plates will be tested.

2 Be pebbles and EUROFER97 compatibility tests

Compatibility studies on REM 1mm beryllium pebbles were performed according the scheme, which is shown in Fig. 1. The plates from EUROFER97 were placed in the holder with beryllium pebbles. The pebble bed was loaded by the piston from upper part of the bed. The compatibility tests were performed at temperatures of 600-900°C for times of 0.5, 100, 400 h at different loadings.

(a)



(b)

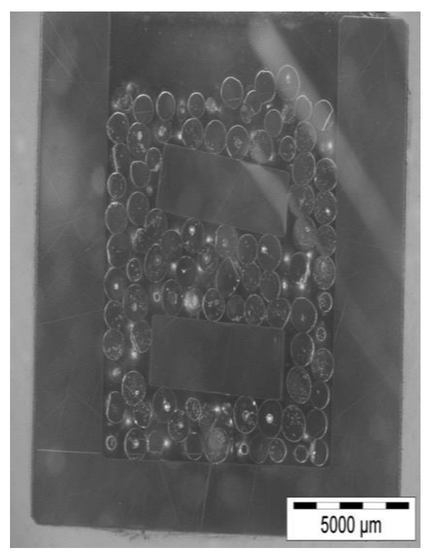


Figure 1. The scheme of compatibility experiment of Be pebbles with EUROFER97 (a) and cross section of a set-up after experiment (b).

Degree of interaction between beryllium pebbles and EUROFER97 depends on parameters of the tests. Examples of the compatibility tests are shown in Figs. 2-3. Increasing temperature, time, or loading give an increase to the thickness of the interaction layer up to 100µm (Table 1). The effect of pore formation occurs in some beryllium pebbles during the compatibility tests (Fig. 4). The reason for this effect might be quite high loading.

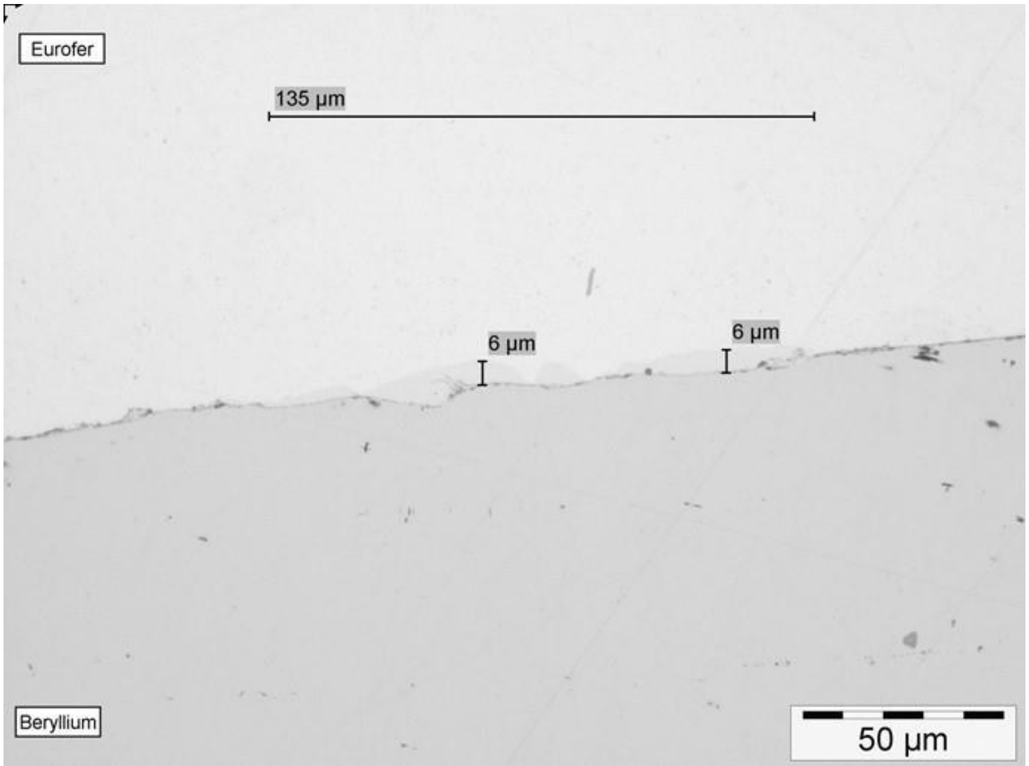


Figure 2. Interaction layer in the compatibility test "Be pebble-Eurofer97" at 700°C at 600 N for 100 h. (O)

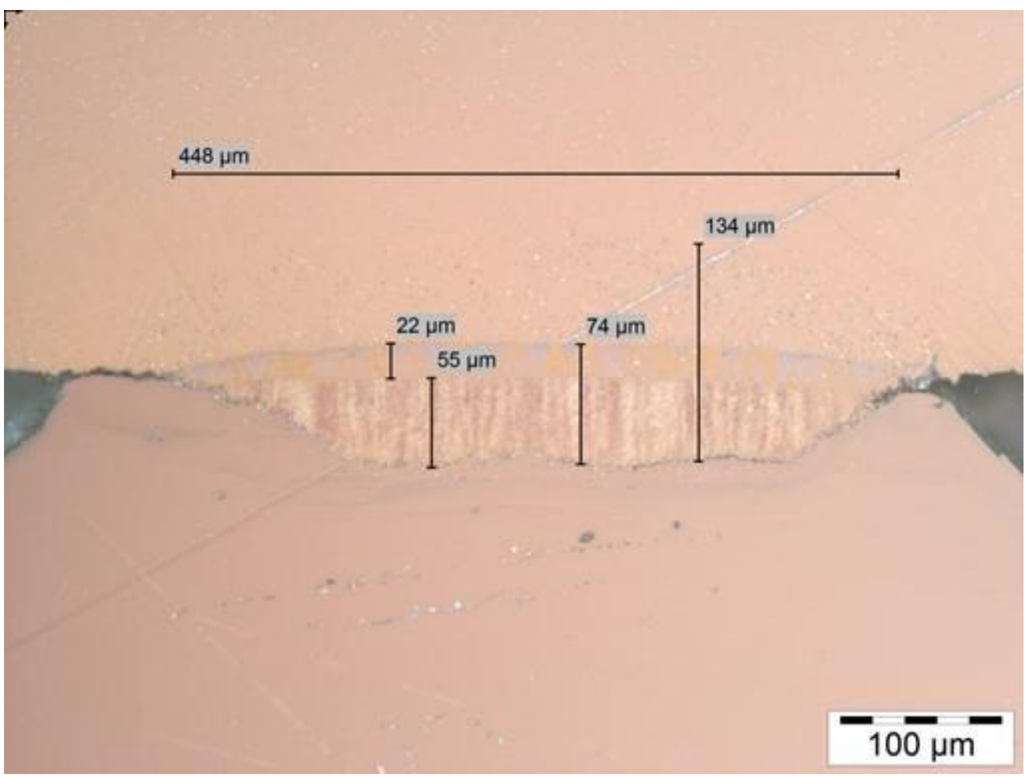


Figure 3. Interaction layer in the compatibility test "Be pebble-Eurofer97" at 800°C at 400 N for 400 h. (OM)

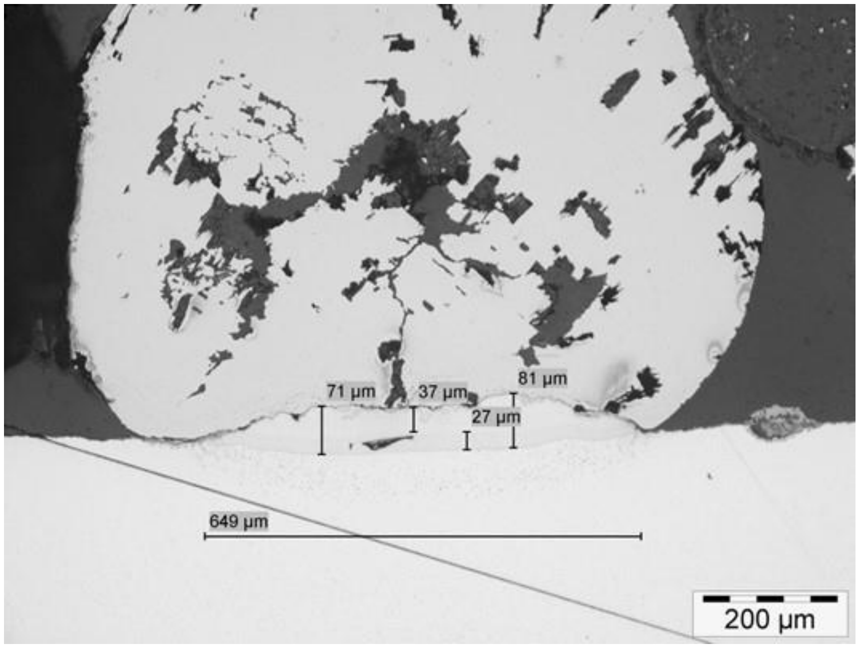


Figure 4. Porosity formation in the Be pebbles during compatibility test "Be pebble-EUROFER97" at 800°C at 400 N for 400 h. (OM)

Table 1: Summary of the interaction layer thickness during compatibility tests "Be pebble- EUROFER97" for different temperature, time, loading values.

Temperature, °C	Time, h	Loading, N	Interaction layer, µm
600	100	600	None
600	400	600	None
700	100	600	~6
700	400	600	~13
800	100	400	~100
800	400	400	~100
900	0.5	50	None

3 Be-Ti composite and EUROFER97 plates compatibility tests

Be-Ti composite plates having 7×7×2 mm^3 were cut using electrical discharge machining from a sample obtained by powder metallurgy. A mixture of Be and 30.8 wt. % Ti powders was extruded at elevated temperature to consolidate the powders. Extruded samples were subjected to hot isostatic pressing (HIP) at 1000°C and 1000 bar for 4 h to synthesize $Be_{12}Ti$ phase. Fig. 5 represents the microstructure of Be-Ti composite after extrusion and HIP. Despite the stoichiometric content of beryllium, it did not fully react with Ti forming beryllide, and the structure is formed of 90% of $Be_{12}Ti$ and 10% of Be phase. Contact surfaces of the plates for compatibility test were perpendicular to extrusion direction.

All contact surfaces were ground and mechanically polished before tests. EUROFER97 plates were cut and prepared in the same way. After tests the container and plates were embedded to electro-conductive resin. The plates were cut along compression direction along the diameter of the container. The surface of contact zone was ground and mechanically polished. Optical microscopy (OM) and scanning electron microscopy (SEM) with energy-dispersive x-ray spectroscopy (EDX) were used to study microstructures.

Compatibility tests of Be-Ti composite and EUROFER97 plates were performed according the scheme shown in Fig. 6. The plates from Be-Ti composite were placed between EUROFER97 plate and stainless-steel container. The compatibility tests were performed at temperatures of 700 and 900°C for time of 200 h at 500 or 1000 N loadings, which corresponds approximately to 10 or 20MPa.

(a) (b)

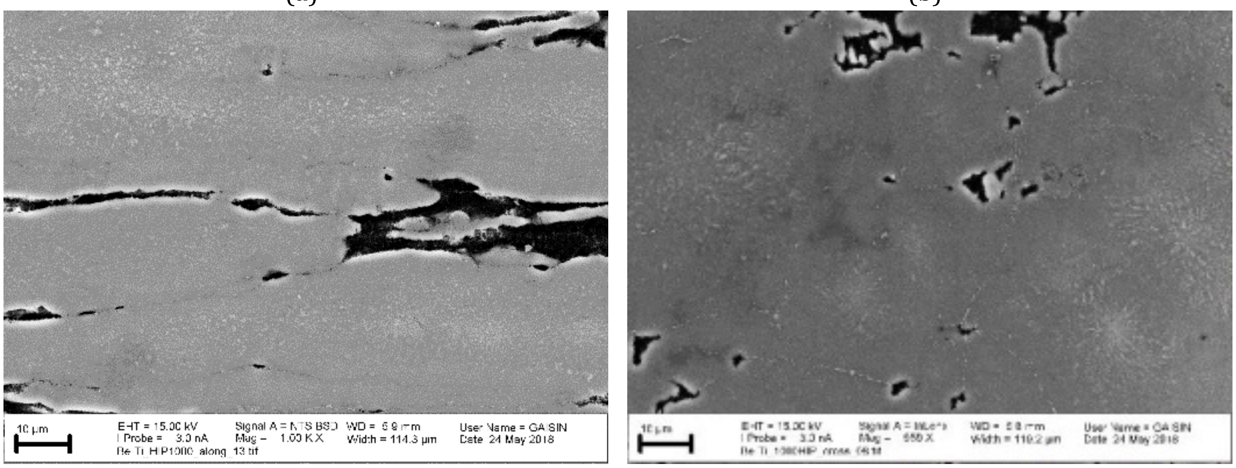


Figure 5. Microstructure of Be-Ti composite after powder extrusion and HIP at 1000°C: (a) along and (b) across extrusion direction. Grey areas correspond to $Be_{12}Ti$ phase, black particles correspond to Be. (SEM)

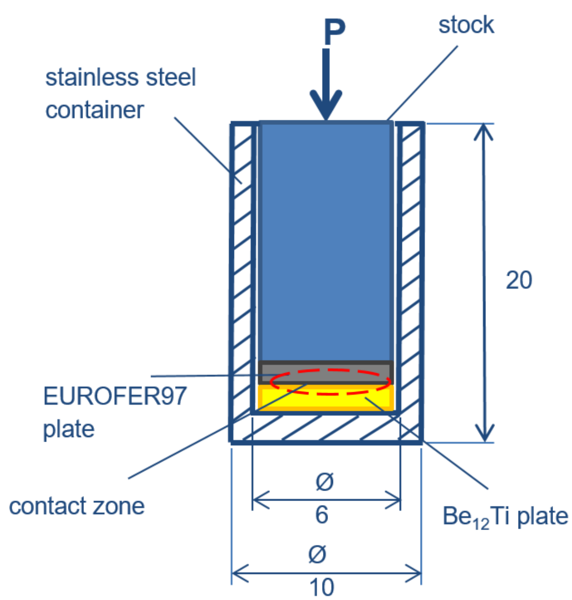


Figure 6. The scheme of compatibility experiment of the Be-Ti composite and EUROFER97 plates.

Fig. 7 represents the microstructure of the Be-Ti composite and the EUROFER97 contact zone after compression test at 700°C, 10MPa (500N) for 200 h. No interaction between the materials was found after the test. Taking into account the high melting point of titanium beryllide and low diffusivity, the test temperature of 700°C seems to be too low to have any interaction effect for a limited test duration. Other tests were conducted at the highest possible test temperature of 900°C.

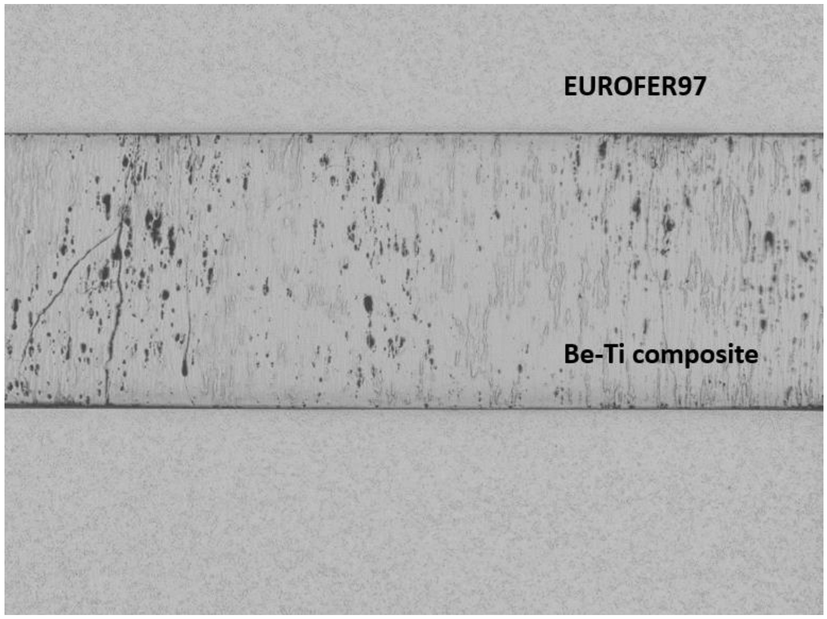


Figure 7. Microstructure of contact zone of Be-Ti composite and EUROFER97 after compression test at 700°C, 10MPa (500N), 200 h. (OM)

Fig. 8 represents the panoramic overview of the interaction zone between Be-Ti composite and EUROFER97 after compression test at 900°C, 10MPa (500N) for 200 h. With temperature increase, the interaction occurred between materials in the form of several point interactions (Fig. 9a) whereas most of the contact zone had only limited or no apparent interaction (Fig. 9b). The thickness of interaction layer in some points reaches 138µm.

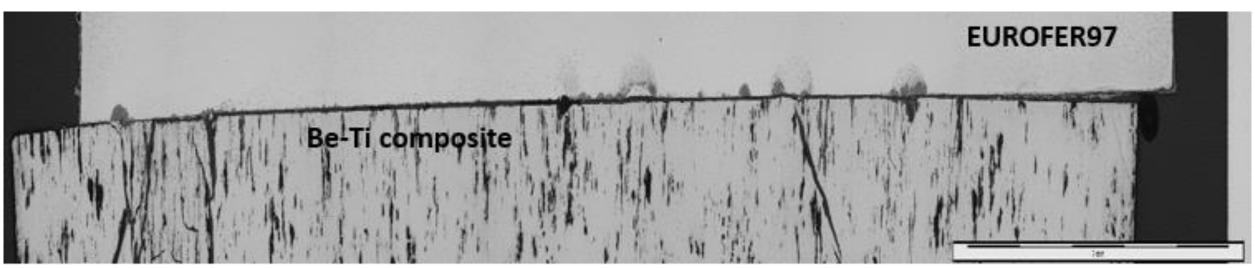


Figure 8. Panoramic overview of the interaction zone between Be-Ti composite and EUROFER97 after compression test at 900°C, 10 MPa (500 N), 200 h. (OM)

Energy-dispersive x-ray spectroscopy maps of Fe, Cr, W, V and Ti distributions do not show any diffusion of these elements from the steel to Be-Ti composite and vice versa. Unfortunately, beryllium cannot be reliably detected by EDX, since low energy characteristic x-ray of Be is known to be mostly absorbed by the sample. Be can be detected indirectly on Fe and Ti distribution maps (Fig. 10). Fig. 10a shows, that there is almost no iron in Be-Ti composite and iron concentration is less in the interaction zone in comparison with the rest EUROFER97.

Since titanium does not diffuse into steel (Fig. 10b), a lower iron content in the interaction layer can only be explained by the diffusion of beryllium atoms from the Be-Ti composite into steel. Beryllium has high bonding energy with Ti in the $Be_{12}Ti$ phase, and therefore beryllium most likely diffuses from the beryllium phase into steel and not from beryllide phase. Fig. 10b shows lower amount of Ti in the central part of Be-Ti composite.

Apparently, beryllium phase reached the surface of the contact zone there and caused the point interaction owing to diffusion of Be atoms into EUROFER97. In areas without beryllium on the contact surface, no interaction is observed (Fig. 9b). With the aim to obtain a more intense interaction of titanium beryllide with steel, the third test was carried out under the same conditions, but at a higher pressure.

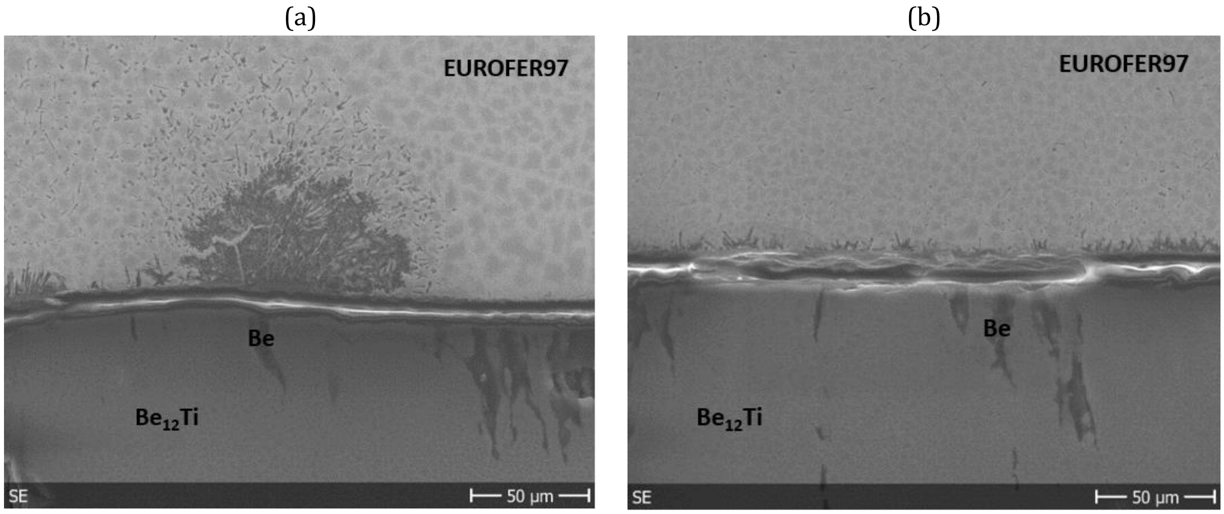


Figure 9. Microstructure of the interaction zone between Be-Ti composite and EUROFER97 after compression test at 900°C, 10MPa (500N), 200 h: (a) point interaction, (b) no interaction. (SEM)

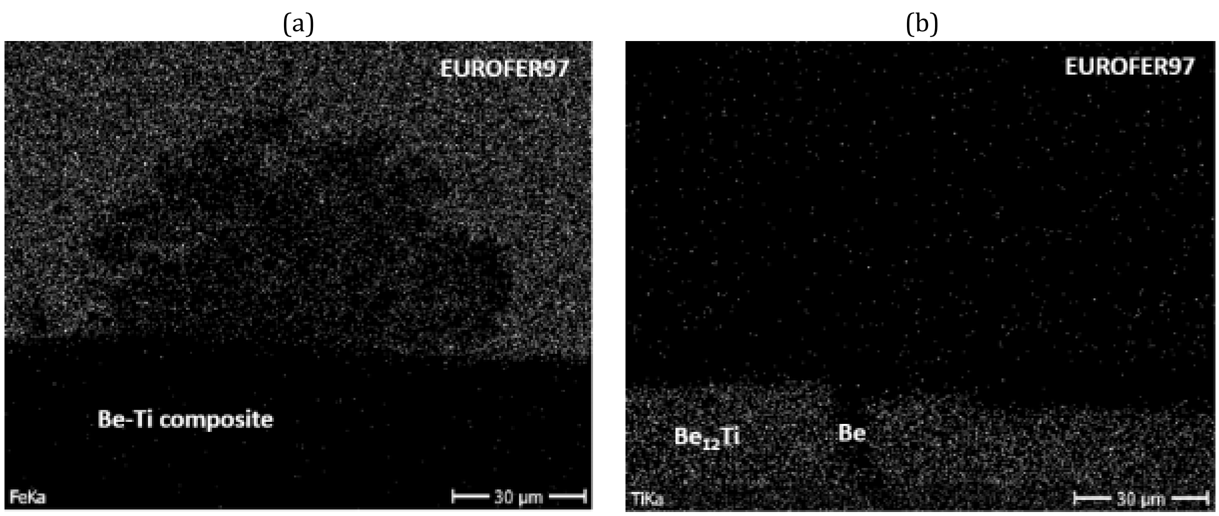


Figure 10. EDX maps of the point interaction zone between Be-Ti composite and EUROFER97 after compression test at 900°C, 10MPa (500N), 200 h: (a) Fe and (b) Ti elemental maps.

Fig. 11 represents the panoramic overview of the interaction zone between Be-Ti composite and EUROFER97 after compression test at 900°C, 20MPa (1000N) for 200 h. With pressure increase, continuous interaction layers (Fig. 12a) were observed as well as several point interactions (Fig. 12b) and no interaction zones (Fig. 12c). The thickness of the continuous layer reaches 47μm, the whole interaction zone is up to 150μm in depth.

Linear energy-dispersive x-ray spectroscopy showed no titanium and lower iron in the interaction layer (Fig. 13). Higher pressure resulted in better contact of materials and better diffusion of Be atoms from Be-Ti composite into EUROFER97. Since there is no interaction at many sites (Fig. 12c), it is likely that beryllium diffuses into the steel from the beryllium phase. The pressure increase led to an increase in the interaction area, while the maximum depth of the interaction zone did not change significantly. The conducted tests show that with the aim to eliminate the interaction of Be-Ti blocks with EUROFER97, the amount of beryllium phases should be reduced to a minimum or a single beryllide-phase material should be obtained.

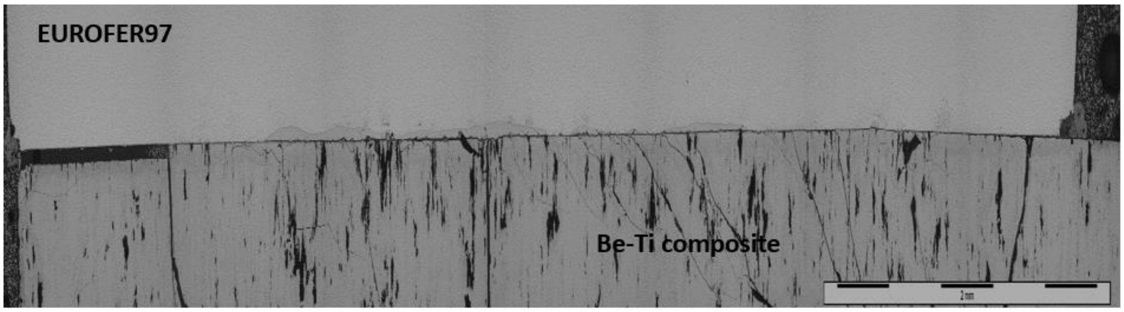


Figure 11. Panoramic overview of the interaction zone between Be-Ti composite and EUROFER97 after compression test at 900°C, 20MPa (1000N), 200 h. (OM)

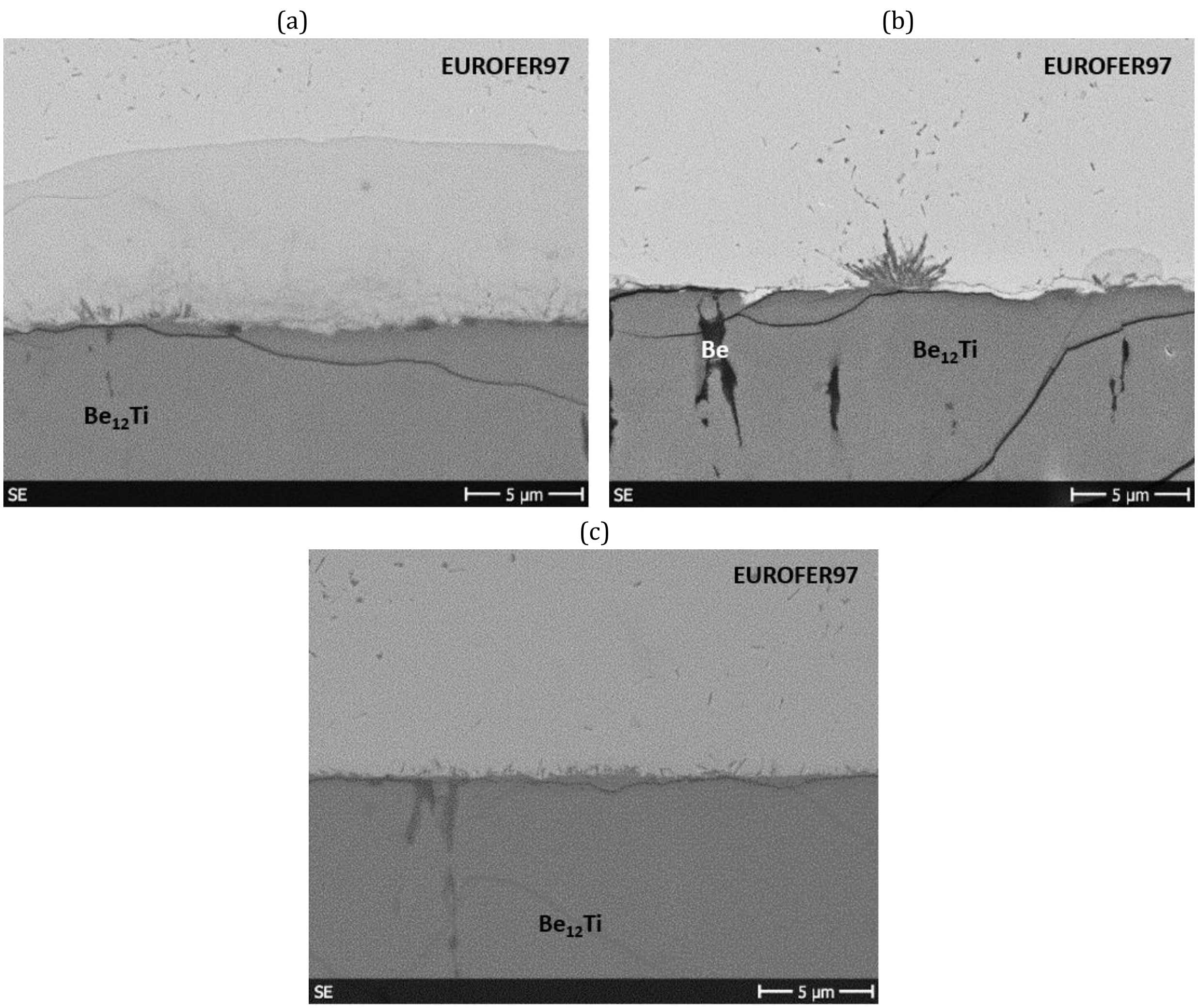
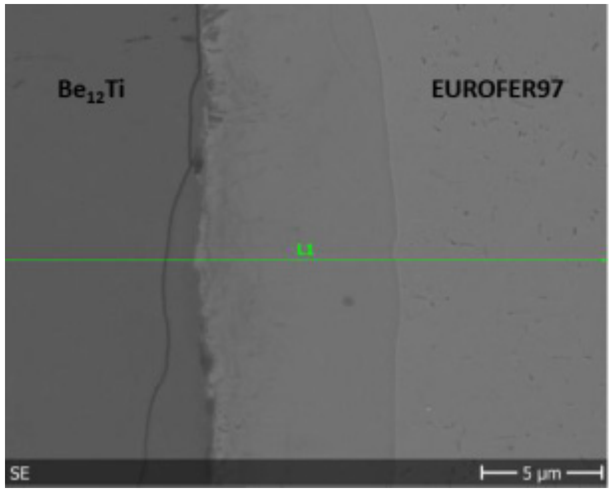
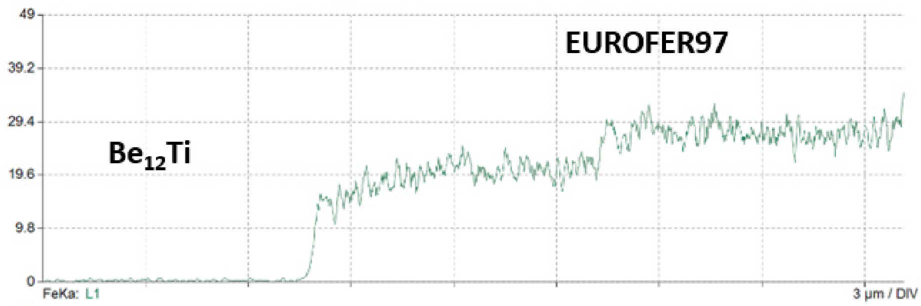


Figure 12. Microstructure of the interaction zone between Be-Ti composite and EUROFER97 after compression test at 900°C, 20MPa (1000N), 200 h: (a) continuous layer, (b) point interaction, (c) no interaction. (SEM)

(a)



(b)



(c)

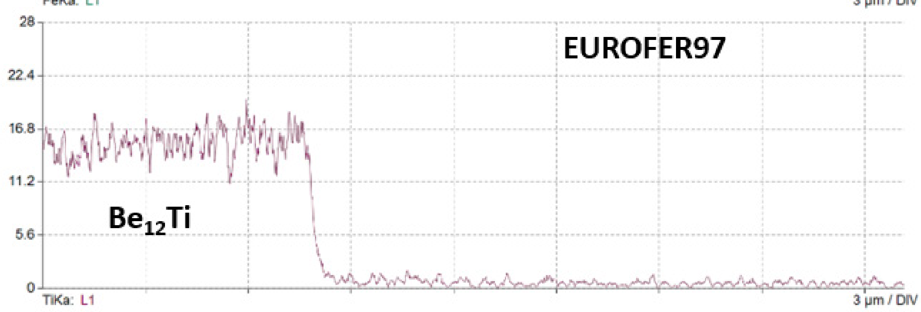


Figure 13. EDX linear analysis across interaction zone between Be-Ti composite and EUROFER97 after compression test at 900°C, 20MPa (1000N), 200 h: (a) SEM, (b) Fe, and (c) Ti elemental distributions.

4 Conclusion

Interaction between beryllium pebbles and EUROFER97 depends on parameters of the tests. With temperature and load increase interaction layer reaches 100µm in depth.

Compatibility tests of Be-Ti composite and EUROFER97 plates showed that interaction is mainly caused by diffusion of Be atoms from beryllium phase in the composite. There was no interaction layer after test at 700°C, 10MPa for 200 h. With temperature increase up to 900°C, continuous interaction layer or point interactions were observed. The maximum depth of the interaction zone reaches 150µm. Titanium beryllide in some zones does not interact with steel even at the highest possible temperature and load test.

References

[1] K.A. Walsh, Beryllium Chemistry and Processing, ASM International, 2009.

[2] R.M. Hunt, S.H. Goods, A. Ying, C.K. Dorn, M. Abdou. Diffusion bonding beryllium to Reduced Activation Ferritic Martensitic steel: Development of processes and techniques. Fusion Engineering and Design 87, 9 (2012) pp. 1550–1557.

[3] A. Amulevicius, K. Mazeika, C. Sipavicius, A. Baltusnikas. Studies of Be-Fe Compounds Synthesized by Laser Irradiation. Acta Physica Polonica A. 108, 3 (2005) pp. 467-478.

[4] H. Okamoto, L.E. Tanner, D.E. Peterson. Iron-Beryllium Phase Diagram, Phase Diagrams of Binary Iron Alloys, ASM International, 1993.

[5] R. Gaisin, V. Chakin. Results on compatibility of Be MM with structural materials. WPBB-DEL- BB-1.3.5-T004-D001, 2019.

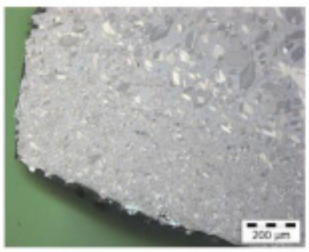
14th IEA International Workshop on Beryllium Technology
Long Beach, USA, October 24-25, 2019

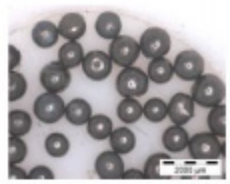
Karlsruhe Institute of Technology

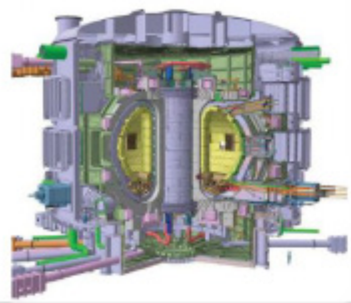
COMPATIBILITY OF NMM WITH STRUCTURAL MATERIALS

Karlsruhe Institute of Technology: R. Rolli, R. Gaisin, V. Chakin, P. Vladimirov, H.-C. Schneider

INSTITUTE OF APPLIED MATERIALS – MECHANICS OF MATERIALS AND BIOMECHANICS
(IAM-WBM-FML) Fusionmateriallaboratory







Outline

- Introduction
- Installation
- Experiments EUROFER - Be
  - Experimental results
  - Surface investigation
- Experiments EUROFER - BeTi
  - Experimental results
  - Surface investigation
- Conclusions

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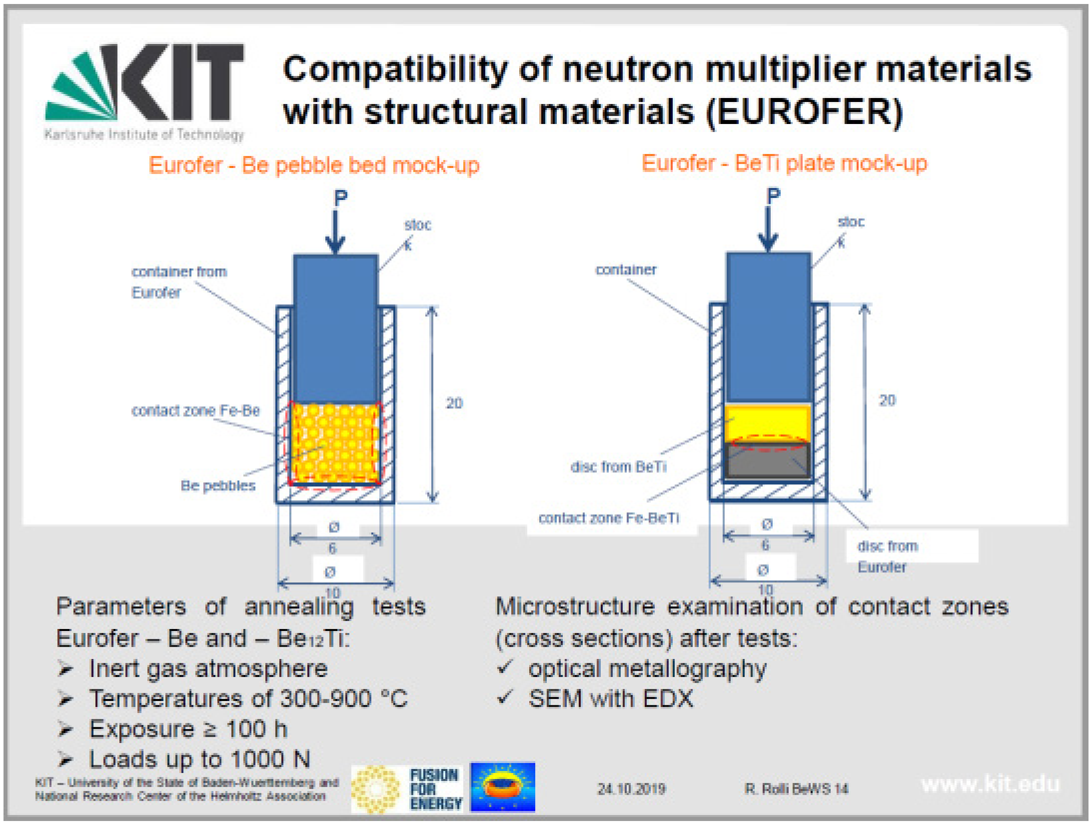
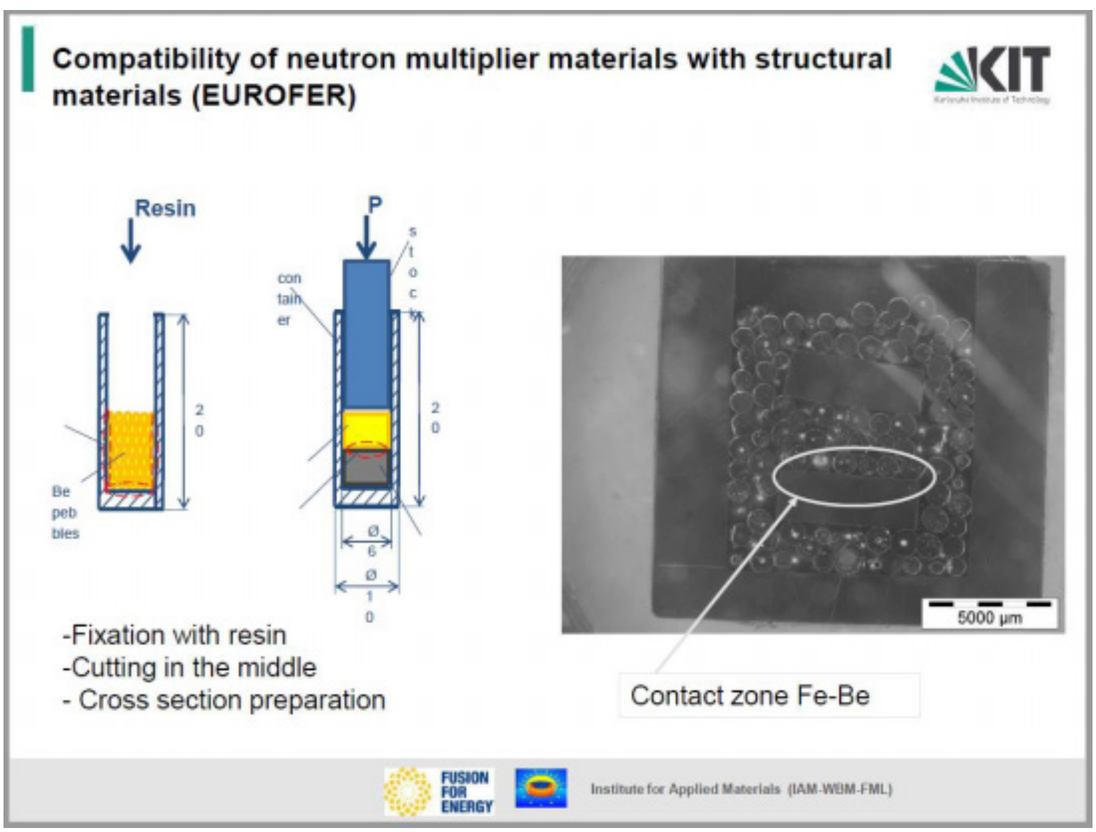

Karlsruhe Institute of Technology
Compatibility of neutron multiplier materials with structural materials (EUROFER)
Eurofer - Be pebble bed mock-up
Eurofer - BeTi plate mock-up
P
container from Eurofer
contact zone Fe-Be
Be pebbles
20
container
disc from BeTi
contact zone Fe-BeTi
disc from Eurofer
Parameters of annealing tests Eurofer – Be and – Be12Ti:
➢ Inert gas atmosphere
➢ Temperatures of 300-900 °C
➢ Exposure ≥ 100 h
➢ Loads up to 1000 N
Microstructure examination of contact zones (cross sections) after tests:
✓ optical metallography
✓ SEM with EDX
KIT – University of the State of Baden-Wuerttemberg and National Research Center of the Helmholtz Association
FUSION FOR ENERGY
24.10.2019
R. Rolli BeWS 14
www.kit.edu


Compatibility of neutron multiplier materials with structural materials (EUROFER)
Resin
P
Be pebbles
-Fixation with resin
-Cutting in the middle
- Cross section preparation
5000 µm
Contact zone Fe-Be
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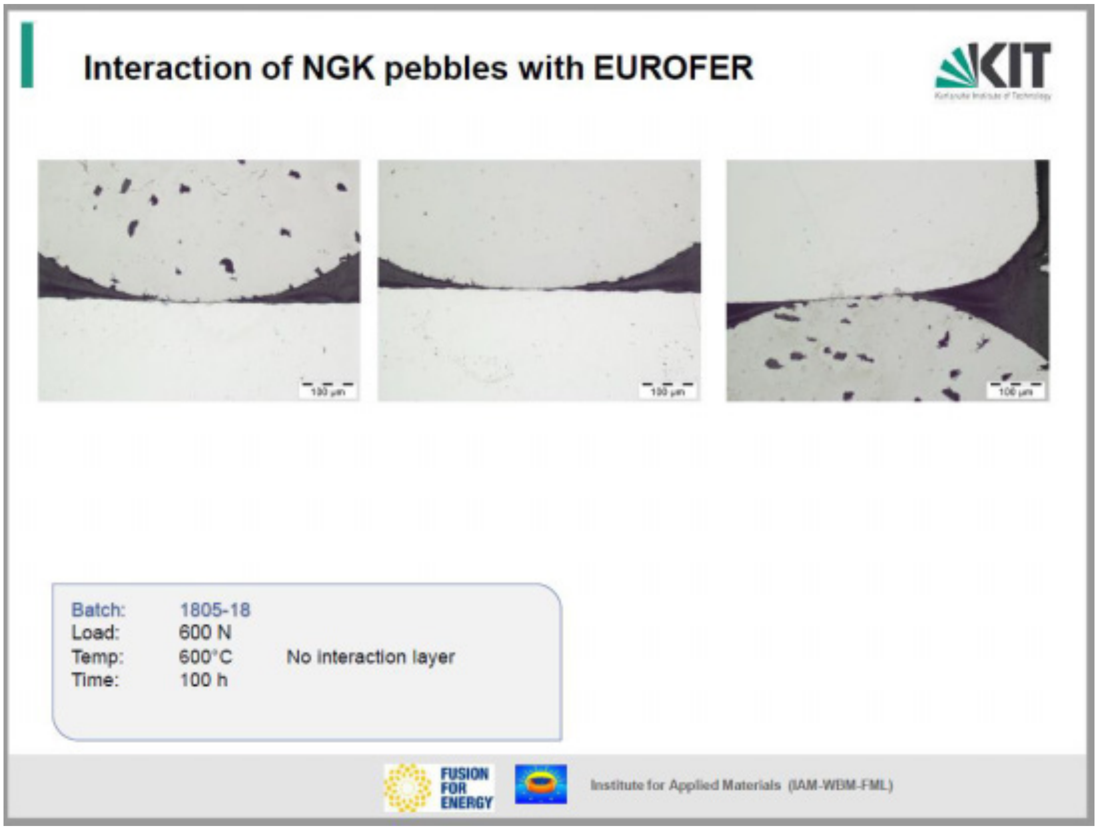
Institute for Applied Materials (IAM-WBM-FML)


Interaction of NGK pebbles with EUROFER
KIT
Batch: 1805-18
Load: 600 N
Temp: 600°C
Time: 100 h
No interaction layer
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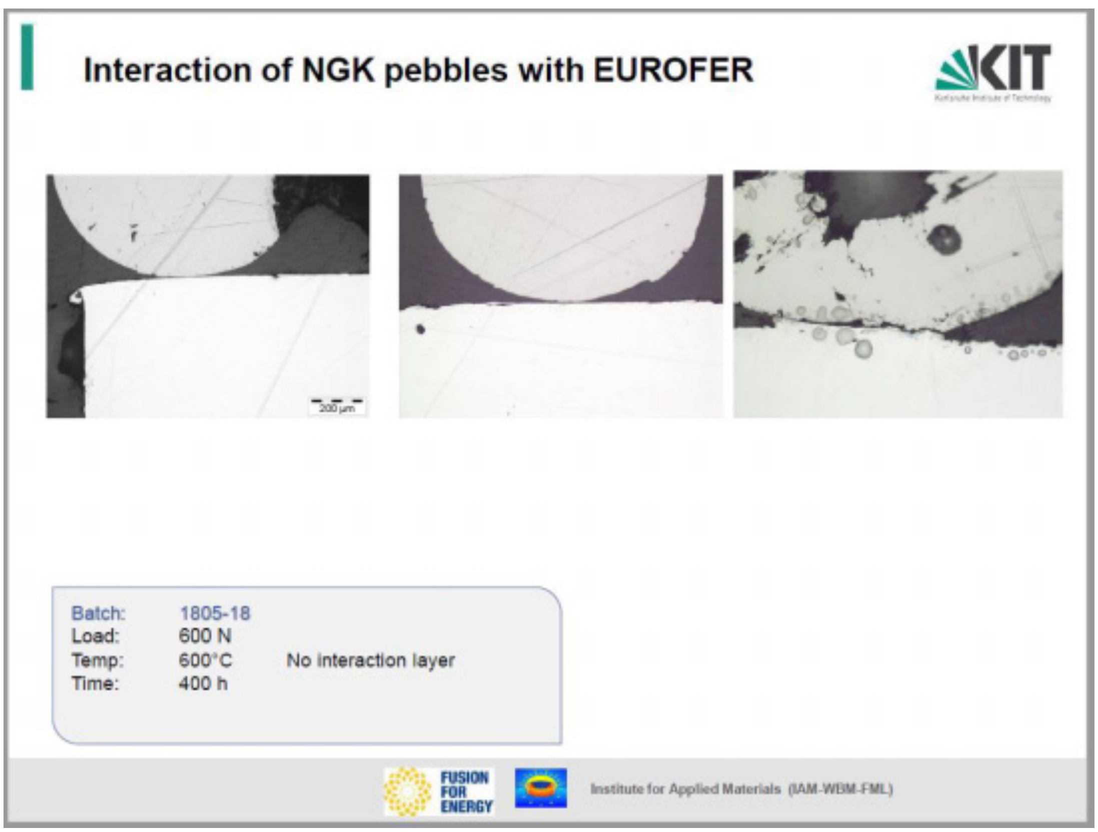
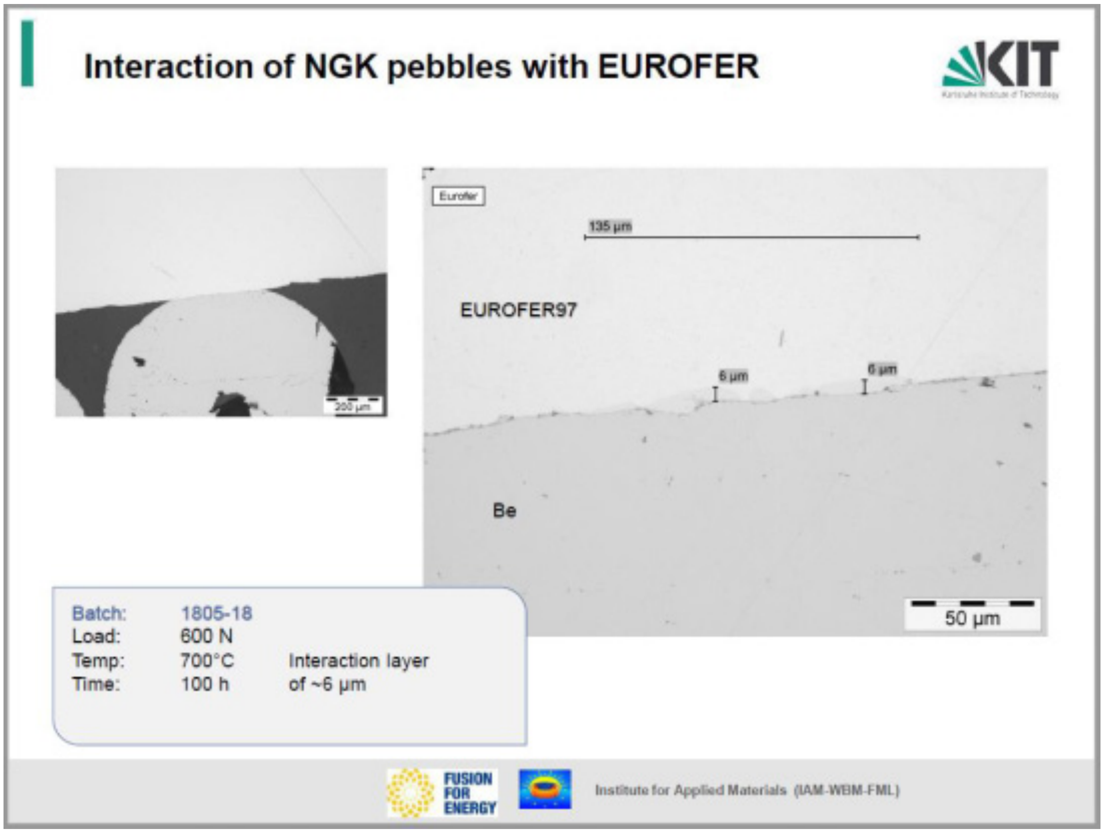
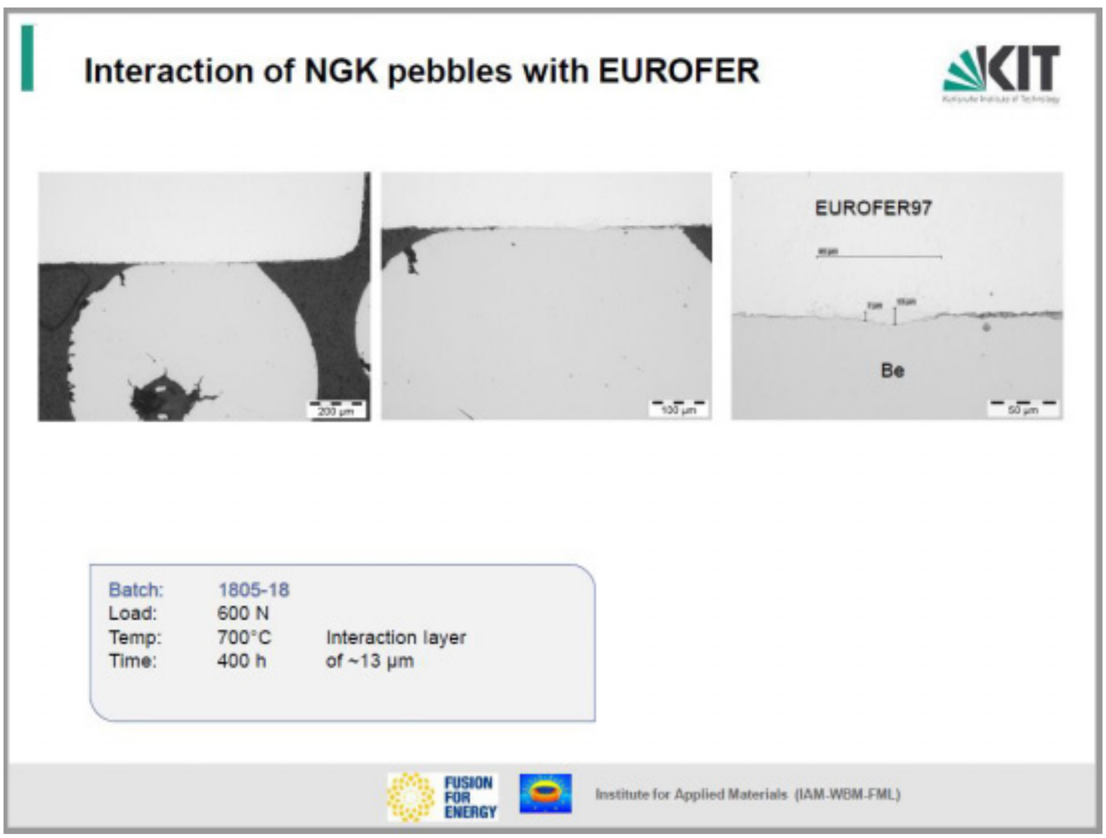
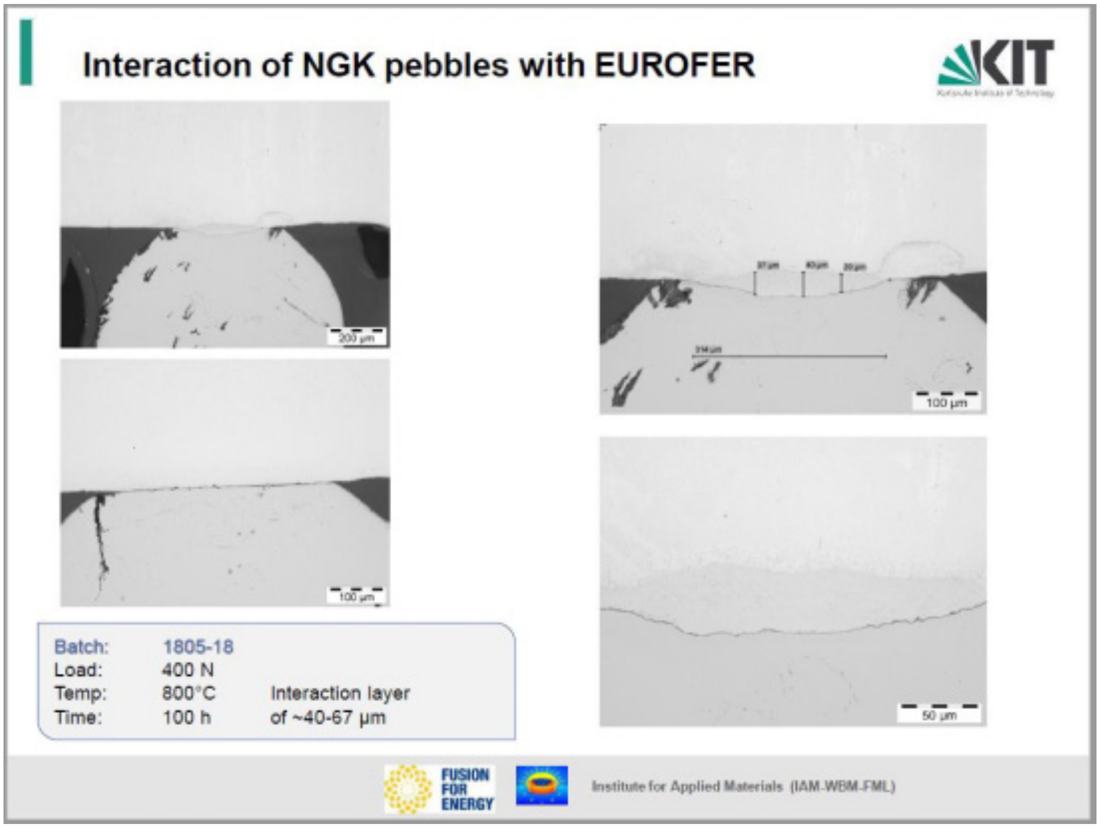
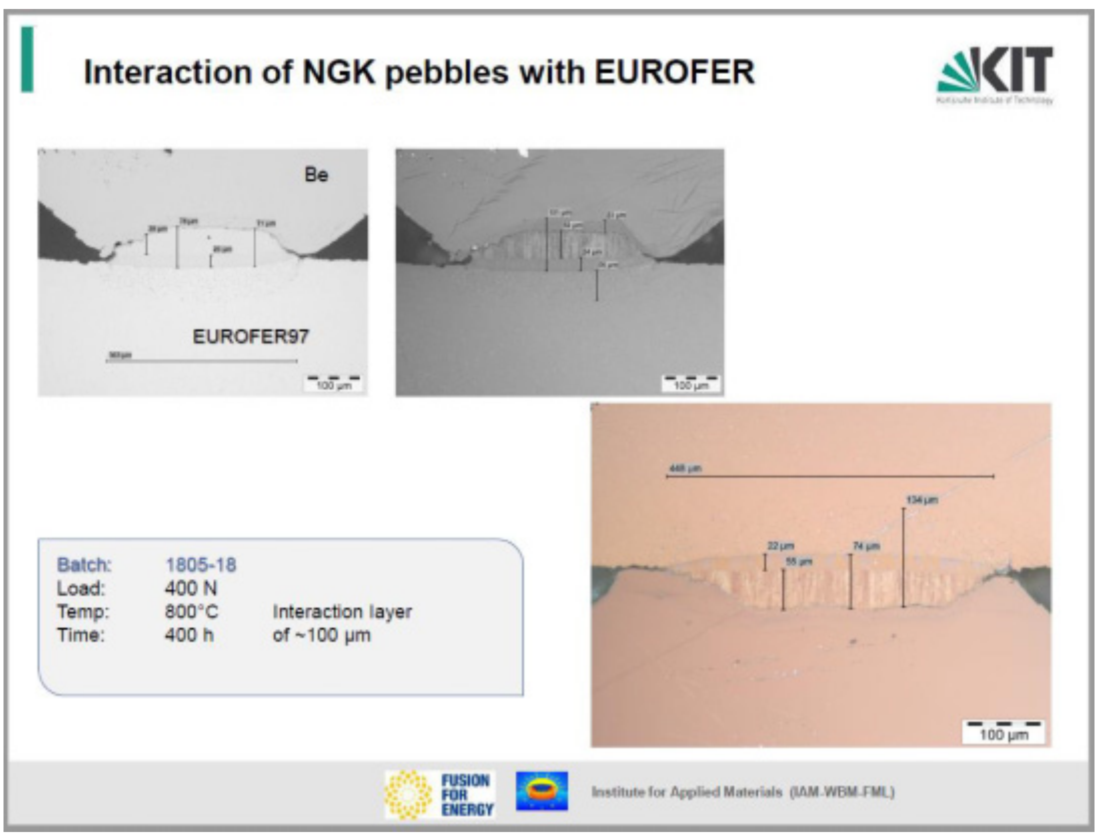
Institute for Applied Materials (IAM-WBM-FML)


Interaction of NGK pebbles with EUROFER
KIT
Batch: 1805-18
Load: 600 N
Temp: 600°C
Time: 400 h
No interaction layer
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)


Interaction of NGK pebbles with EUROFER
KIT
Eurofer
135 µm
EUROFER97
6 µm
6 µm
Be
200 µm
50 µm
Batch: 1805-18
Load: 600 N
Temp: 700°C
Time: 100 h
Interaction layer of ~6 µm
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)


Interaction of NGK pebbles with EUROFER
KIT
200 µm
100 µm
EUROFER97
Be
50 µm
Batch: 1805-18
Load: 600 N
Temp: 700°C
Time: 400 h
Interaction layer of ~13 µm
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)


Interaction of NGK pebbles with EUROFER
KIT
200 µm
100 µm
100 µm
50 µm
Batch: 1805-18
Load: 400 N
Temp: 800°C
Time: 100 h
Interaction layer
of ~40-67 µm
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)


Interaction of NGK pebbles with EUROFER
KIT
Be
EUROFER97
100 µm
100 µm
448 µm
134 µm
22 µm
74 µm
55 µm
100 µm
Batch: 1805-18
Load: 400 N
Temp: 800°C
Time: 400 h
Interaction layer
of ~100 µm
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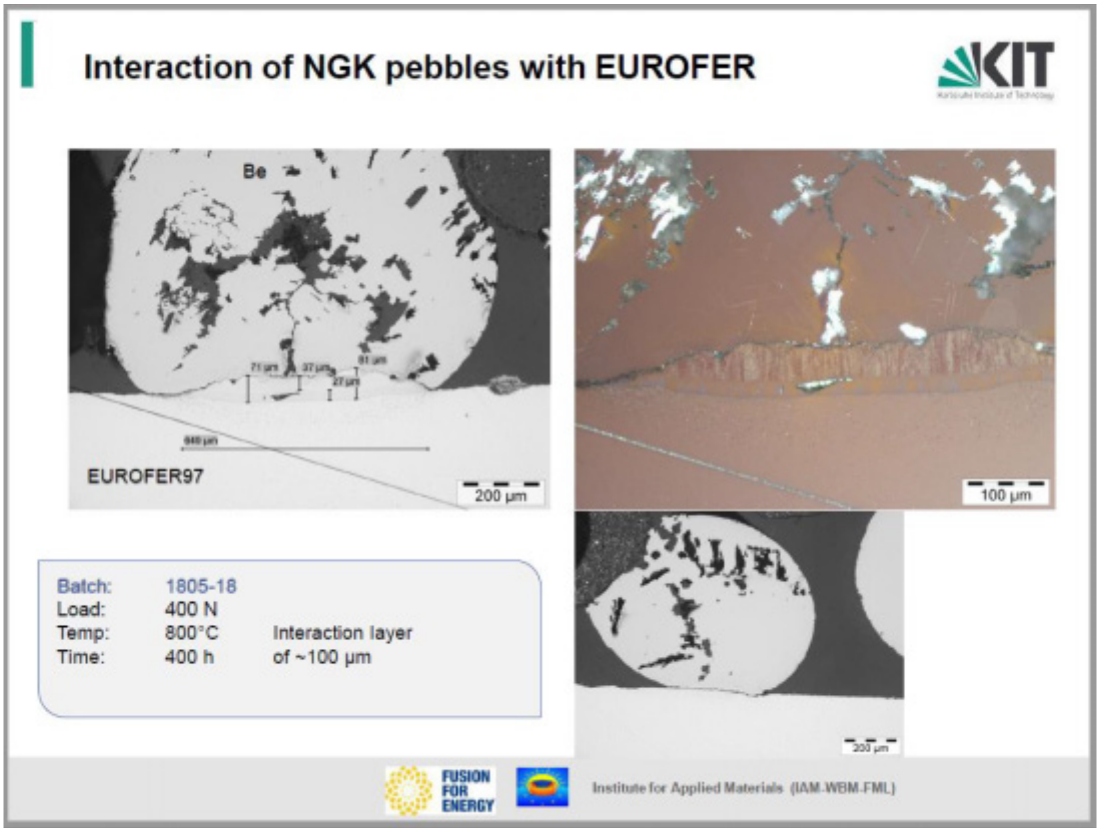
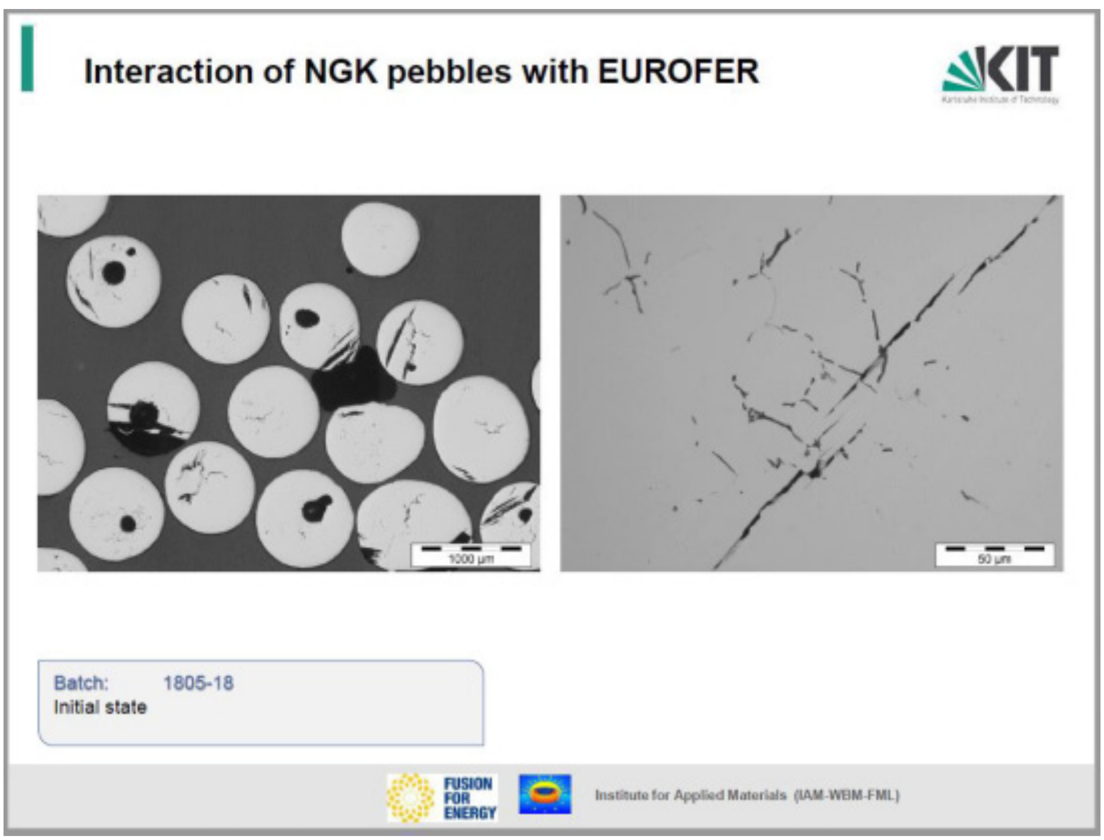
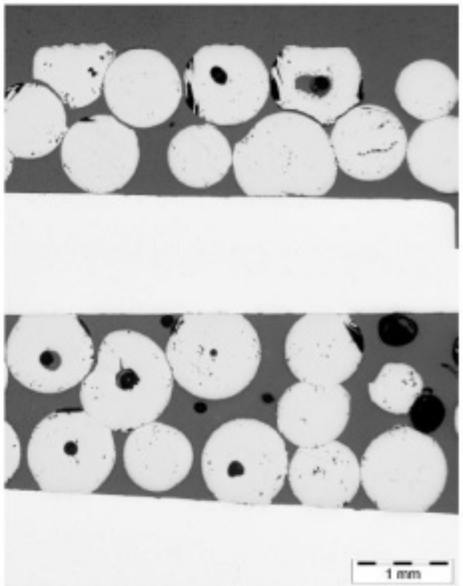
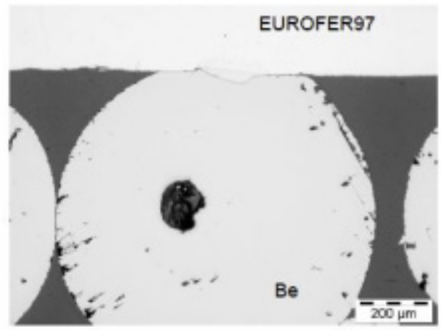
Institute for Applied Materials (IAM-WBM-FML)


Interaction of NGK pebbles with EUROFER
KIT
Be
EUROFER97
200 µm
100 µm
Batch: 1805-18
Load: 400 N
Temp: 800°C
Time: 400 h
Interaction layer of ~100 µm
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)


Interaction of NGK pebbles with EUROFER
KIT
1000 µm
50 µm
Batch: 1805-18
Initial state
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)

Interaction of NGK pebbles with EUROFER





Batch:	1473-10	
Load:	400 N	
Temp:	800°C	Interaction layer
Time:	100 h	of ~100 µm

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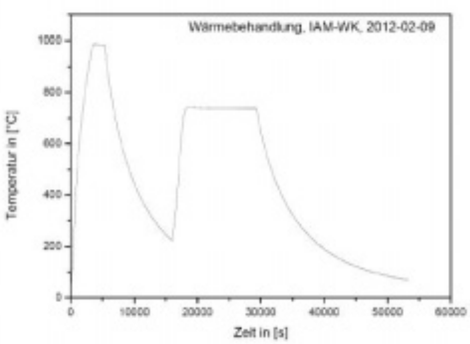
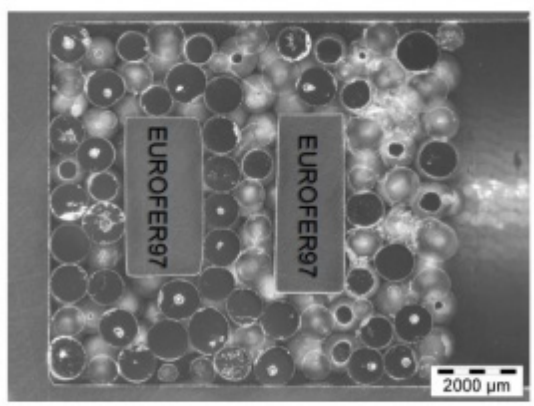
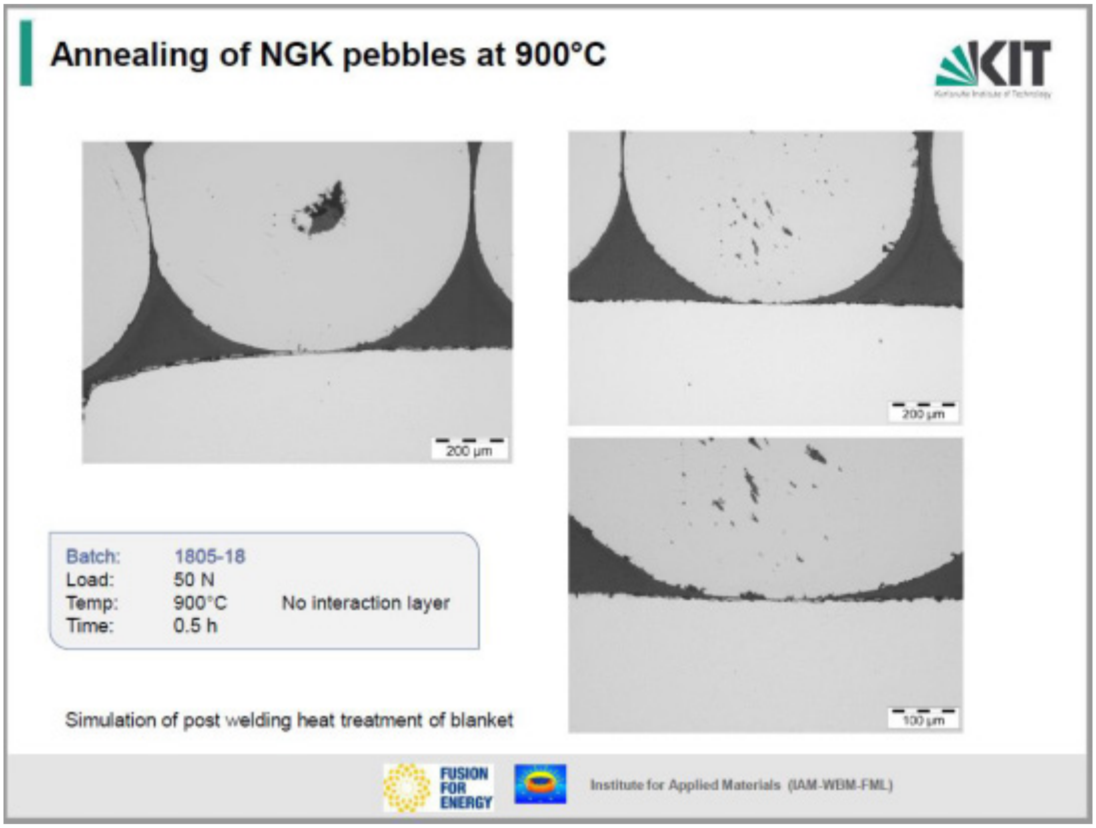
Annealing of NGK pebbles at 900°C

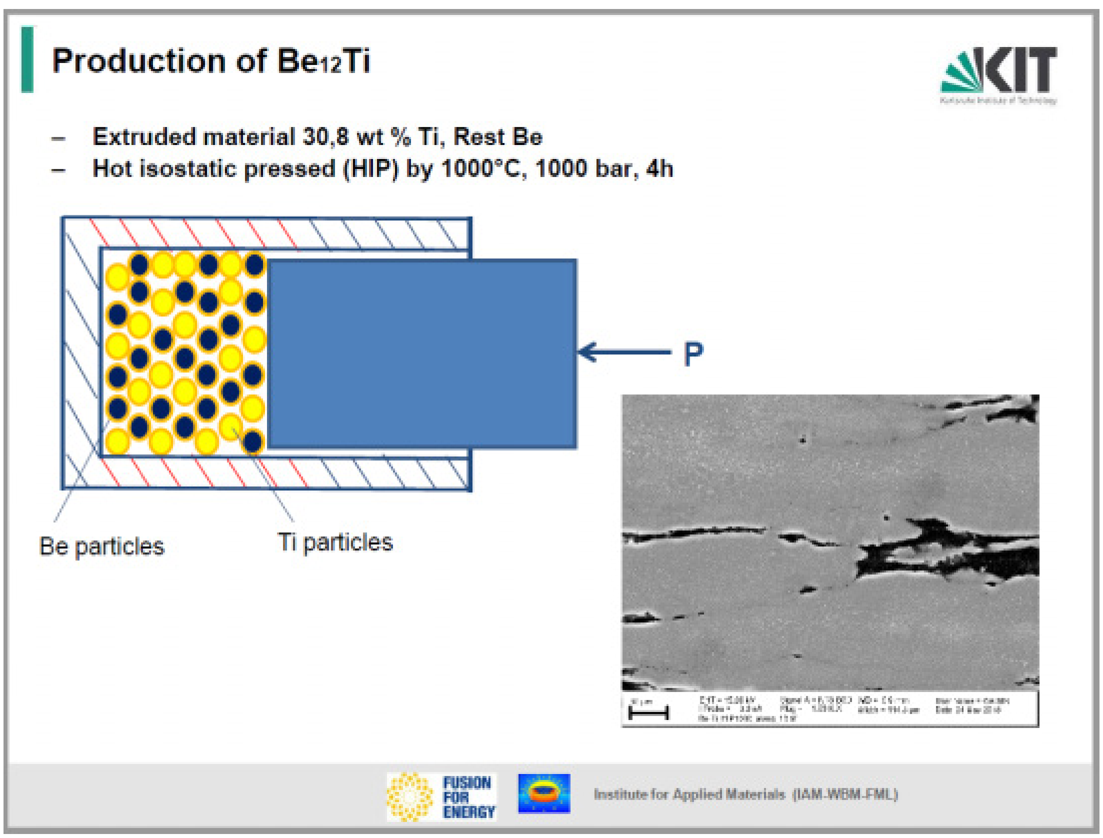
Simulation of post welding heat treatment of blanket





FUSION FOR ENERGY — Institute for Applied Materials (IAM-WBM-FML)


Annealing of NGK pebbles at 900°C
KIT
200 µm
200 µm
100 µm
Batch: 1805-18
Load: 50 N
Temp: 900°C
Time: 0.5 h
No interaction layer
Simulation of post welding heat treatment of blanket
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)


Production of Be12Ti
KIT
– Extruded material 30,8 wt % Ti, Rest Be
– Hot isostatic pressed (HIP) by 1000°C, 1000 bar, 4h
P
Be particles
Ti particles
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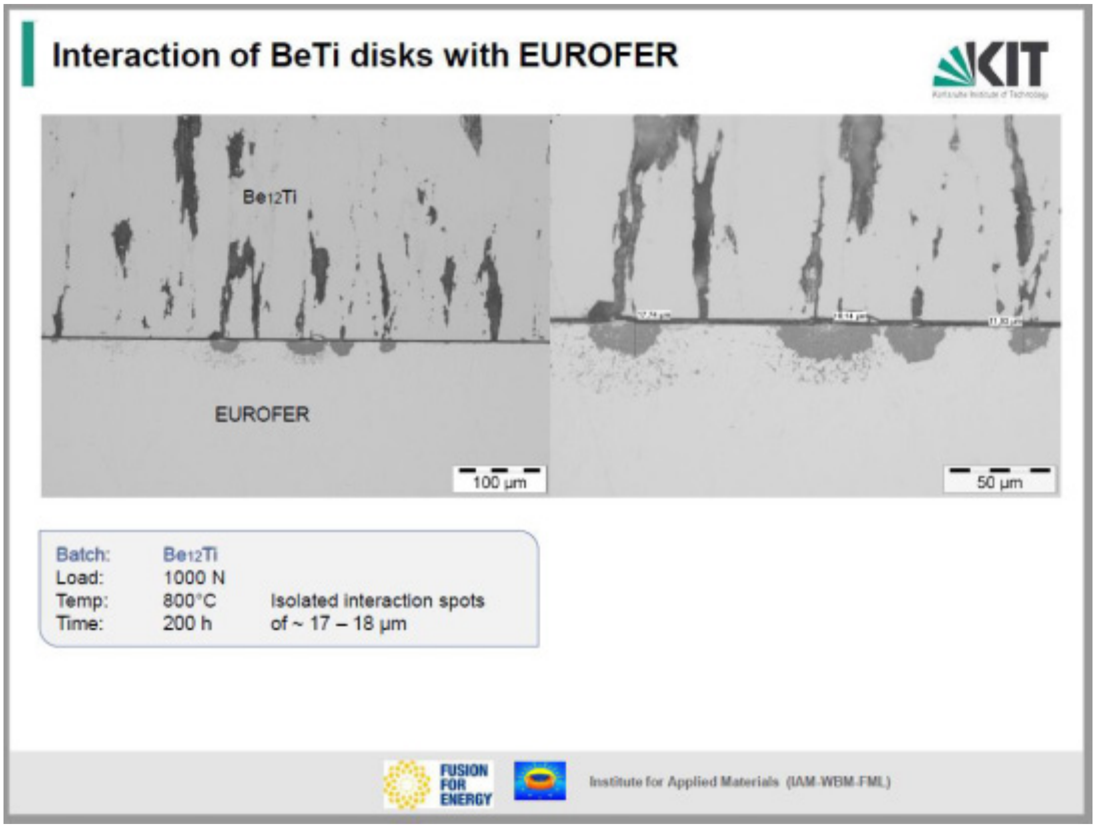
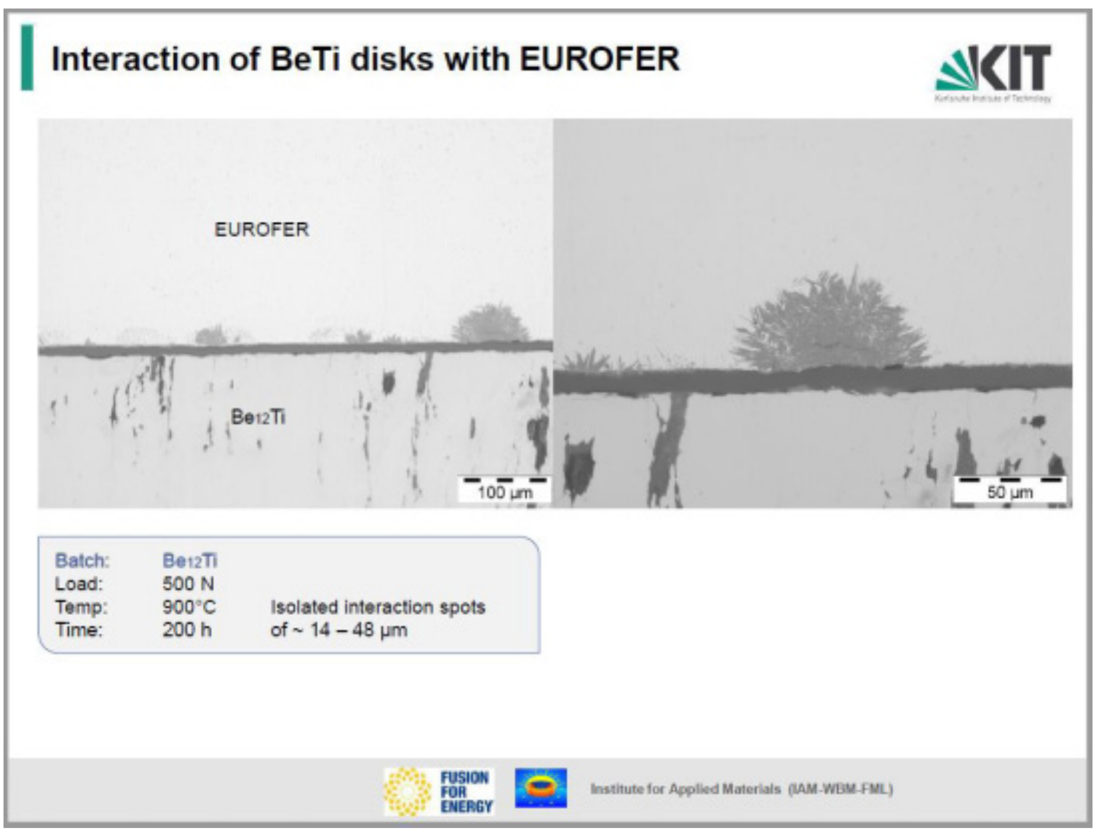
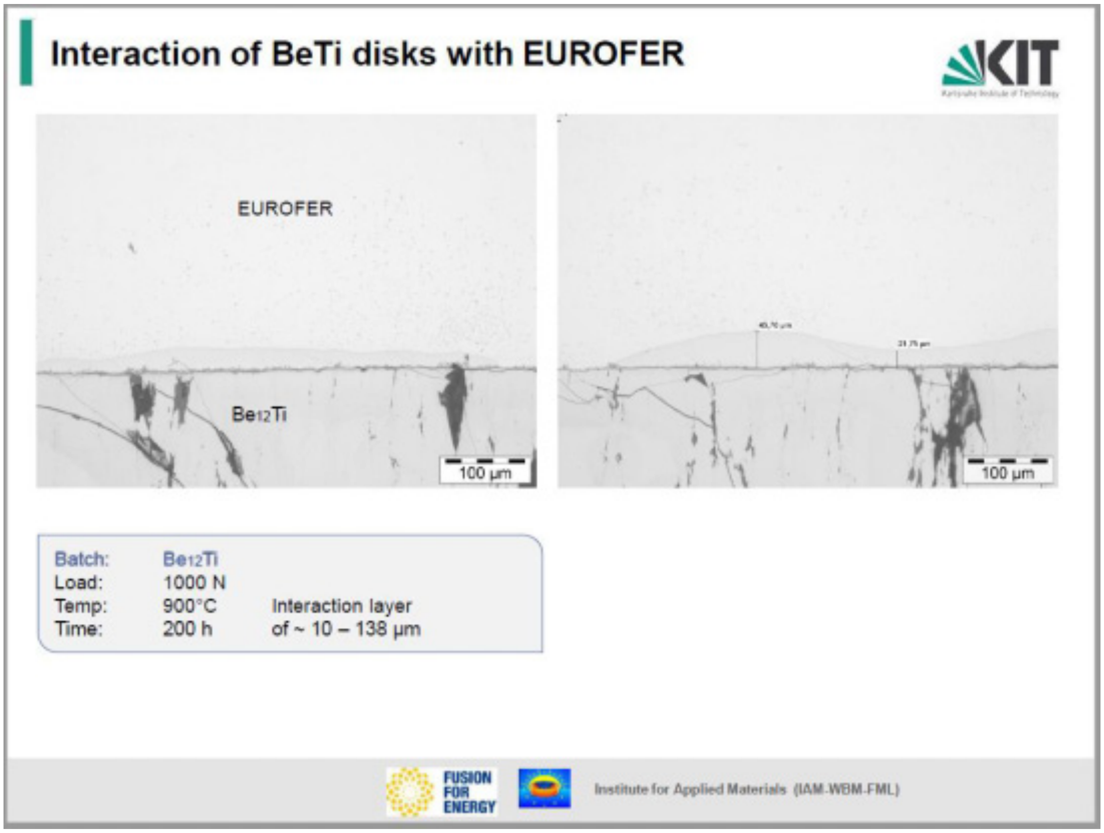
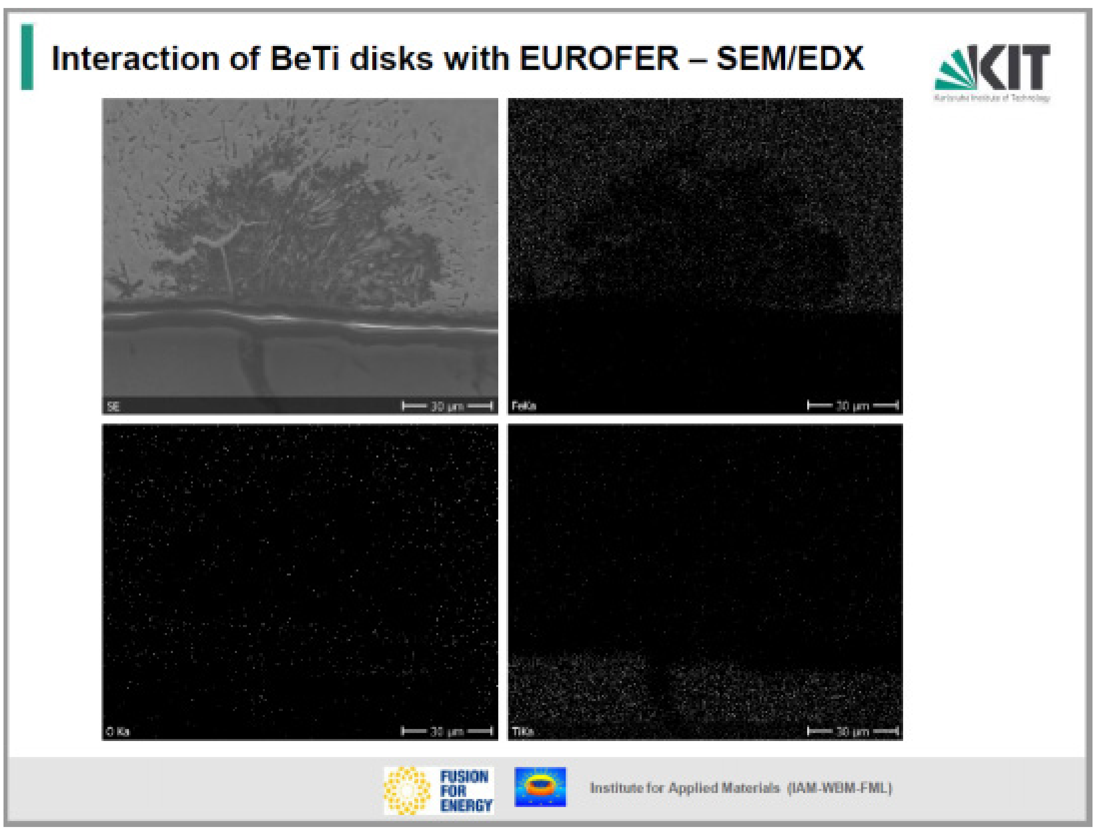
Institute for Applied Materials (IAM-WBM-FML)

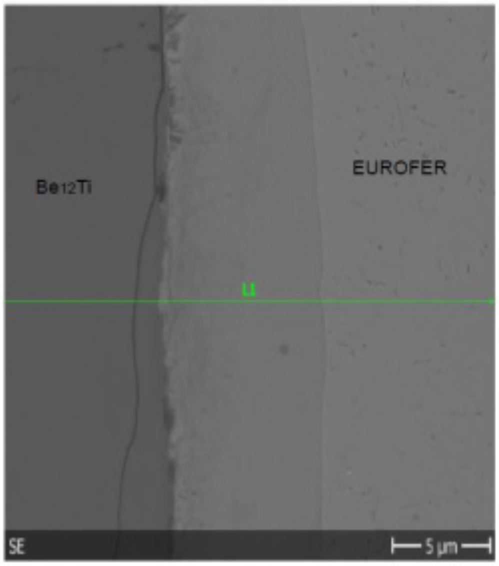

Interaction of BeTi disks with EUROFER
KIT
Be12Ti
EUROFER
100 µm
50 µm
Batch: Be12Ti
Load: 1000 N
Temp: 800°C
Time: 200 h
Isolated interaction spots of ~ 17 – 18 µm
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)


Interaction of BeTi disks with EUROFER
KIT
EUROFER
Be12Ti
100 µm
50 µm
Batch: Be12Ti
Load: 500 N
Temp: 900°C
Time: 200 h
Isolated interaction spots of ~ 14 – 48 µm
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)

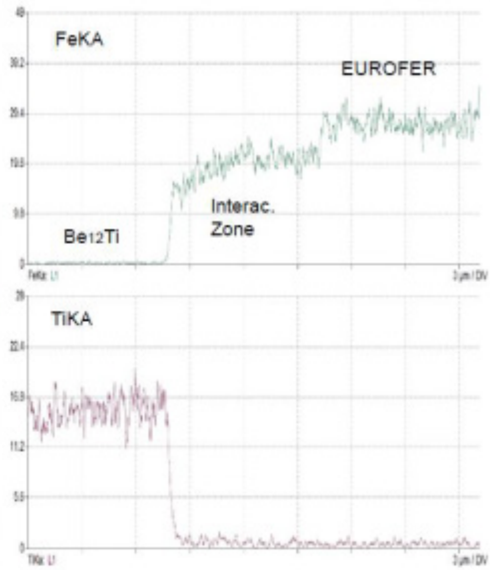

Interaction of BeTi disks with EUROFER
KIT
EUROFER
Be12Ti
100 µm
100 µm
Batch: Be12Ti
Load: 1000 N
Temp: 900°C
Time: 200 h
Interaction layer
of ~ 10 – 138 µm
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)


Interaction of BeTi disks with EUROFER – SEM/EDX
KIT
FUSION FOR ENERGY
Institute for Applied Materials (IAM-WBM-FML)

Interaction of BeTi disks with EUROFER – SEM/EDX



Highest temperature with highest load 900°C and 1000N



EDX linear analysis across interaction zone

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Compatibility of neutron multiplier materials with structural materials (EUROFER)

Interaction with Be pebbles – HCPB design

Temperature, °C	Time, h	Loading, N	Interaction layer, µm
600	100	600	no
600	400	600	no
700	100	600	~6
700	400	600	~13
800	100	400	~100
800	400	400	~100
900	0.5	50	no

Interaction with $Be_{12}Ti$ disks – Demo design

Temperature, °C	Time, h	Loading, N	Interaction layer, µm
800	200	1000	Spots, 17-18
900	200	500	Spots, 14-138
900	200	1000	Layer, 10-47

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Conclusion

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- Experiments by interaction study of Beryllium pebbles and $Be_{12}Ti$ disks with EUROFER were performed
- Determination of interaction layer by different parameter (Be pebbles NGK)
  - Be pebbles (NGK) show no layer by 600°C by contact times up to 400h
  - Interaction layer was measured by 700°C (6µm) up to 100µm by 800°C
  - Post welding experiment show no interaction
- Determination of interaction spots/layer by different parameter ($Be_{12}Ti$)
  - Isolated interaction spots by 800°C (18µm) up to 50µm by 900°C, interaction layer (max. 138µm) detected by highest load 1000N
  - Compatibility tests of Be-Ti composite and EUROFER plates showed that interaction is mainly caused by diffusion of Be atoms from beryllium phase in the composite
  - Because EDX mapping show titanium does not diffuse into steel
  - And lower iron content in the interaction layer can only be explained by the diffusion of beryllium atoms from beryllium phases of the disks
  - $Be_{12}Ti$ interact not directly with the steel

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Thanks

KIT Karlsruhe Institute of Technology

- Mr. Gaisin IAM-AWP
- Mr. Vladimirov IAM-AWP
- Mr. Chakin IAM-AWP
- Mr. Schneider IAM-WBM-FML

- And my group

- Mr. Holzer
- Mr. Ries
- Mr. Rietschel
- Mr. Weber
- Mr. Ehrmann
- Mr. Lautensack
- (IAM-WBM-FML)

- Thank you for your interest

FUSION FOR ENERGY Institute for Applied Materials (IAM-WBM-FML)

Comparison of the thermal shock damage on beryllium induced by electron beam and laser

B. Spilker, G. Pintsuk, M. Wirtz, and M. Zlobinski

Forschungszentrum Jülich GmbH, Institut für Energie- und Klimaforschung, 52425 Jülich, Germany

The choice of beryllium as plasma facing material for ITER was driven by its low atomic number, which ensures an excellent plasma compatibility, its oxygen getter capability, and favorable thermomechanical properties. In contrast, the rather low melting point of 1560 K makes beryllium susceptible to significant damage during thermal transients. In recent years, efforts have been undertaken to thoroughly characterize the response of beryllium to ITER-relevant transient thermal loads with electron beam testing. Detailed insights about the damage, cracking, and melting thresholds have been obtained but at the same time it was noticed that the electron penetration depth might have an influence on the thresholds and the general damaging behavior. The volumetric loading with energetic electrons leads to a less steep thermal gradient in the heat affected layer compared to near surface loading methods like plasma and laser.

In this work, a comprehensive testing campaign exerting ITER-relevant transient thermal loads with a 1064nm Nd:YAG laser on mirror finish S-65 beryllium specimen has been carried out. The facility used for this experiment was FREDIS (fuel retention diagnostic setup) located at Forschungszentrum Jülich. The loading conditions were chosen to mimic thermal loads originating from edge localized modes on thick beryllium layers in the divertor and off-normal events like massive gas injections/shattered pellet injections on the first wall. Namely, absorbed power densities in the range of 50-900MWm^{-2} with pulse durations of 1-10ms were applied for up to 1000 pulses. The samples were tested at room temperature and 250°C base temperature. After the laser loading, the damaged areas were investigated using scanning electron microscopy, laser profilometry, and energy dispersive X-ray spectroscopy.

It was found that the cracking and melting thresholds were about 200-300MWm^{-2} lower for the laser loading in comparison to electron beam loading. The damage threshold dropped from 150-200MWm^{-2} for electron beam loading to 50-80MWm^{-2} for laser loading. This behavior was qualitatively expected due to the different thermal gradients into the depth for the same absorbed power density for both loading methods. For an absorbed power density of 800MWm^{-2} with a pulse duration of 1ms and 1000 pulses, significant evaporation of beryllium was observed. Prompt redeposition of beryllium in inverse cone structures was detected up to a distance of several mm from the loaded area.

The obtained results demonstrated that transient thermal loading with fusion relevant characteristics in FREDIS is feasible and that the electron penetration depth needs to be considered for the interpretation of earlier results. Future plans involve studying the influence of damaged surfaces on the fuel retention characteristics of beryllium.

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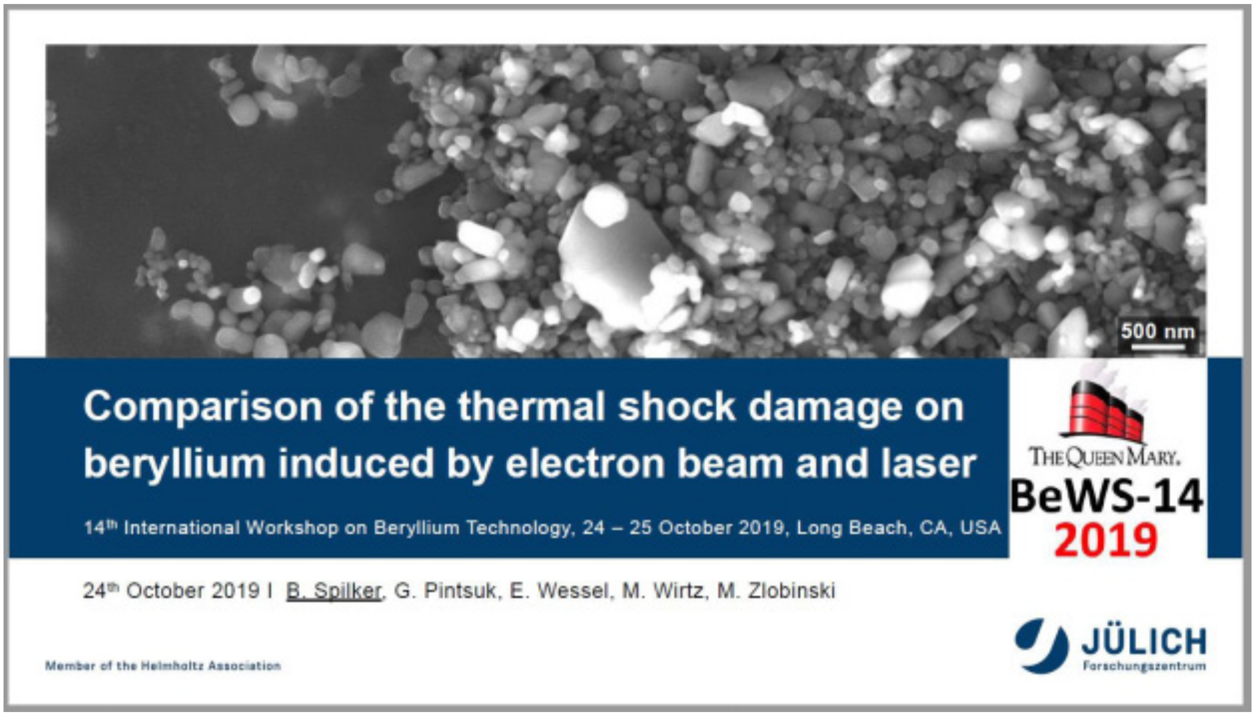
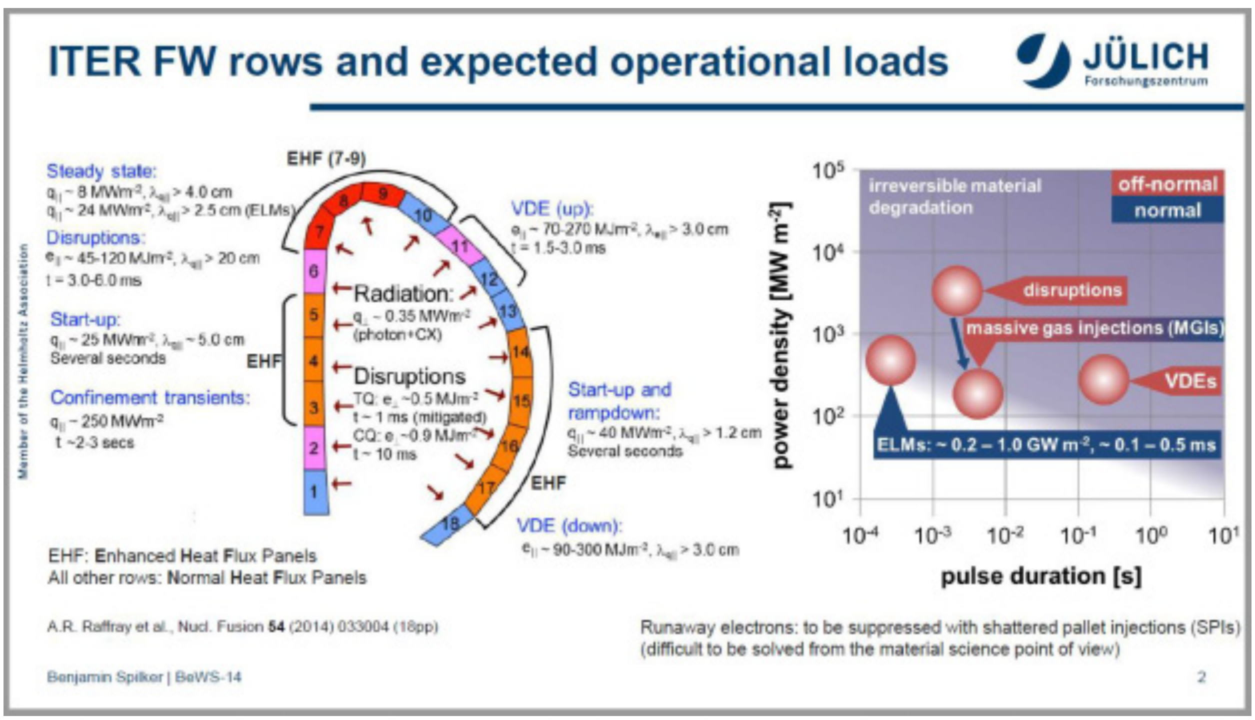

500 nm
Comparison of the thermal shock damage on beryllium induced by electron beam and laser
14th International Workshop on Beryllium Technology, 24 – 25 October 2019, Long Beach, CA, USA
THE QUEEN MARY.
BeWS-14
2019
24th October 2019 I B. Spilker, G. Pintsuk, E. Wessel, M. Wirtz, M. Zlobinski
Member of the Helmholtz Association
JÜLICH
Forschungszentrum


ITER FW rows and expected operational loads
JÜLICH
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Steady state:
$q_{||}$ ~ 8 MWm^{-2}, $\lambda_{q||}$ > 4.0 cm
$q_{||}$ ~ 24 MWm^{-2}, $\lambda_{q||}$ > 2.5 cm (ELMs)
Disruptions:
$e_{||}$ ~ 45-120 MJm^{-2}, $\lambda_{q||}$ > 20 cm
t = 3.0-6.0 ms
Start-up:
$q_{||}$ ~ 25 MWm^{-2}, $\lambda_{q||}$ ~ 5.0 cm
Several seconds
Confinement transients:
$q_{||}$ ~ 250 MWm^{-2}
t ~2-3 secs
EHF (7-9)
EHF
EHF
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
Radiation:
$q_{\perp}$ ~ 0.35 MWm^{-2}
(photon+CX)
Disruptions
TQ: $e_{\perp}$ ~0.5 MJm^{-2}
t ~ 1 ms (mitigated)
CQ: $e_{\perp}$ ~0.9 MJm^{-2}
t ~ 10 ms
VDE (up):
$e_{||}$ ~ 70-270 MJm^{-2}, $\lambda_{q||}$ > 3.0 cm
t = 1.5-3.0 ms
Start-up and rampdown:
$q_{||}$ ~ 40 MWm^{-2}, $\lambda_{q||}$ > 1.2 cm
Several seconds
VDE (down):
$e_{||}$ ~ 90-300 MJm^{-2}, $\lambda_{q||}$ > 3.0 cm
EHF: Enhanced Heat Flux Panels
All other rows: Normal Heat Flux Panels
power density [MW m^{-2}]
pulse duration [s]
10^{1} 10^{2} 10^{3} 10^{4} 10^{5}
10^{-4} 10^{-3} 10^{-2} 10^{-1} 10^{0} 10^{1}
irreversible material degradation
off-normal
normal
disruptions
massive gas injections (MGIs)
VDEs
ELMs: ~ 0.2 – 1.0 GW m^{-2}, ~ 0.1 – 0.5 ms
A.R. Raffray et al., Nucl. Fusion **54** (2014) 033004 (18pp)
Runaway electrons: to be suppressed with shattered pallet injections (SPIs)
(difficult to be solved from the material science point of view)
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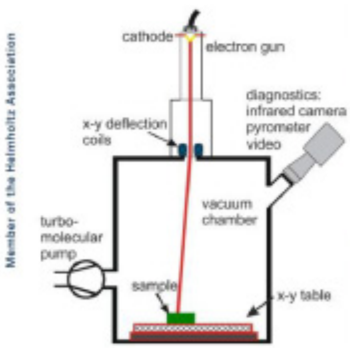
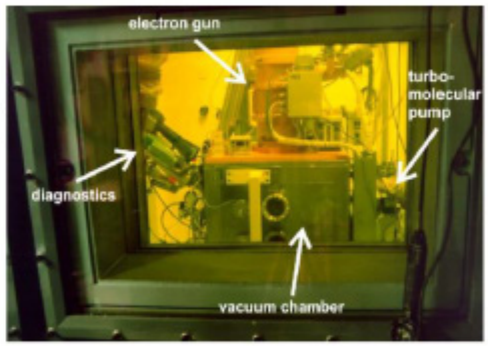
2

Electron beam facility JUDITH 1

JÜLICH Forschungszentrum





Electron beam parameters

- Max. power: 60 kW
- Max. acc. voltage: 150 kV
- Pulse duration: ≥ 1 ms
- Beam diameter: ~ 1 mm
- Sweeping freq.: ≤ 100 kHz
- Beam is swept over loaded area
- Capable of handling toxic/radioactive materials

Retired, replacement with JUDITH 3 facility in progress

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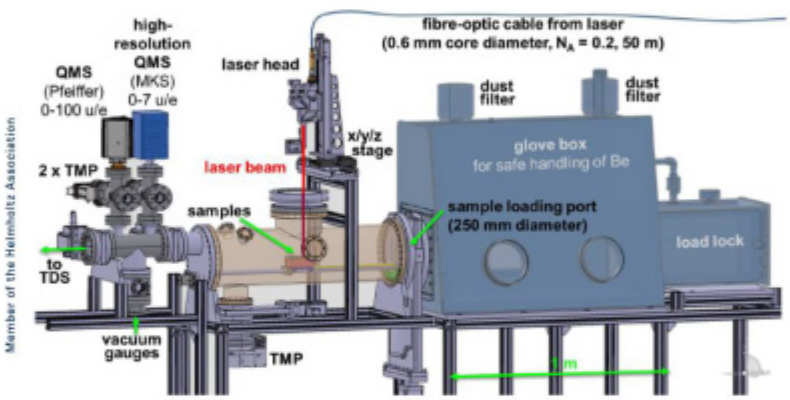
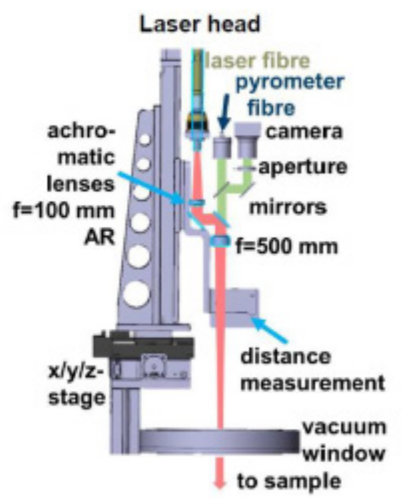
Fuel Retention Diagnostic Setup (FREDIS)

JÜLICH Forschungszentrum





Laser beam parameters

- Max. energy: 100 J
- Pulse length: 0.1-20 ms
- Beam diameter: ~3 mm
- Wavelength: 1064 nm

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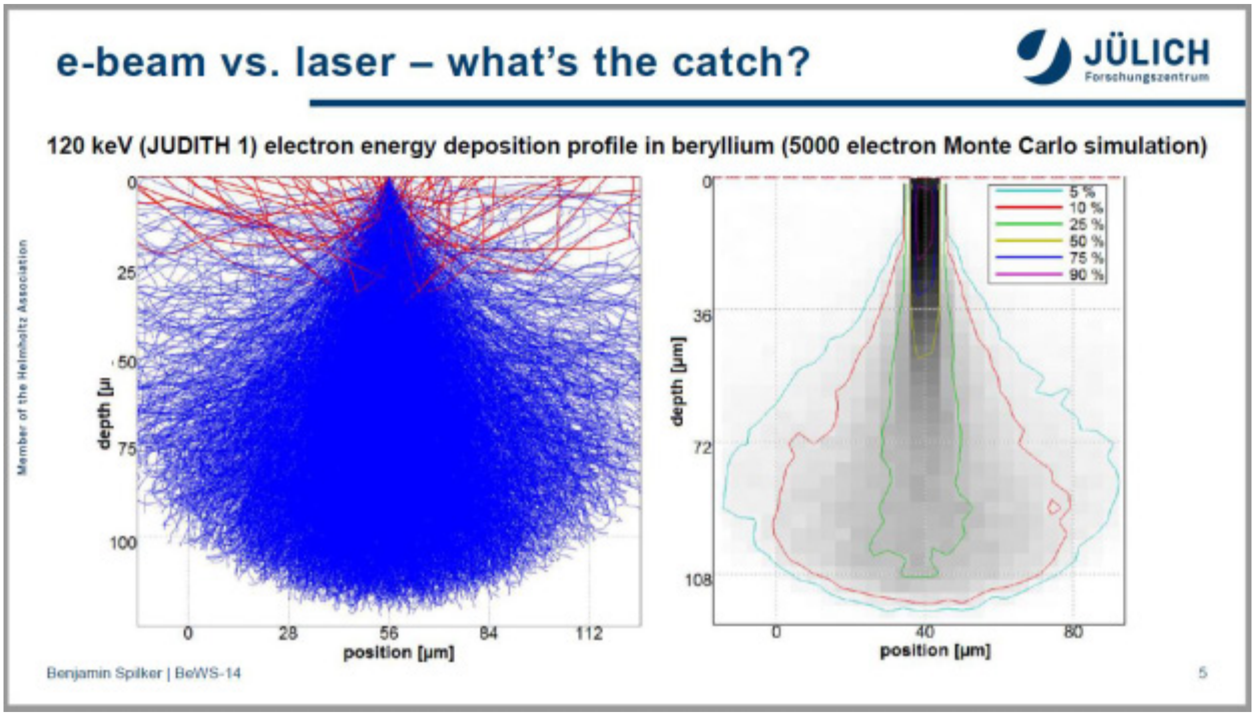
e-beam vs. laser – what's the catch?
JÜLICH
Forschungszentrum
120 keV (JUDITH 1) electron energy deposition profile in beryllium (5000 electron Monte Carlo simulation)
Member of the Helmholtz Association
depth
position [µm]
depth [µm]
position [µm]
5 %
10 %
25 %
50 %
75 %
90 %
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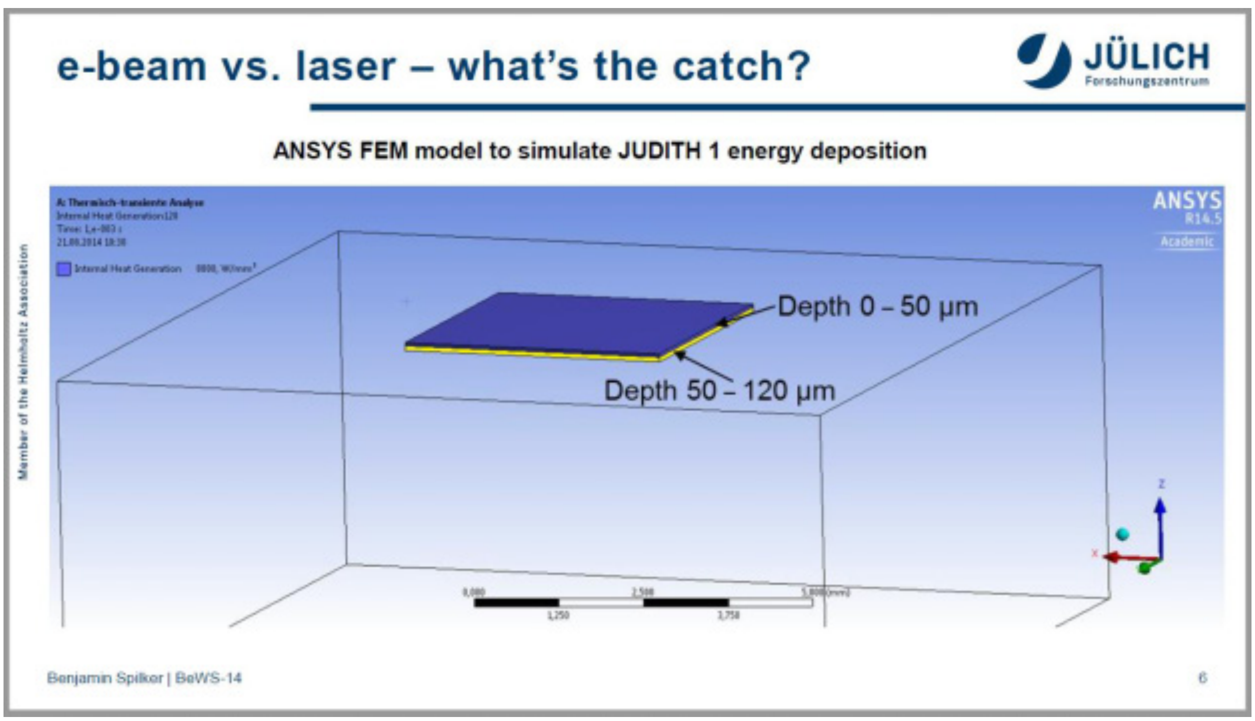
5


e-beam vs. laser – what's the catch?
JÜLICH
Forschungszentrum
ANSYS FEM model to simulate JUDITH 1 energy deposition
Member of the Helmholtz Association
ANSYS
R14.5
Academic
Depth 0 – 50 µm
Depth 50 – 120 µm
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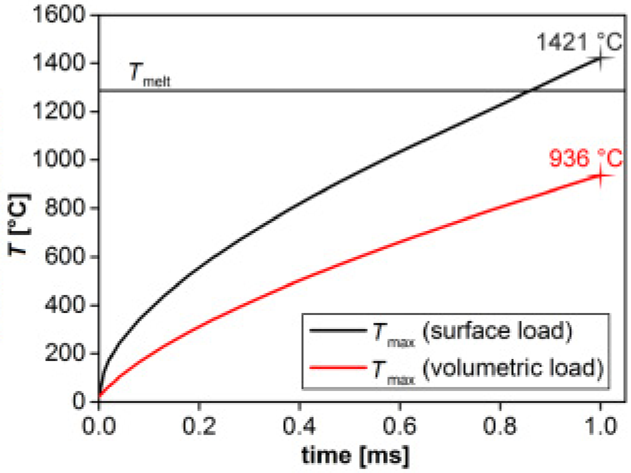
6

e-beam vs. laser – what's the catch?



Loading conditions

L_{abs} = 0.8 GWm^{-2}
t = 1 ms
T_{base} = RT

- In the surface load scenario, the melting threshold is exceeded for these loading conditions while the volumetric energy distribution with electrons leads to a maximum temperature below the melting threshold
- Difference between e-beam and laser loading ~500 °C in the simulation
- Can this be experimentally confirmed?

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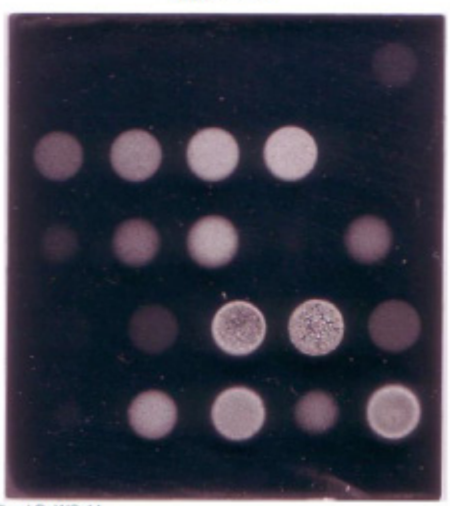
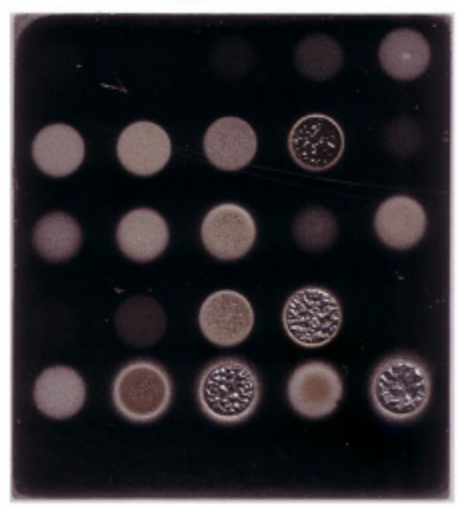
Transient laser loading in FREDIS

T_{base} = RT



T_{base} = 250 °C



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Damage mapping

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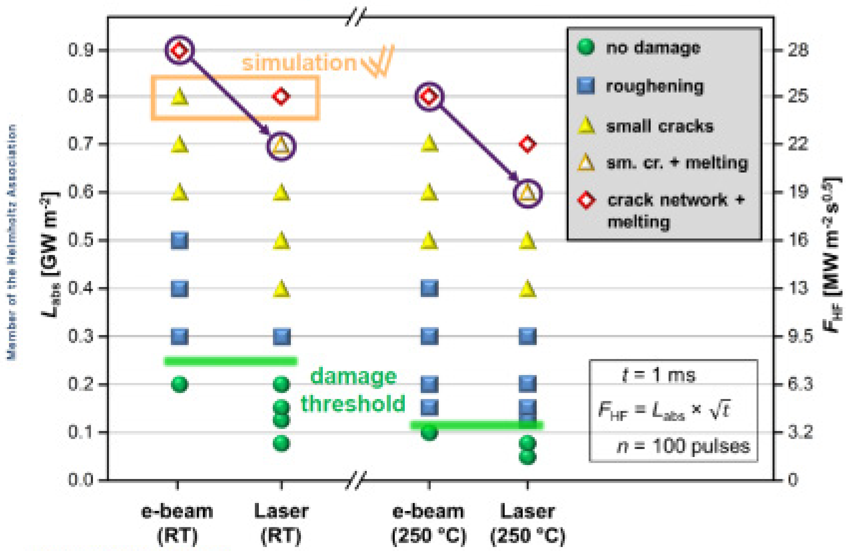

- Melting threshold for laser about 0.2 GWm^{-2} lower (~300 °C difference between e-beam and laser)
- Experimental difference less pronounced than predicted by simulation
- Cyclic loading/roughening enlarges surface area absorbing light (while all electrons are already absorbed on a flat surface)
- Higher base temperature weakens material (lower damage and cracking thresholds)

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Roughening

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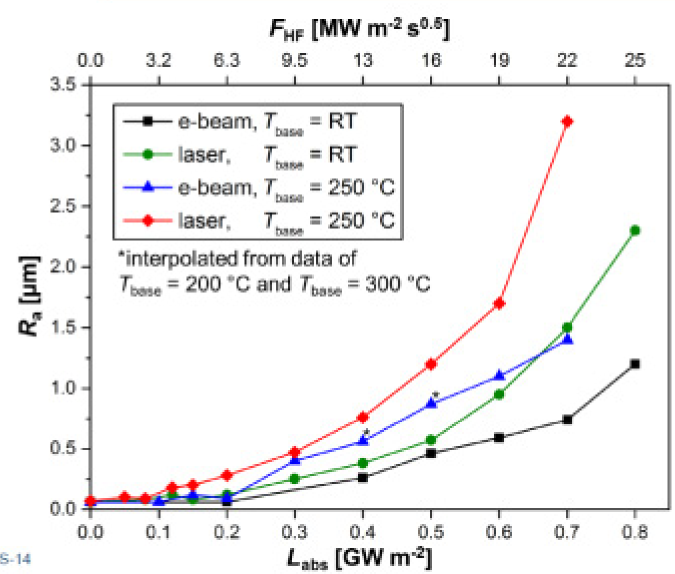

- Arithmetic mean roughness R_a measured by laser profilometry
- Thermally induced stresses (and maximum surface temperature) higher for laser loading at any given L_{abs} and T_{base}

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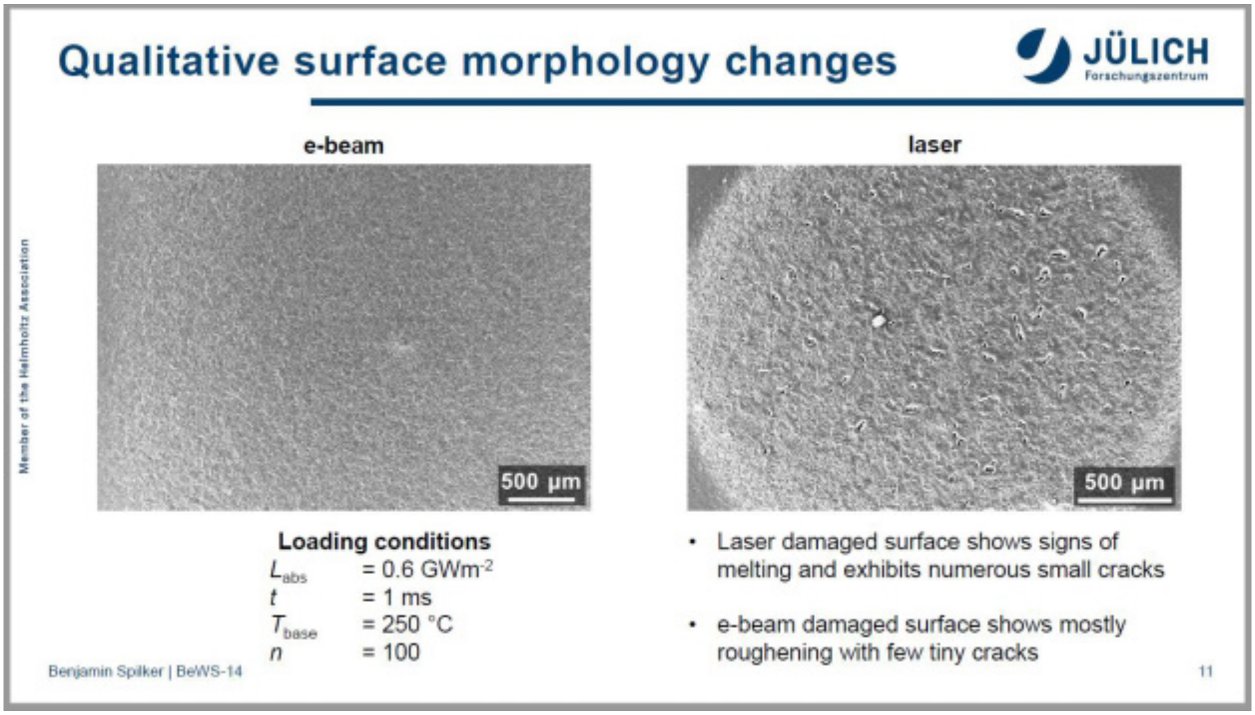
Qualitative surface morphology changes
JÜLICH
Forschungszentrum
e-beam
laser
500 µm
500 µm
Loading conditions
L_{abs} = 0.6 GWm^{-2}
t = 1 ms
T_{base} = 250 °C
n = 100
• Laser damaged surface shows signs of melting and exhibits numerous small cracks
• e-beam damaged surface shows mostly roughening with few tiny cracks
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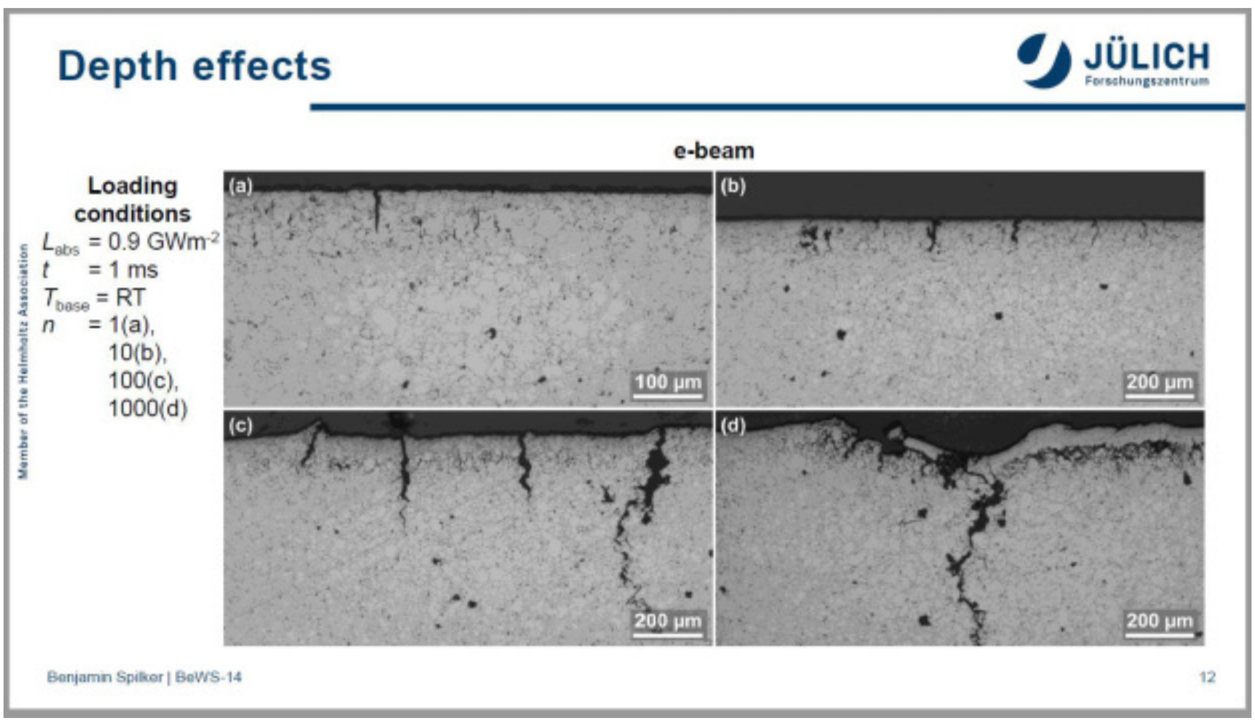
11


Depth effects
JÜLICH
Forschungszentrum
e-beam
Loading conditions
L_{abs} = 0.9 GWm^{-2}
t = 1 ms
T_{base} = RT
n = 1(a), 10(b), 100(c), 1000(d)
(a)
(b)
(c)
(d)
100 µm
200 µm
200 µm
200 µm
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Depth effects

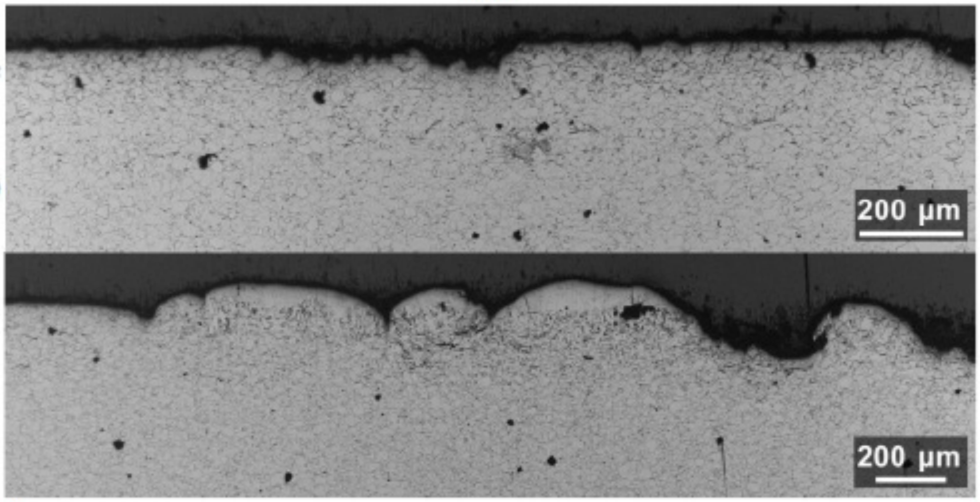
JÜLICH Forschungszentrum

Member of the Helmholtz Association

laser

Loading conditions

L_{abs} = 0.6 GWm^{-2}
t = 1 ms
T_{base} = 250 °C
n = 100 (top)
1000 (bot.)



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Conclusions

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- Electron penetration depth in beryllium is considerably high due to low Z (~120 µm for 120 keV electrons)
- Damage threshold seems to be very similar for e-beam and laser loading
- Cracking for laser loading is hardly observed until close to the melting point
- Cracking for e-beam loading is strongly pronounced even for lower loads (might be promoted by the beam scanning mode with several ten kHz)
- Melting threshold is overestimated by e-beam loading by about 0.2 GWm^{-2} due to the volumetric energy deposition and lower resulting surface temperature (laser loading is more relevant to the application)
- Adhesion between melt and bulk significantly better for laser loading (good news!)

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Session 4: Industrial Fabrication

Overview of the US Beryllium Industry

K. Smith (Materion Brush, USA)

Overview of the United States Beryllium Industry

Keith J. Smith

Materion Brush, Inc., Elmore, Ohio, U.S.A.

Beryllium is a critical material of construction for the ITER First Wall Panels. The supply and fabrication of beryllium is key to the success of the ITER project. This presentation will provide an overview of the US beryllium industry including mining, manufacturing, and fabrication capabilities. Beryllium raw material availability, ITER-relevant First Wall and instrumentation beryllium grades, and engineering and program management services provided in support to those products will be discussed. Additionally, activities related to process improvements within Materion's beryllium manufacturing plants will be addressed.

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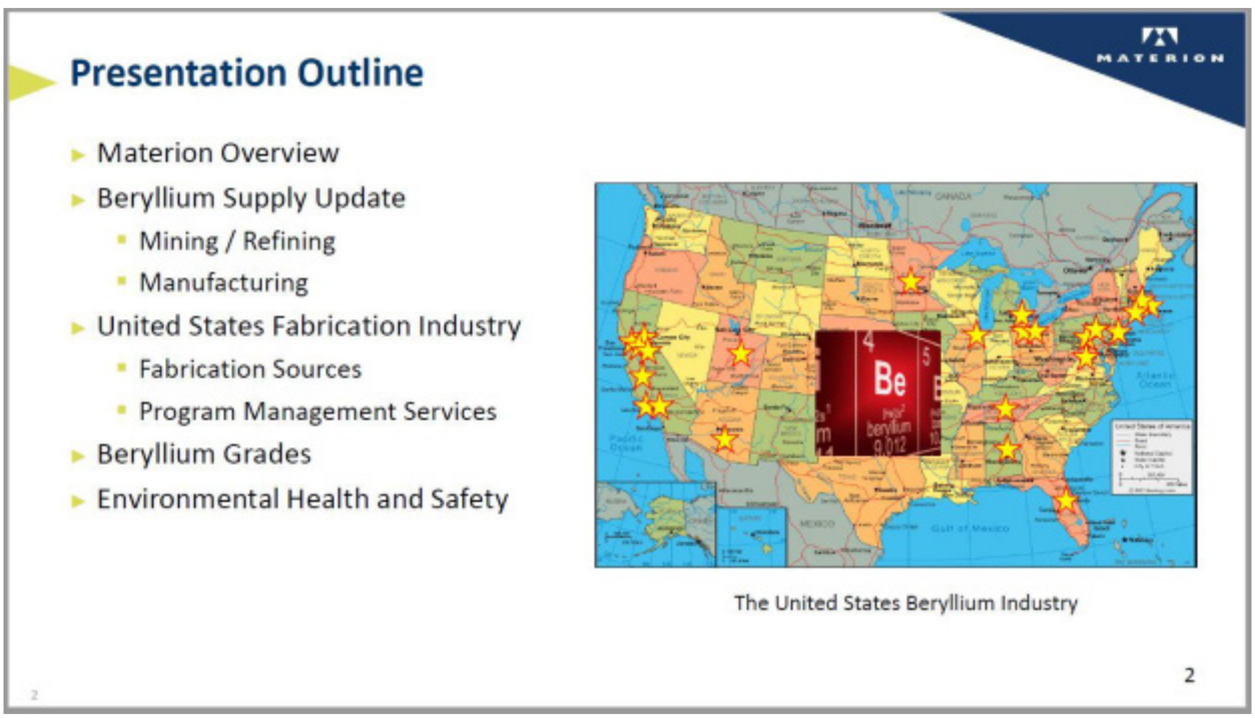
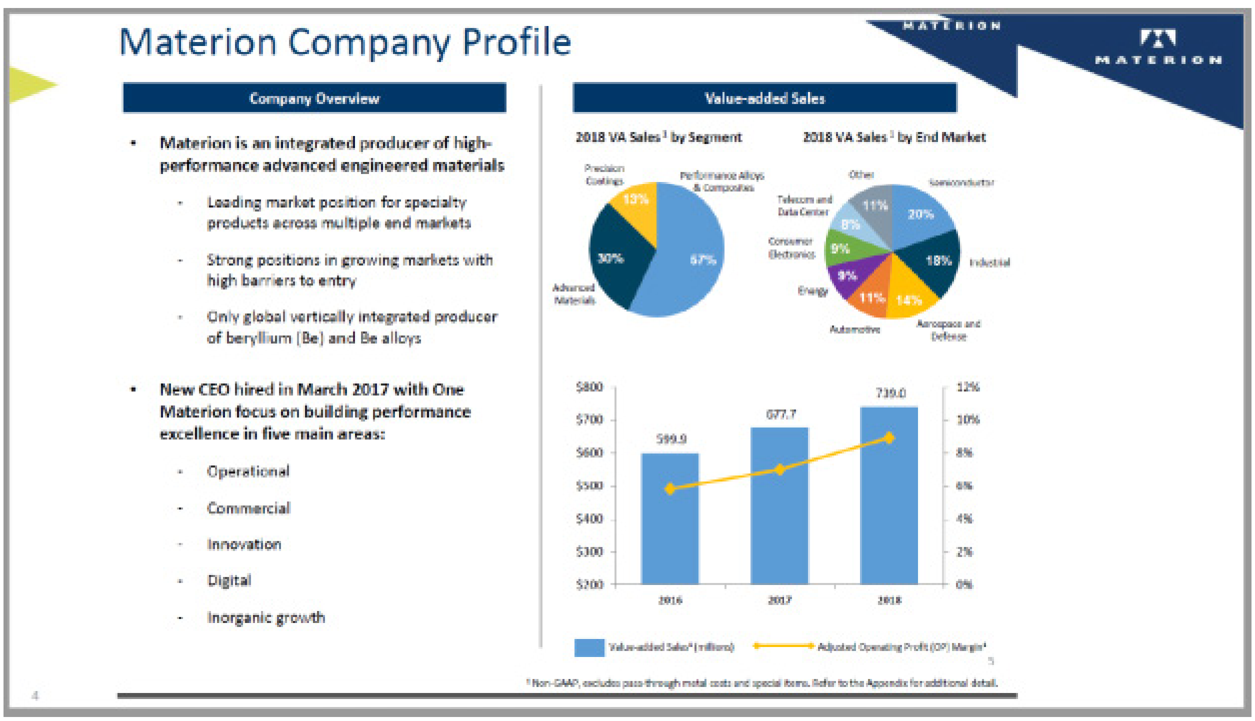
14710 W Portage River South Road
Elmore, Ohio 43416-9500
U.S.A.


MATERION
Overview of the United States
Beryllium Industry
Mr. Keith Smith
Materion Performance
Alloys & Composites
Elmore, OH USA
THE QUEEN MARY
BeWS-14
2019
14th International Workshop
On Beryllium Technology
Long Beach, California, USA
24-25 October 2019
25 October 2019


Presentation Outline
MATERION
Materion Overview
Beryllium Supply Update
Mining / Refining
Manufacturing
United States Fabrication Industry
Fabrication Sources
Program Management Services
Beryllium Grades
Environmental Health and Safety
Be
beryllium
9.012
The United States Beryllium Industry
2


MATERION
Materion Overview
Materion, Inc
www.Materion.com
Headquarters - Mayfield Heights, OH
2400
Employees
50
Are serving customers in more than 50 countries
30
From more than 30 facilities
10
Located in 10 countries around the globe
3


Materion Company Profile
MATERION
Company Overview
Materion is an integrated producer of high-performance advanced engineered materials
Leading market position for specialty products across multiple end markets
Strong positions in growing markets with high barriers to entry
Only global vertically integrated producer of beryllium (Be) and Be alloys
New CEO hired in March 2017 with One Materion focus on building performance excellence in five main areas:
Operational
Commercial
Innovation
Digital
Inorganic growth
Value-added Sales
2018 VA Sales 1 by Segment
Precision Coatings
13%
Performance Alloys & Composites
67%
Advanced Materials
30%
2018 VA Sales 1 by End Market
Other
11%
Semiconductor
20%
Industrial
18%
Aerospace and Defense
14%
Automotive
11%
Energy
9%
Consumer Electronics
9%
Telecom and Data Center
8%
$800
$700
$600
$500
$400
$300
$200
599.9
677.7
739.0
12%
10%
8%
6%
4%
2%
0%
2016
2017
2018
Value-added Sales1 (millions)
Adjusted Operating Profit (OP) Margin1
1 Non-GAAP, excludes pass-through metal costs and special items. Refer to the Appendix for additional detail.

Materion Markets

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Market	2018 % of Value-added Sales[1]	Key Drivers
Semiconductor	20%	• Smart device growth • Non-volatile memory • Big data (data storage)
Industrial	18%	• Heavy equipment builds • Plastic tooling • Fire protection (R and C construction)
Aerospace and Defense	14%	• Structural and electronic components for satellites, combat vehicles, and aircraft • Precision-guided munitions
Automotive	11%	• Electronic systems and engine control • Increasing emissions standards • Electric vehicles and autonomous driving
Energy	9%	• Deep sea drilling and completion • Directional drilling • Solar, batteries, and smart grid devices
Consumer Electronics	9%	• Smart device growth • Sensing devices • Internet of Things (IoT)
Telecom and Data Center	8%	• 5G rollout • Undersea repeater housings • High reliability connectors
Total	89%	

[1] Non-GAAP; excludes pass-through metal costs. Refer to the Appendix for additional detail.

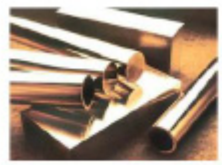
5

7

Beryllium Supply

- **Materion is the only fully integrated beryllium supplier in the world.**
- Be production facilities in the US:
  - Delta, UT — Mining
  - Elmore, OH — Extraction, casting, metal fabrication, machining
  - Tucson, AZ — BeO
  - Fremont, CA — Be foil and fabrication
  - Lincoln, RI — Clad products
  - Reading, PA — CuBe strip and wire
  - Warren, MI — CuBe
  - Chicago, IL — CuBe

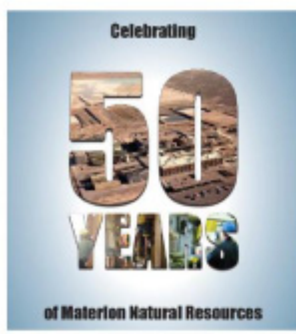



7

Beryllium Supply

- Materion operates a beryllium mine in the **Topaz-Spor Mountain** area of western Utah.
- The mineral is identified as **bertrandite**, a hydrous beryllium silicate ($Be_4Si_2O_7(OH)_2$)
- **Materion holds estimated ore reserves for 70 years of production.**
  - USGS estimates 21,000tons of Be reserves @ Utah.
- **Celebrated 50th year of operation in Delta, UT in 2019.**





8

Beryllium Supply – World Mine Production

MATERION

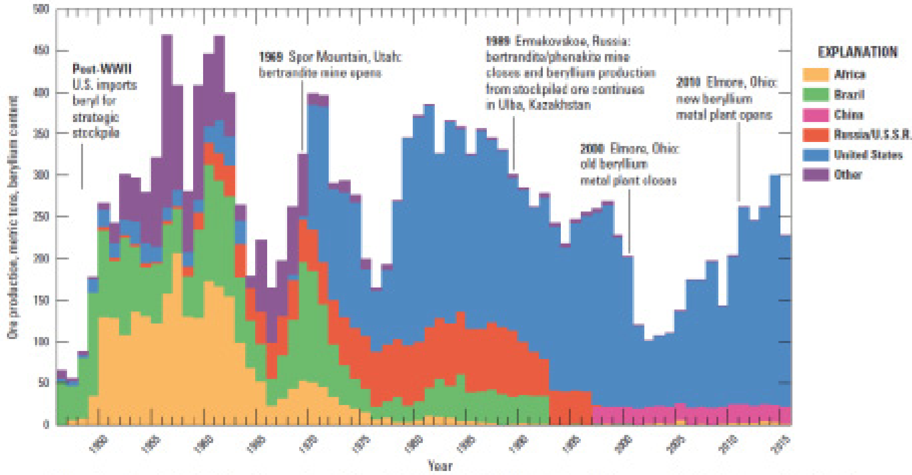


Figure 1. Trends in beryllium mine production from 1946 through 2015 illustrate the dominance of United States production since the opening of the Spor Mountain, Utah, bertrandite mine in 1969. With the exception of Spor Mountain and Ermakovskoe, Russia, beryllium production was derived from beryl mined from pegmatite deposits.

Beryllium – A Critical Mineral Commodity – Resources, Production, and Supply Chain,
US Geological Survey, October 2016

9

Beryllium Supply – Flow diagram (Materion)

MATERION

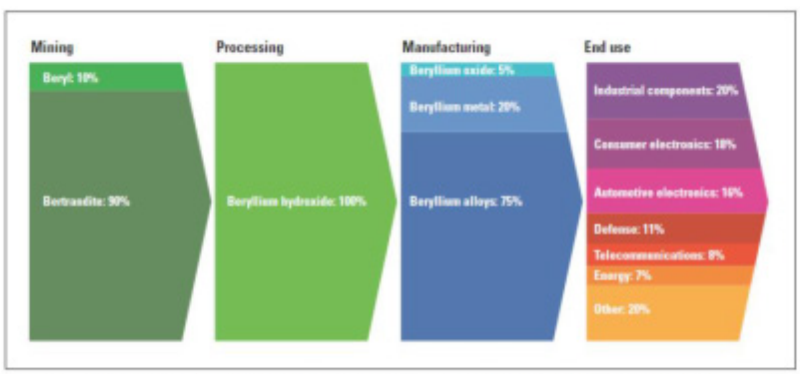


Figure 2. Simplified flow diagram of the beryllium industry. Percentages are relative to the total beryllium flow at each stage; flows between stages do not account for losses, changes in stocks, or recycling. End use category labelled "Other" includes 2 percent used in medical applications (Materion Corporation, 2016).

Beryllium – A Critical Mineral Commodity – Resources, Production, and Supply Chain,
US Geological Survey, October 2016

10

10

Be Production at Elmore, Ohio Plant

11

Beryllium Pebble Plant

- The Be Pebble Plant in Ohio is the world's most modern beryllium refining production facility.
- Cost: $110,000,000
- First production: April 2011.
- Plant is fully operational.
- Design capacity of 70,000 kg/yr

12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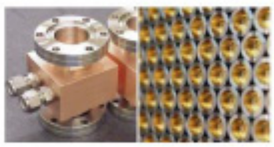
Beryllium Manufacturing
Melting and Casting
Powder Metallurgy
Powder Consolidation
Machining
13


Beryllium Fabrication
Capabilities in the US
MATERION

Materion Fabrication www.materion.com

- **Materion** – Elmore, OH
  - Broad range of machining operations including:
    - Milling, turning, EDM, saw, grinding

- **Materion** – Electrofusion – Fremont, CA
  - Machining: Milling, turning, electron beam welding
  - Be Foil
  - Fabrication - UHV



- **Materion** – Ceramics – Tucson, AZ
  - Machining: Ultrasonic machining, grinding, lapping, turning

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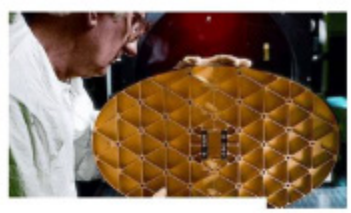
General Dynamics Mission Systems

- GDMS
- Cullman, AZ 35057
- 256-737-5200
- www.gd-imaging.com

GENERAL DYNAMICS
Mission Systems



Beryllium Machining

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General Dynamics offers close tolerance machining and processing of beryllium and its alloys to deliver optics and optical assemblies with precise, complex geometric features. With more than 50 years of expertise and state-of-the-art equipment, we are able to achieve virtually unmatched tolerances.

One-third the weight of aluminum, six times stiffer than steel and with a high thermal stability, beryllium is the ideal material for applications in space or other harsh environments. We machine our beryllium to tolerances up to 1/100,000 of an inch (0.000001 in.) to meet the most demanding mission requirements.

We machine and build precision metal optics and optical assemblies including mirrors, telescopes, gimbals, black bodies, scan mechanisms and collimators. We machine flat, aspheric or spherical beryllium mirrors which can be nickel plated, polished, heat treated and anodized in-house. With diameters ranging from 3in to 5ft, our telescopes support critical airborne and space-based targeting, surveillance and observation missions. Operating within a 120,000 sq ft, environmentally controlled manufacturing facility, we provide complete in-house manual and computer numerically-controlled (CNC) fabrication services.

Precision Machining
In-House Capabilities

EDM
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Lapping
Milling
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Brazing
Ion Milling
Photo Etch
Precision Cleaning
Heat Treat
Stress Relieve
Thermal Cycling
Assembly
Chem. Mill / Etch
Plasma Spray
Anodize
Chromate Conversion Coating
Nickel Sulfamate Plate
Nickel Plate
Gold Plate
Black Nickel Plate
Copper Plate/Strike
Passivation
Painting

16


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LABORATORIES INC
MATERION
Hardric Laboratories was founded in 1954 in Waltham, Massachusetts, to design and manufacture complex machined parts from exotic materials including Beryllium.
In 1980, Hardric developed its process for polishing bare beryllium for use on highly accurate scanning mirrors and other applications.
Using the most advanced machines available. Hardric's capabilities includes CNC Milling Centers up to 5-axis, CNC Turning and multiple Wire EDM machines.
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Working with difficult materials is our specialty!
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American precision at its best
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Large Pallet Sizes
All Machines Equipped with Dust Collection System
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Ultra-Precision Machining
L.A. Gauge provides customers with a one-stop-shop for precision machining. With stringent controls and state-of-the-art equipment, we avoid part stress and micro-cracking to provide high quality components.
Our capabilities include:
CNC 4 and 5 Axis Milling
CNC Turning
Electron Discharge Machining (EDM)
O.D. and I.D. Grinding
Jig Grinding
CNC Jig Boring
M 3.5 (Class 100) Clean Room
Temperature Controlled Metrology Lab
18


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PEREGRINE
20th ANNIVERSARY
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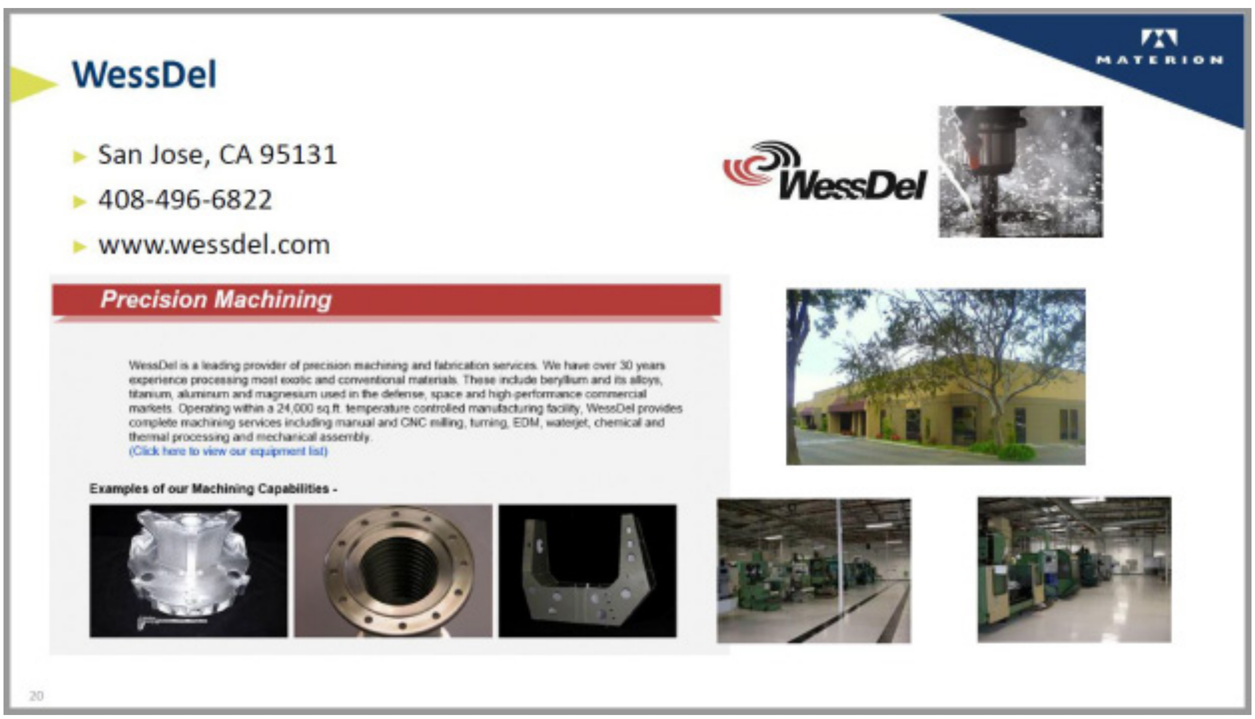
Conventional and CNC Machining through 5-Axis
EDMing; plunge and wire
Grinding; cylindrical and surface
Gun Drilling
Metallurgical Joining; soldering and brazing (ATM and VAC)
Liquid Interface Diffusion Bonding, Diffusion Bonding, GTAW, MIG, TIG and EB Welding
Epoxy Bonding
Finishing: surface prep, chem film, primers, thermal coatings, dielectric coatings
Thermal Processing: heat treat, stress relief, hot form, and super plastic forming
Others; per customer requirements
Type of Testing
Thermal Vac
Tensile testing: room temperature and elevated temperatur
Thermal Cycling
Dye Penetrant (both special level and standard level)
Ultrasonic Testing
Thermography
Radiographic
Neutron
Vibration
Other (product specific)
Design and Analysis
Computer Aided Engineering/Modeling
Simulation/Analysis
Linear Static Stress Analysis (tension, compression, torsion and bending
Vibration (dynamic) Analysis
Nonlinear and Buckling Analysis
Thermal Analysis (conduction, convection and radiation)
Fatigue Analysis o CFD Fluid Flow Analysis
FMEA, Failure Mode Effect Analysis
Fracture Toughness Analysis
Design Optimization
Computer Aided Manufacturing
Computer Aided Inspection
19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WessDel
San Jose, CA 95131
408-496-6822
www.wessdel.com
WessDel
Precision Machining
WessDel is a leading provider of precision machining and fabrication services. We have over 30 years experience processing most exotic and conventional materials. These include beryllium and its alloys, titanium, aluminum and magnesium used in the defense, space and high-performance commercial markets. Operating within a 24,000 sq.ft. temperature controlled manufacturing facility, WessDel provides complete machining services including manual and CNC milling, turning, EDM, waterjet, chemical and thermal processing and mechanical assembly.
(Click here to view our equipment list)
Examples of our Machining Capabilities -
20

Rev Manufacturing Inc.

MATERION

REV MANUFACTURING

- Palmdale, CA
- 661-272-0200
- www.revmfg.com

5 Axis Milling shop, Chemical Machine shop California, USA, Job Shop / Contract Manufacturer

Rev Manufacturing's first priority is quality. We try our best to ensure that the quality of our service and parts are what you expect. Our work will speak for itself. Every bit of our process from start to finish has an open door for our customers to be involved in which ever way they need to be. We look forward to working closely and building relationships to ensure that you get what you're entitled to as our customer. Quality First! Update 3/3/2015 We are currently implementing AS9100 and will be certified this year.

- Turning – CNC to 0.0001"
- Milling – 3,4,5 axis CNC
- Honing – to .00005"
- Lapping to 0.00005"
- Wire EDM
- Be AlBeMet Ti Mg SS

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Tinsley Custom Optics - Coherent Inc.

MATERION

- Coherent Inc.
- Richmond, CA 94806
- www.Coherent.com

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Superior Reliability & Performance

We provide design-to-specification and build-to-print subsystems for aspheric, off-axis lenses, objectives, and mirror-based optical systems. Wavelength and applications range from UV-VIS-IR to multispectral and hyperspectral imaging and non-imaging systems.

The technology for creating high-performance optical surfaces is a sophisticated, computer-driven manufacturing, testing and mea-sure-ment technique called "Computer-Controlled Optical Surfacing" (CCOS). The CCOS process is capable of processing materials of all types including optical glasses, germanium, silicon, stainless steel, beryllium, copper, nickel-plated substrates and silicon-clad silicon carbide. In addition to CCOS, Single Point Diamond Turning (SPDT) and con-ventional optical polishing techniques are employed, depending on requirements.

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Beryllium Alloys, other fabrication

- BeCu Alloys: **Materion Performance Alloys** – Elmore, OH, Reading, PA, Lorain, OH, Chicago, Detroit
- BeCu alloys: **NGK Berylco** – Sweetwater, TN:

- Cast AlBe, BeCu: **IBC Advanced Alloys** – Wilmington, MA, Franklin, IN, Philadelphia, PA

Copper Alloys

Beryllium Alloys

- Al-Be Machining: **AMM Advanced Metals Machining** - Olyphant, PA
- Al-Be Machining: **ARC** – Minneapolis, MN
- Wire EDM: **Skinner Machining** - Cleveland, OH

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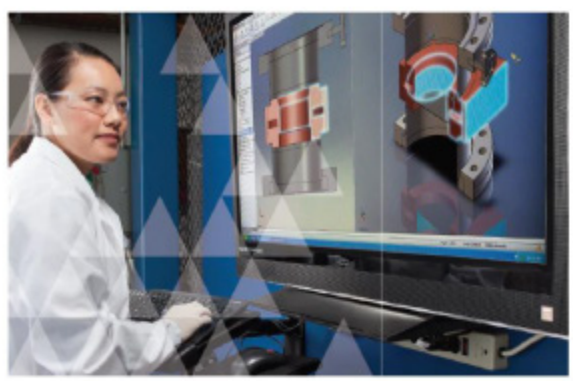
Applications Engineering and Program Management

- **Materion - Performance Solutions**
  - **Fabricated Solutions**:
    - Application engineering, Program management, engineering design
  - **Electrofusion:**
    - Application engineering, Program management, engineering design
- **US Industry**:
  - **All fabricators** maintain various levels of program management and applications engineering support to customers.

"Supporting beryllium end users"

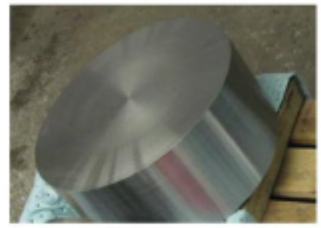


24

Materion Beryllium Grades

Beryllium:

- **Nine commercial grades of Beryllium metal:**
  - **S-65, S-65 H**, S-200-F, S-200-F-H, S-200-F-C, I-70,
  - I-220, I-220-H
  - Block, Parts, Sheet, Extrusions
- **Beryllium Foil** – multiple grades (Materion Electrofusion)
- Ultra High-purity Beryllium
- Beryllium hydroxide
- Beryllium fluoride
- **AlBeMet** – Be-38Al
- **AlBeCast** – Investment cast Al-Be
- Be/BeO Composites (E-Material)
- **BeO** ceramic components
- **Cu-Be** Alloys



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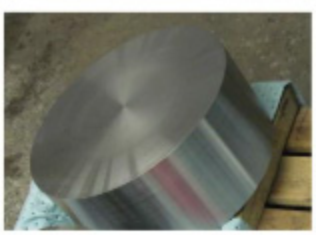
Specifications



SPEC SHEET

S-65
BERYLLIUM GRADE

Effective: April 11, 2018 Rev. 5

1. SCOPE

This specification defines the requirements for a structural grade of hot pressed beryllium block which is designated S-65. This material is recommended for applications requiring higher purity.

2. CHEMICAL COMPOSITION

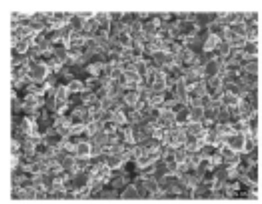
2.1. The chemical composition shall conform to the following:

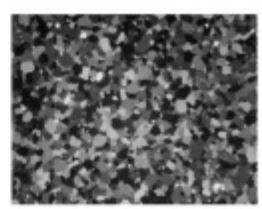
Beryllium Assay, % minimum (1)	99.2
Beryllium Oxide, % maximum (2)	0.9
Al, % maximum (3)	0.05
C, % maximum (4)	0.09
Fe, % maximum (3)	0.08
Mg, Cr, each % maximum (3)	0.01
Ni, Cu, Ti, Zr, each, % maximum (3)	0.025
Zn, Mn, Ag, Co, Pb, Ca, Mo, each, % maximum (3)	0.005
Silicon, % maximum (3)	0.040
U, % maximum (5)	0.015
Other Metallic impurities, each, % maximum (3)	0.04

Note:
(1) Difference (i.e. 100%-other elements)
(2) Leco Inert Gas Fusion
(3) Spectrochemical Methods
(4) Leco Combustion
(5) Glow Discharge Mass Spectrometry, Neutron Activation Analysis or Fluorescence

MATERION BERYLLIUM & COMPOSITES

MATERION BRUSH INC.





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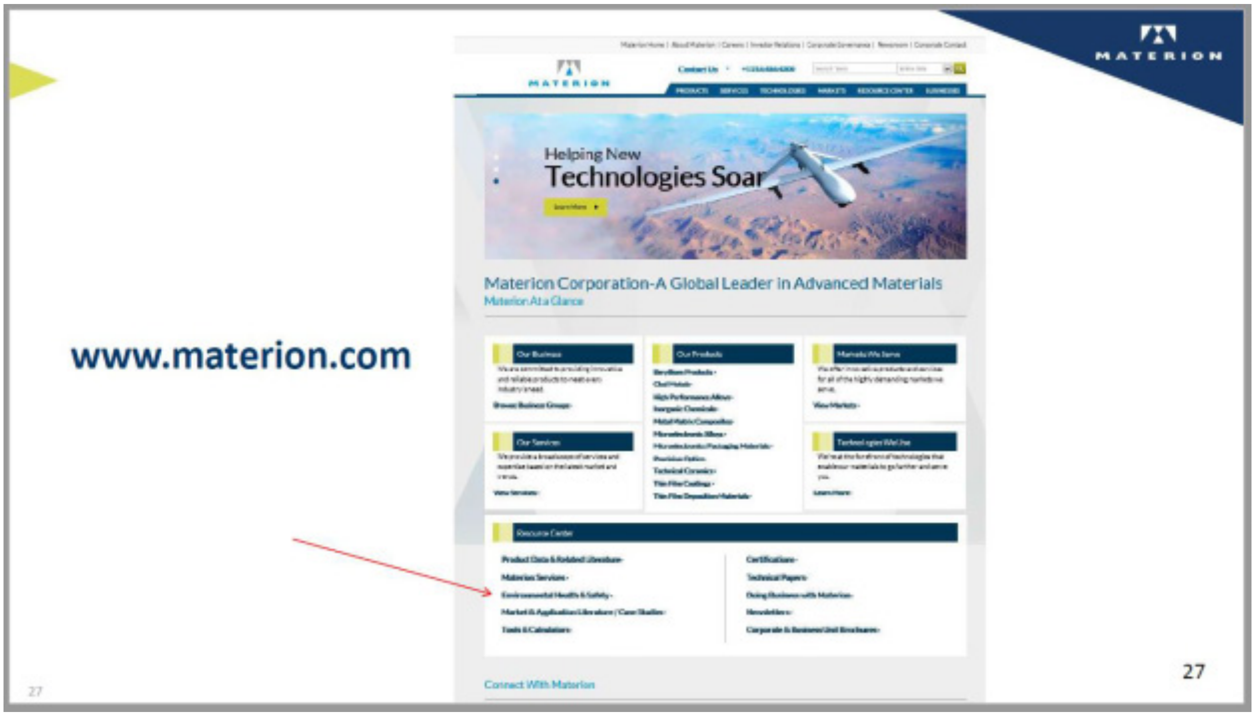
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Status of the BP1 Pebble Production

Keigo NOJIRI

NGK Insulators, Ltd., 1 Maegata-cho, Handa, Aichi 475-0825 Japan

Beryllium metal is used in various industrial and R&D scenes due to its unique and extremely excellent properties. One of the representative properties is a nuclear reaction. Beryllium is candidate as neutron multiplier in ITER. Beryllium neutron multiplier will be used in form of pebbles, small spheres of high quality.

"BP-1" beryllium pebble is the reference material for the multiplier of the TBM (Test Blanket Module) for ITER. The specifications of "BP-1" shall contain a minimum beryllium content of 99.0%. And it should be a sphere made by REP (Rotation Electrode Process) with diameter of 0.8-1.2mm. REP is a method to produce metal sphere. High voltage electric power is charged between the both electrodes. The rotating beryllium electrode ejects beryllium droplets by centrifugal force. The droplets of melted beryllium form into spherical shapes before solidification.

When beryllium pebble is adopted as neutron multiplier of TBMs, the beryllium pebble should be needed in the amount of several hundred kilograms. The productivity improvement is one of the most important issues for the pebble production. As the first plasma of ITER is approaching, the production development is now coming to its practical stage.

The details will be presented in this workshop.

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JAPAN

25th October 2019, Queen Mary, USA

"KUROKO-kun"

NGK kero/dwarf

Status of the BP1 Pebble Production

Keigo NOJIRI
NGK Insulators, ltd
Aichi Japan

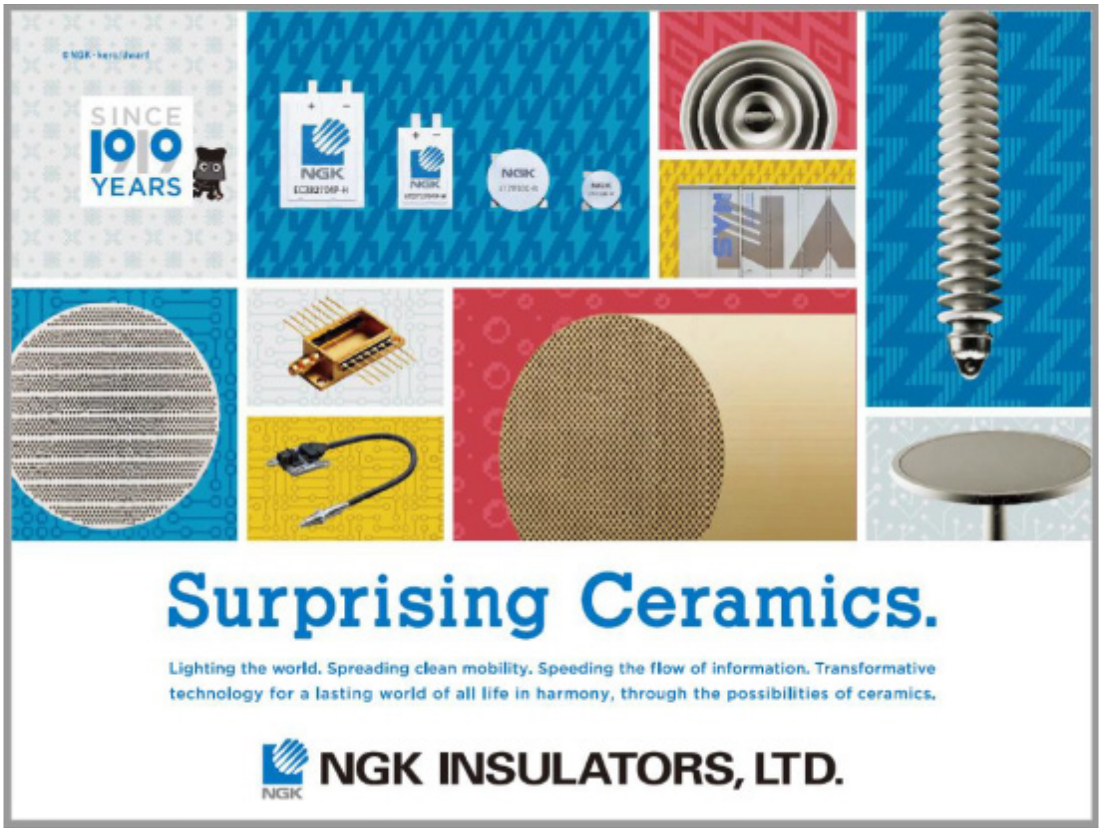
NGK INSULATORS, LTD.

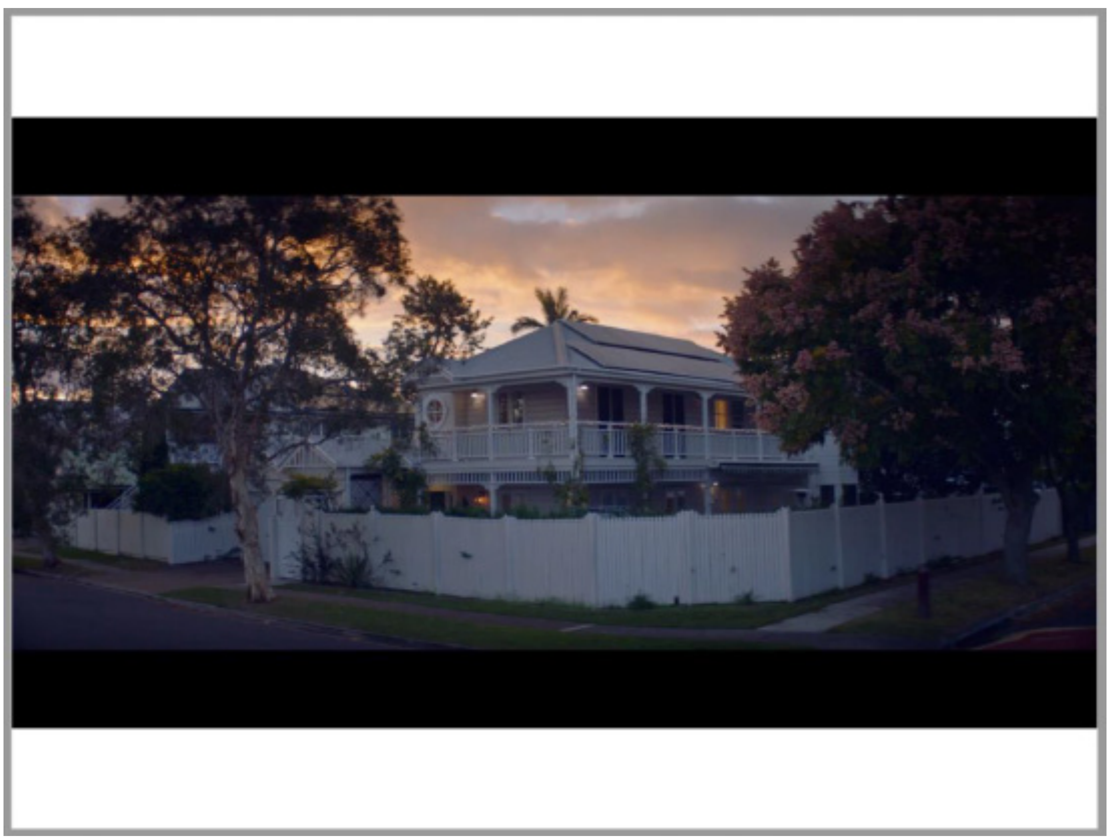
Outline of NGK

Company Name	**NGK INSULATORS, LTD.**
Date of Establishment	**May 5, 1919**
Paid-in Capital	**69,849 Million Yen**
Representative Directors	President **Taku Oshima** Executive Vice President **Yukihisa Takeuchi** **Hiroshi Kanie**
Number of Employees (consolidated)	**20,115** ※Outside Japan employees **63%**
Consolidated Subsidiaries	**57 companies** ※Outside Japan Subsidiaries **36**

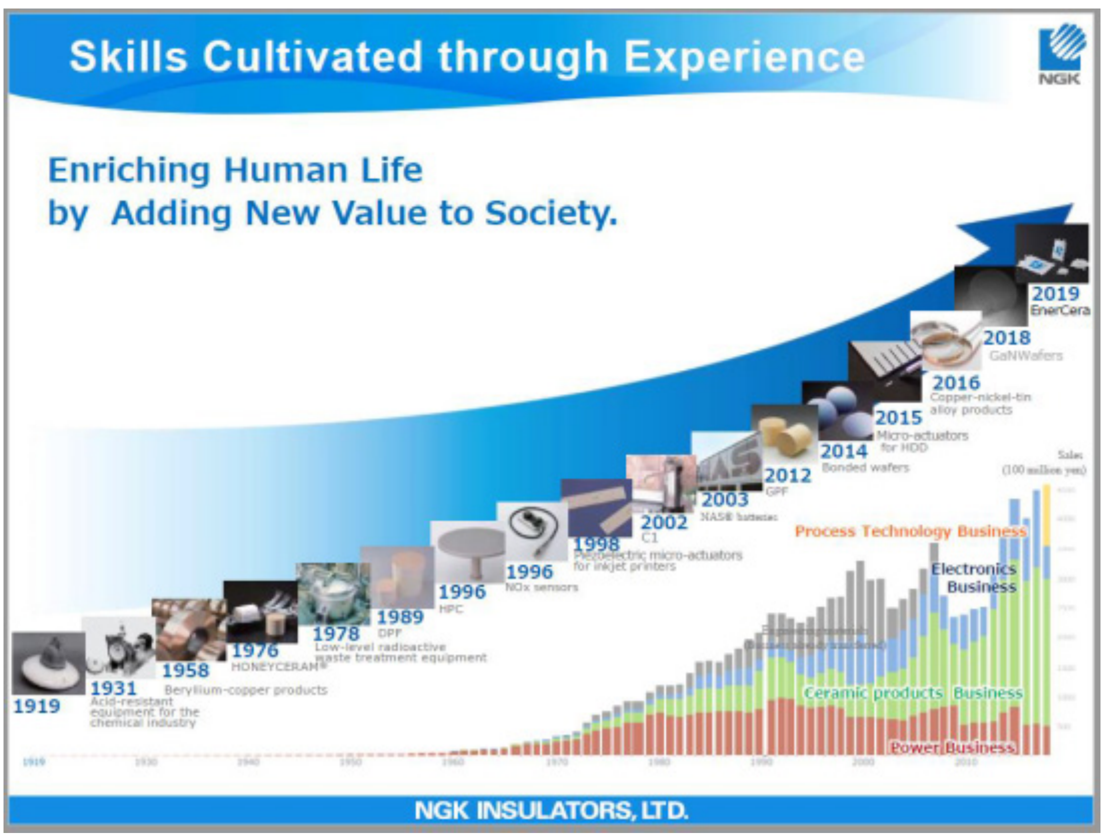
As of September, 2018

2


SINCE
1919
YEARS
Surprising Ceramics.
Lighting the world. Spreading clean mobility. Speeding the flow of information. Transformative technology for a lasting world of all life in harmony, through the possibilities of ceramics.
NGK INSULATORS, LTD.

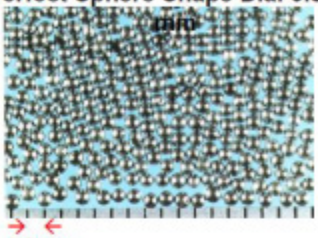



Contributing to society
NGK
1919
NGK was created to help Japanese society modernize by meeting the growing demand for electricity
A piece of the insulator that sparked the foundation of NGK Insulators. (1905)
The first president Kazuchika Okura stated,
"It is our duty to our country to produce insulators in Japan."
▲Workers put the finishing touches on insulators in the early days.
◀The first tunnel kiln to be installed at our factory at company headquarters. (1920s)
NGK INSULATORS, LTD.


Skills Cultivated through Experience
NGK
Enriching Human Life
by Adding New Value to Society.
2019 EnerCera
2018 GaNWafers
2016 Copper-nickel-tin alloy products
2015 Micro-actuators for HDD
2014 Bonded wafers
2012 GPF
2003 NAS® batteries
2002 C1
1998 Piezoelectric micro-actuators for inkjet printers
1996 NOx sensors
1996 HPC
1989 DPF
1978 Low-level radioactive waste treatment equipment
1976 HONEYCERAM®
1958 Beryllium-copper products
1931 Acid-resistant equipment for the chemical industry
1919
Sales (100 million yen)
Process Technology Business
Electronics Business
Ceramic products Business
Power Business
NGK INSULATORS, LTD.

NGK BP1 Pebble

Perfect Sphere Shape Dia. 0.8-1.2 mm

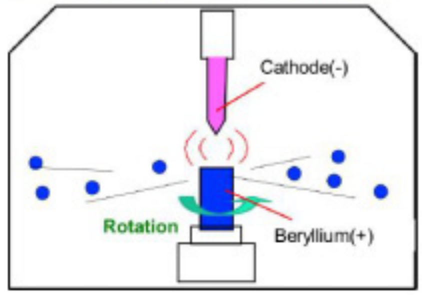


1.0mm

Chemical composition(wt%)

Beryllium	≥ 99.0
Beryllium Oxide	≤ 0.50
Aluminum	≤ 0.09
Cobalt	≤ 0.001
Iron	≤ 0.10
Magnesium	≤ 0.08
Silicon	≤ 0.06
Uranium	≤ 0.005

Method : Rotation Electrode Process



Properties

Density : 1.79 – 1.80 g/cm^3
Sphericity : 0.010 – 0.012 mm
Grain Size : 0.4 – 0.6 mm
Compressive Strength : 11 – 14 kgf

BP1 is the most suitable pebble for ITER evaluation stages by its Shape, Purity, Stable Qualities

NGK INSULATORS, LTD.

Development Road Map

Basic Study → REP Prototype → Production of samples: 1990'～

Basic parameter optimisation
- Pebble size control 1 (1.0mm)
- Yield improvement

Productivity improvement
- Bigger Electrode
- Higher Operation Ratio ← Today
- Pebble size control 2 (smaller/bigger)

Mass Production Preparation
- Mass Production Equipment Design
- Preparation of Place, Equipment, Workforce, etc.

Mass Production

NGK INSULATORS, LTD.


Development Progressing,,,
NGK
Surprising Ceramics.
NGK
日本ガイシ
NGK INSULATORS, LTD.

Studies for Production of Billets and Articles from Tantalum and Titanium Beryllides

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E-mail: FrantsEV@ulba.kz

Abstract
The research results of tantalum and titanium beryllides synthesis and production of billets and articles from these materials are represented in this scientific work. It shows that articles from intermetallic compounds of compositions Ta_2Be_{17}, $TiBe_{12}$ can be obtained using powder metallurgy methods. Conditions for conducting synthesis of beryllides, x-ray structural analysis results, microstructural studies results, microhardness, and electrical conductivity are listed. Methods and techniques of the produced billets machining are described in this scientific work.

Keywords: beryllium, tantalum, titanium, beryllide, synthesis, x-ray structural analysis, microstructure, powder metallurgy

1 Introduction
In the recent ten-year period interest to heat-resistant beryllides, especially to titanium and tantalum beryllides grew significantly.

Titanium beryllide ($TiBe_{12}$) is considered as the alternative to beryllium material of neutron multiplier in the breeder of international fusion reactor DEMO. Based on [1] the material is rather less “swelled” under neutron radiation and is less capable to hold tritium in comparison to pure beryllium.

Application of Ta_2Be_{17} resulted from using it as the material to fabricate and manufacture high-temperature components for the aerospace industry. As this material has a relatively low density (5.05g/cm^3), high melting temperature (1990°C) and high heat resistance. Depending on the work duration, its temperature modes lie in the range from 1300 to 1700ºC and are 1.5 times higher than those allowed for heat-resistant alloys of niobium, cobalt, and nickel [2].

Usually those materials were handled as small laboratory samples without any assessment of possible large-scale manufacture.

Studies earlier (in early 2000) conducted at UMP JSC on producing billets of $TiBe_{12}$ by casting showed that there was a principal technical possibility to produce ingots of big size. With that there was a notice that despite using heat insulation for side part of a mold and forced cooling-down of a bottom part, ingots of titanium beryllide tend to form a shrink-hole of a big size. Some had cracks. As an example, see Fig. 1 with the image of a central part of Ta_2Be_{17} ingot with casting defects visible.

In parallel with producing cast billets, we worked on fabricating a block of titanium beryllide using the methods of powder metallurgy. This paper is based on the successful experience of those years.

Figure 1. Ingots of titanium beryllide with casting defects.

2 Experimental Procedure

As initial materials, we used commercial powders of beryllium, ПТБ-56 grade and fragmented powders of tantalum, 5Б grade produced at UMP JSC. Be powder grain size made less than 56μm, that of tantalum, less than 40μm. Titanium powder of commercial grade ПТОМ-1 produced by reducing titanium oxide with calcium hydride was purchased in Russia; its grain size was less than 50μm. Table 1 below shows chemical composition of the powders used and Figures 2-4 show the view.

Table 1. Chemical composition of initial powders and their process properties.

Element	Material (Lot No.)		
	Be (56) Assay, wt. %	Ta (25) Assay, wt. %	Ti (ПТОМ-1) Assay, wt. %
Be	98.93	--	--
Si	0.025	<0.0003	0.10
Mn	0.012	<0.00003	--
Fe	0.11	0.00036	0.20
Mg	0.019	<0.00008	--
Ni	0.018	0.000059	0.20
Pb	<0.005	--	--
Al	0.019	<0.0002	--
Cu	<0.01	0.000037	--
C	<0.05	--	0.05

O	0.78	--	--
F	<0.001	--	--
Cr	0.021	0.00015	--
Ti	--	<0.00008	Base
Sn	--	<0.00003	--
Nb	--	<0.0030	--
Zr	--	<0.0003	--
Ca	--	<0.0003	--
W	--	<0.0003	--
Mo	--	<0.0001	--
Co	--	<0.0001	--
N	--	--	0.08
H	--	--	0.35
Other Properties			
Specific surface, m^2/g	0.32	--	--
Bulk weight, g/cm^3	0.57	4.97	1.36
Bulk weight after bumping down, g/cm^3	0.98	--	1.62

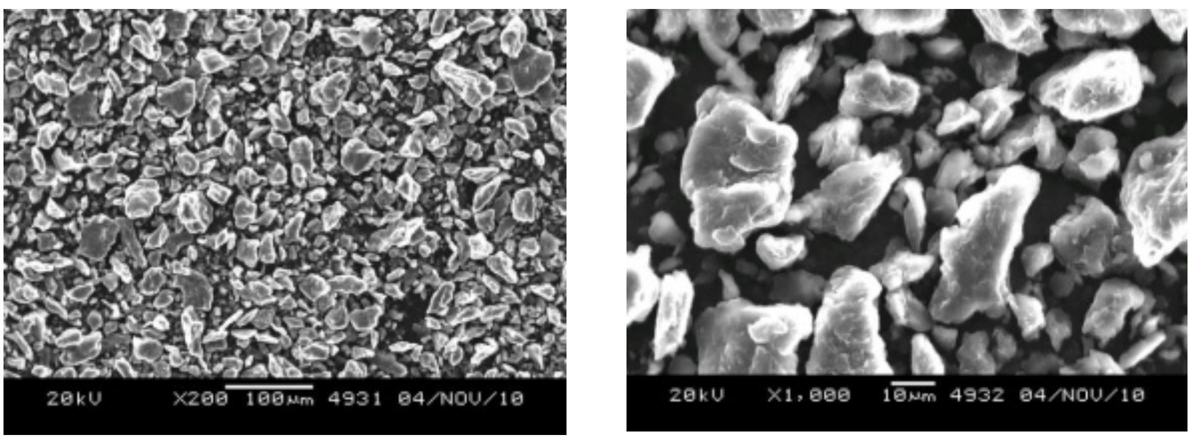


Figure 2. 200x and 1,000x photomicrographs of beryllium powder, ПТБ-56 grade.

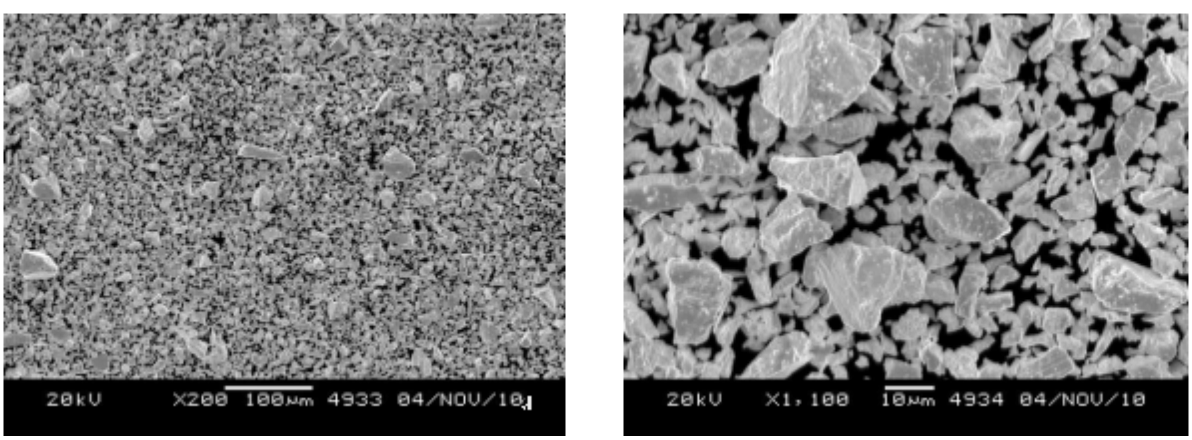


Figure 3. 200x and 1,000x photomicrographs of fragmented tantalum powder, 5 "Б" grade.

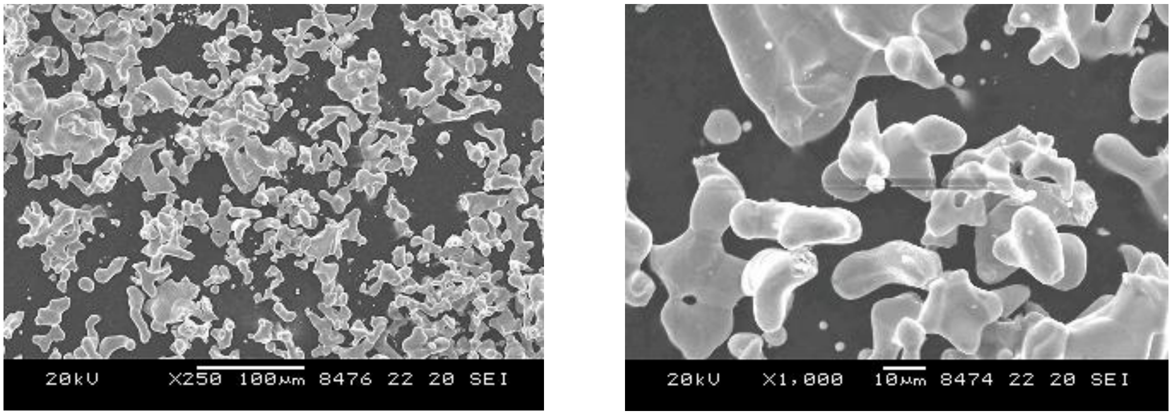


Figure 4. 250x and 1,000x photomicrographs of titanium powder, ПТОМ-1 grade.

The features of $TiBe_{12}$ synthesis of a mixture of initial powders of beryllium and titanium with the ratio of Be (70 wt. %) and Ti (30 wt. %) were studied at different temperatures with further x-ray structural analysis. Fig. 5 shows comparative diffraction patterns upon heating.

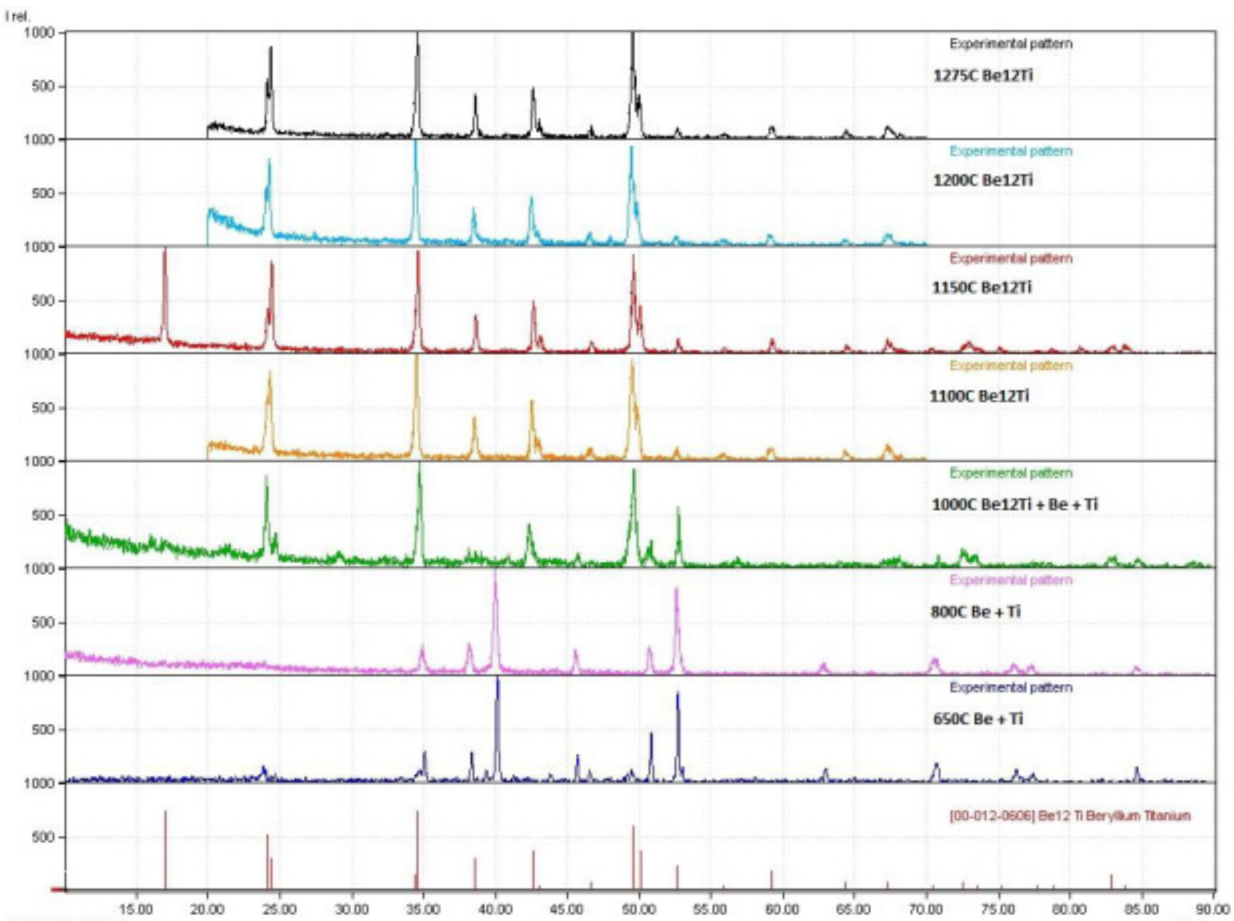


Figure 5. Diffraction patterns of a mixture of powders of Be (70 wt. %) and Ti (30 wt. %) after synthesis at different temperatures.

From the data shown in the figure above, you can see that full synthesis with producing low-temp tetragonal (I4/mmm) phase of $TiBe_{12}$ is at a temperature of 1100°C. With that no lower titanium beryllides could be found in diffraction patterns.

Same method was applied for studying the synthesis of a mixture of the powders of Ta (70 wt. %) and Be (30 wt. %), which made it possible to state that at a temperature of 1270°C, a compound of Ta_2Be_{17} is synthesized (see Fig. 6).

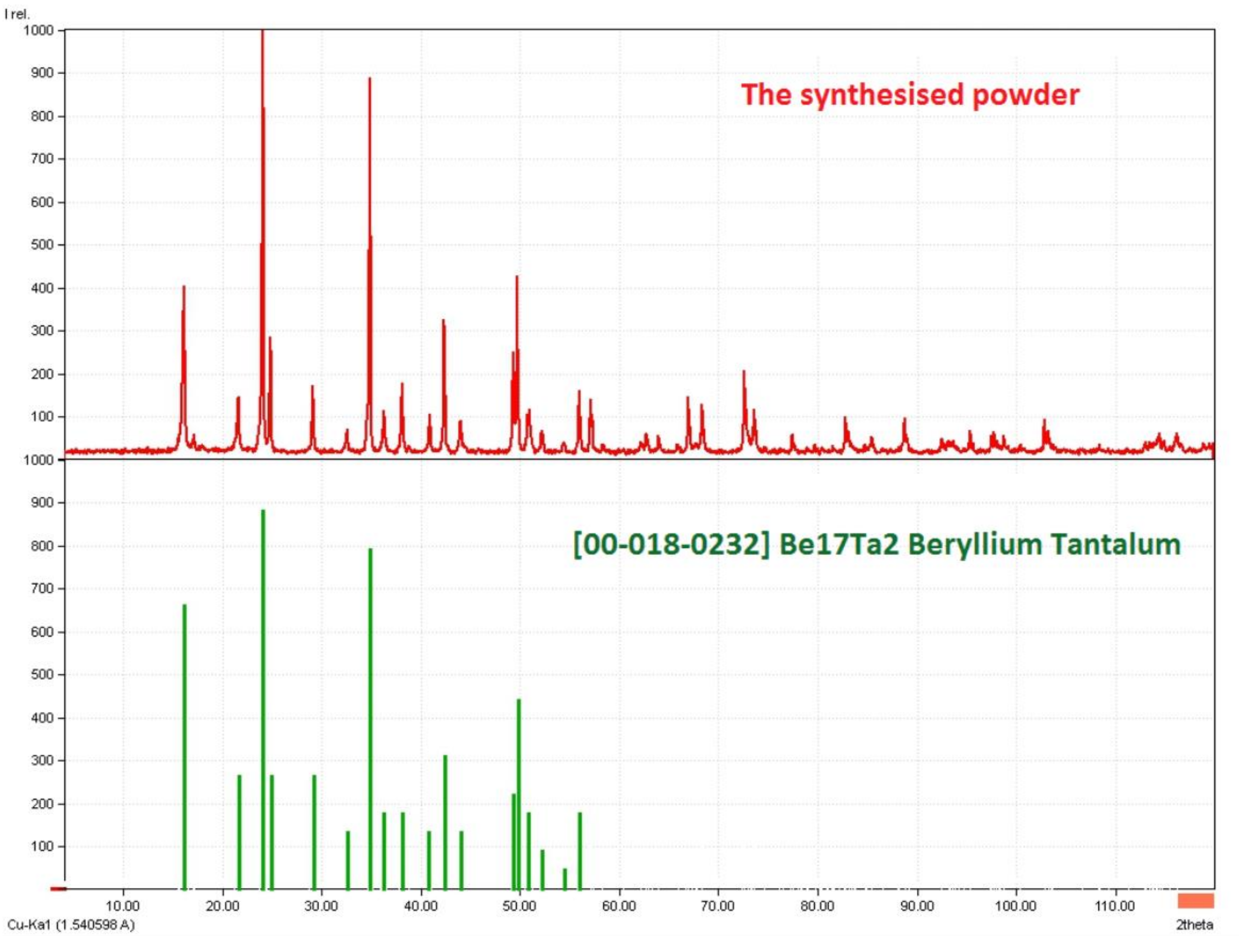


Figure 6. X-ray image of a mixture of powders of Ta (70 wt. %) and Be (30 wt. %) upon synthesis at a temperature of 1270°C.

To produce billets, synthesized powders of titanium and tantalum beryllides were hot-pressed. Fig. 7 shows the view of billets of titanium and tantalum beryllides.

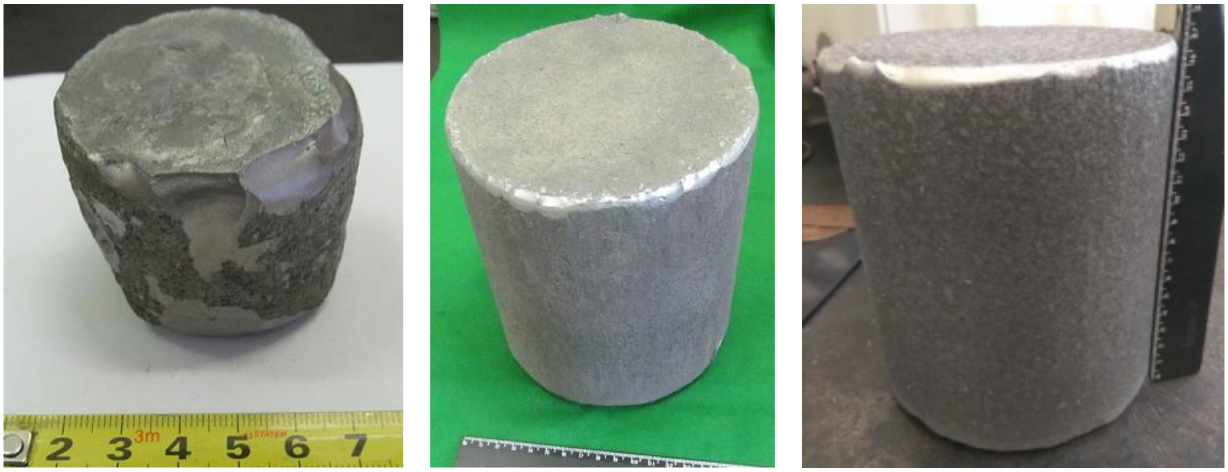


Figure 7. Images of $TiBe_{12}$ billets.

The density of hot-pressed billets made about 2.26g/cm^3, or 99.9% of theoretical density (TD). No defects such as cracks or pores were found in the structure. Figures 8-10 show images of the billets after fabrication and during their processing to become finished items.

The density of Ta_2Be_{17} billets made about 4.78g/cm^3 (or 94.7% of TD). However, a part of billets had shearing distortion. Fig. 8 shows images of these billets.

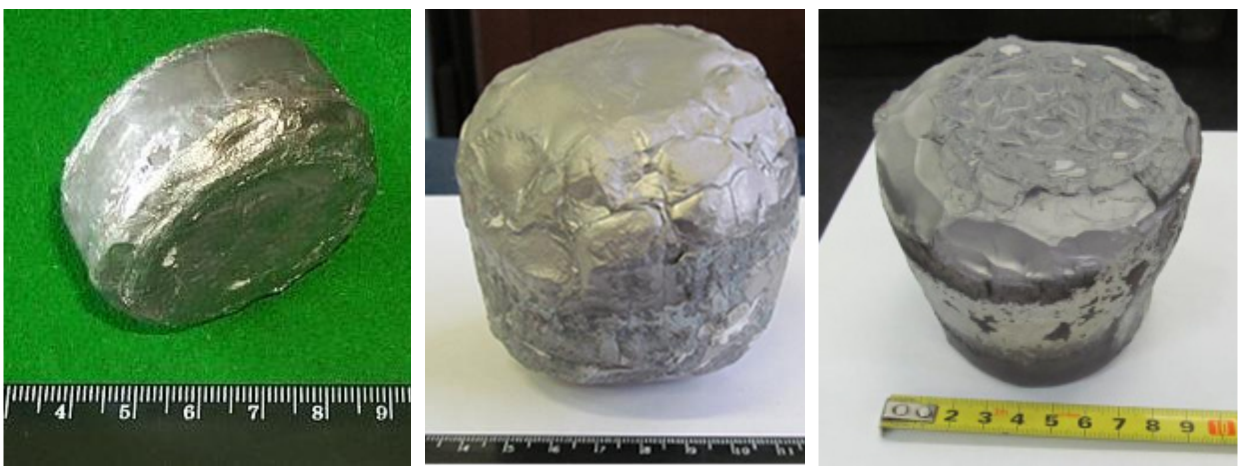


Figure 8. Images of Ta_2Be_{17} billets.

Because of high hardness of the material, it was not possible to cut out articles and items using standard cutting equipment (lathe and milling machine). Positive results were only achieved using wire EDM (electrical discharge machine). Fig. 9 shows a Ta_2Be_{17} billet cut out into samples and other pieces from this material.

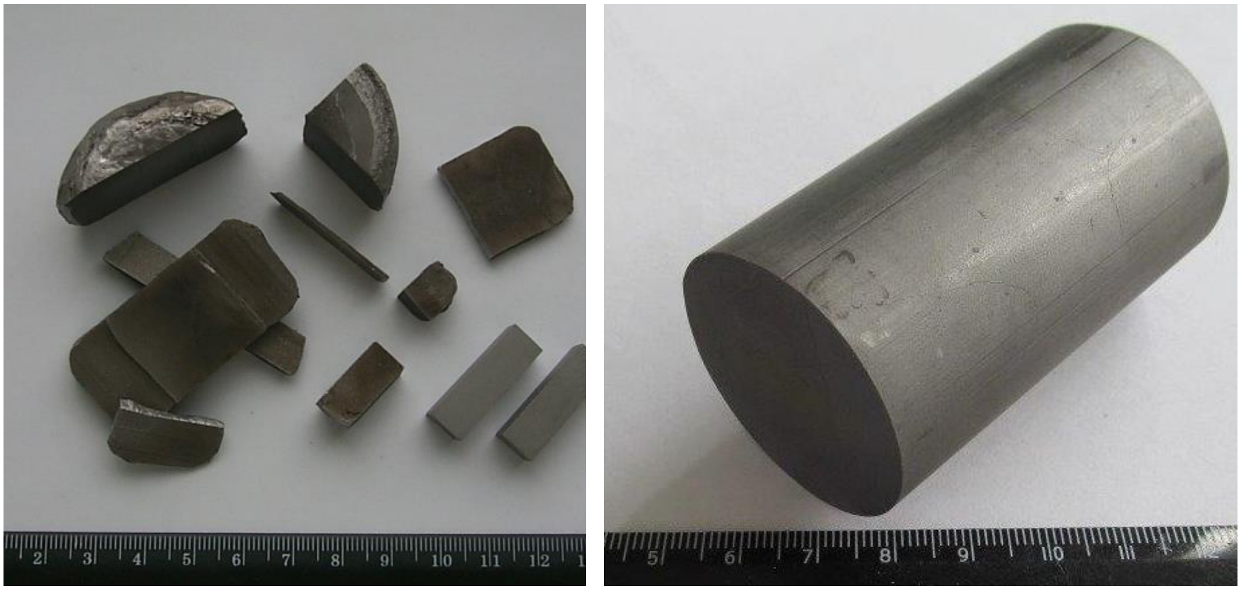


Figure 9. Images of Ta_2Be_{17} billets after cutting by wire EDM.

Holes in billets were made by hydro-abrasive cutting (see Fig. 10). With further wire EDM cutting, we made other $TiBe_{12}$ items.

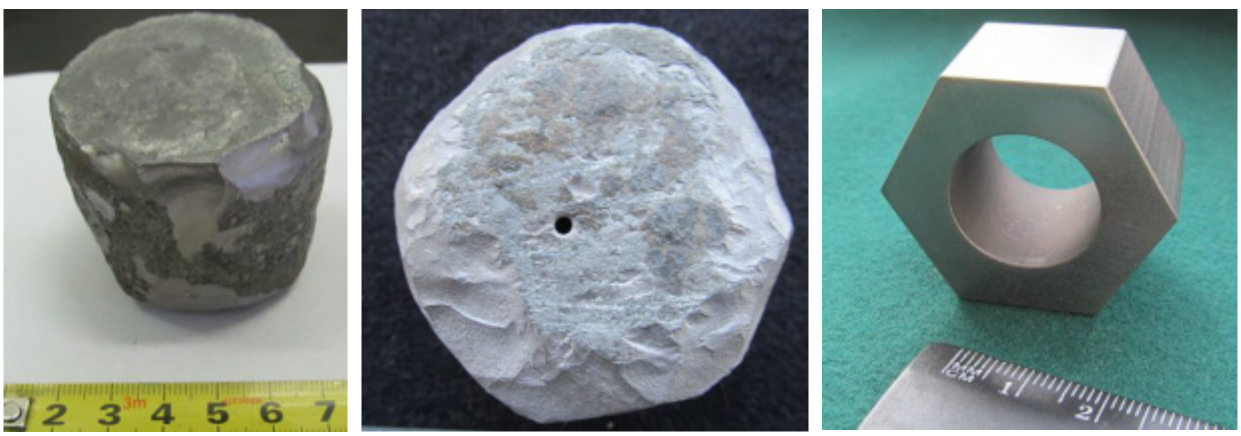


Figure 10. Images of an as-pressed $TiBe_{12}$ billet, first after putting in a central hole, and then as a finished item.

3 Physical & Mechanical Properties of the Materials

From Figs. 11-12, one can see that the material has clear grain structure. Angular chamfers on the grain boundaries are not pores, they are traces of the grain residue (including oxide) chipping during sample grinding. Average grain size of the material was 59.6μm with a micro-hardness of 1,152.8kg/mm^2. Material micro-hardness conforms with the data indicated in the references [3].

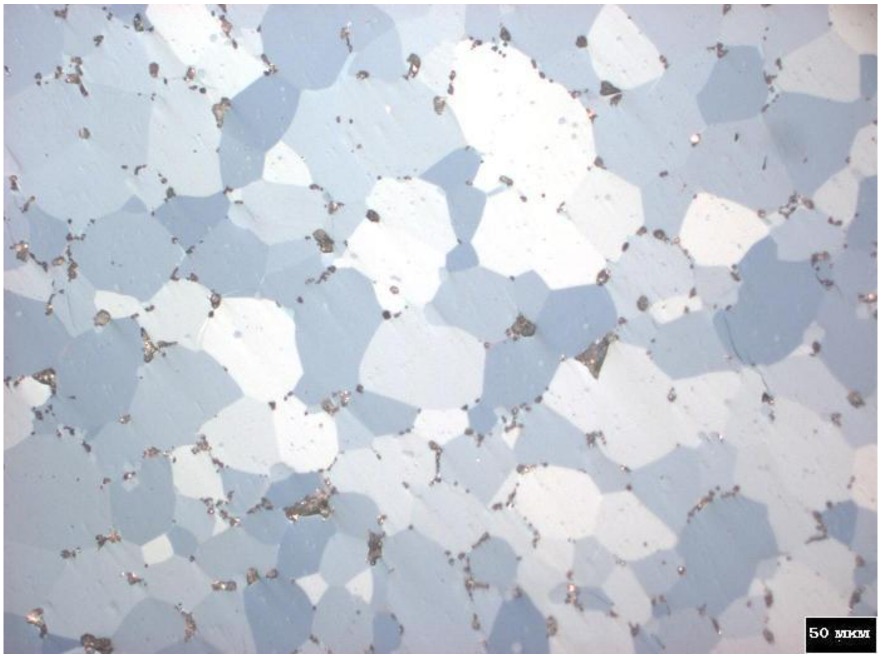


Figure 11. Microstructure of the Ta_2Be_{17} billet.

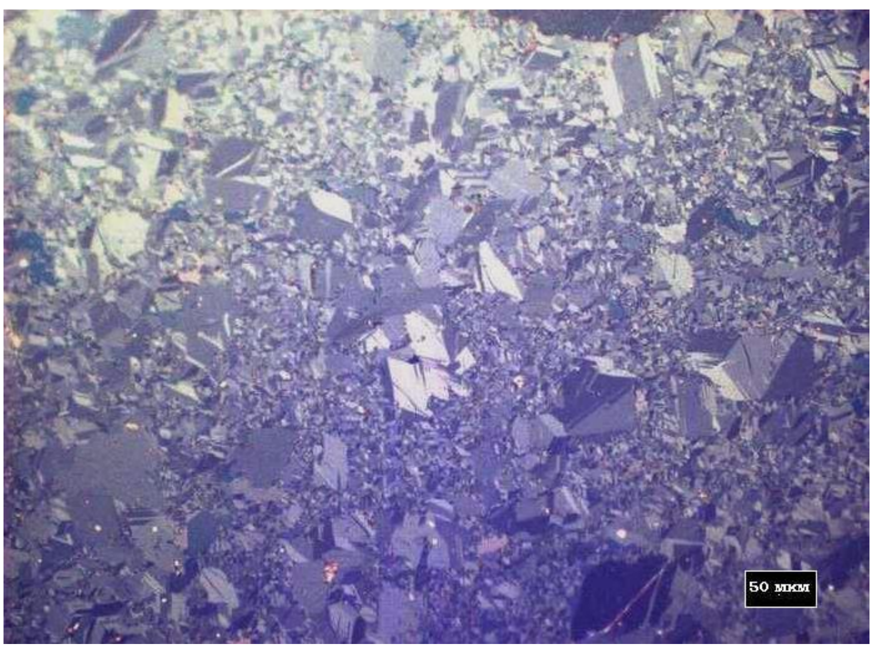


Figure 12. Microstructure of the $TiBe_{12}$ billet.

Average grain size of the material made was 16.2μm, with a micro-hardness of 691.2kg/mm^2. Table 2 shows the results of bend tests of Ta_2Be_{17} samples at a room temperature.

Table 2. Mechanical properties of Ta_2Be_{17} during bend testing.

Sample No.	Bend Strength, σ_B (MPa)	Offset Yield, $\sigma_{0.2}$ (MPa)	Sample Deflection (mm)	Elongation (%)
1	121.5	--	0.051	0
2	133.7	--	0.067	0

From the data indicated in the table above, one can see that the material is not plastic at room temperature. In addition, material bending strength is 1.6 lower than stated in the references, and the obvious reason is the larger grain size.

Depending on the density, electrical conductivity of Ta_2Be_{17} measured at room temperature varied in the range from 2.6 to 9.5 MS/m. The most solid sample had electrical conductivity of 9.5 MS/m. The electrical conductivity of $TiBe_{12}$ samples was about 5.06 MS/m.

Values obtained are lower than those for beryllium (about 17.0 MS/m), however, higher than of those for titanium, which are in the range of 1.25 to 2.6 MS/m. Stronger samples of Ta_2Be_{17} also have electrical conductivity higher than that of pure tantalum, which is about 7.4 MS/m.

Fig. 13 shows the technical coefficient of thermal expansion (CTE or КТЛР) Ta_2Be_{17}, and Fig. 14 shows it for $TiBe_{12}$. CTE (КТЛР) obtained values for $TiBe_{12}$ are close to those stated in the references and for Ta_2Be_{17}, values are also compatible with those as in the references, however slope of the curve is opposite.

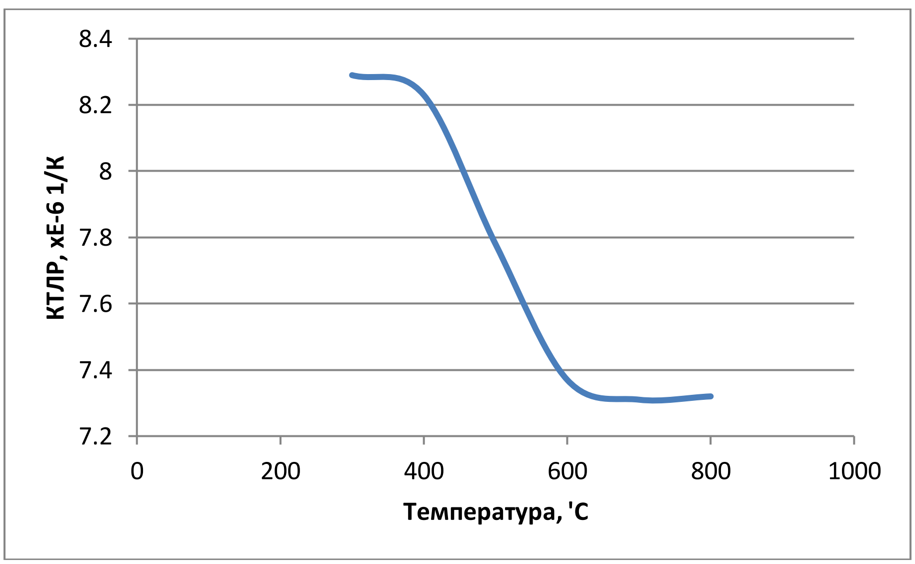


Figure 13. Variation of CTE (КТЛР) of Ta_2Be_{17} plotted versus temperature.

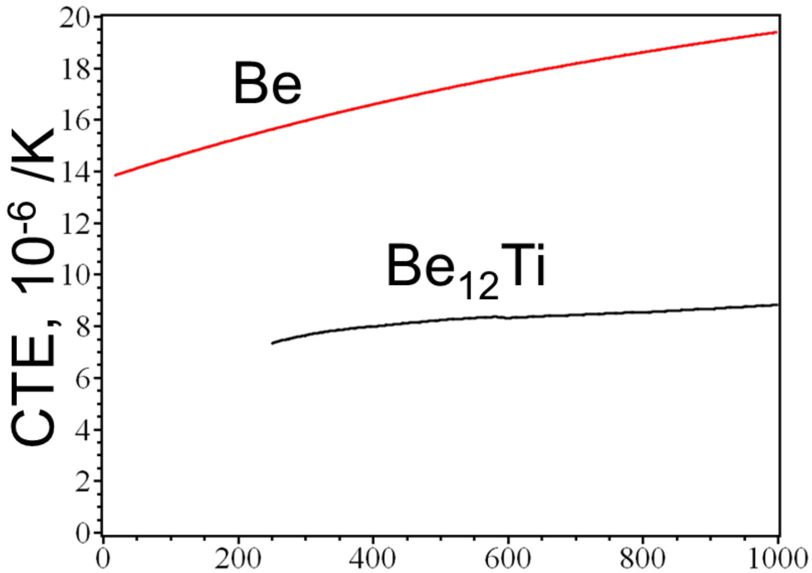


Figure 14. Variation of CTE (КТЛР) of $TiBe_{12}$ plotted versus temperature.

Table 3 shows the results of defining corrosion resistance of Ta_2Be_{17} in the oxidizing atmosphere at a temperature of 1300°C within 30 hours.

Table 3. Results of defining corrosion resistance of Ta_2Be_{17} in an oxidizing atmosphere at a temperature of 1300°C.

Testing time (h)		**10**	**15**	**20**	**25**	**30**
Weight increase	**(mg)**	3.08	5.29	7.29	9.05	11.23
	(%)	0.84	1.45	1.99	2.47	3.07

As can be seen from the data shown in Table 3 above, the produced material has good stability when heated in the oxidizing medium in comparison with the initial materials: tantalum and beryllium, which are fully burnt-out in red heat. Metallography study showed that the average grain size of samples and average value of micro-hardness of samples upon heat treatment in air at a temperature of 1300°C within 30 hours remain as initial samples level.

3.1 Thermal Cycling

Experiments were conducted on thermal cycling heating of $TiBe_{12}$ samples with the size of 20mm diameter x 20mm. Heating mode was as below:

- Heating-up from 200°C to 900°C for 60 sec.
- Hold at 900°C for 45 sec.
- Cooling down from 900°C to 200°C for 60 sec.

With the above conditions, 50 cycles of heating and cooling were performed. Heating took place in an induction-type furnace with air medium inside. Cooling was done with compressed air. The sample remain integral upon testing. No defects like cracks or flakes were found. Fig. 15 shows sample heating status during thermal cycling testing, surface view upon oxide layer removal as well as end surface view in the edge area magnified by 25x.

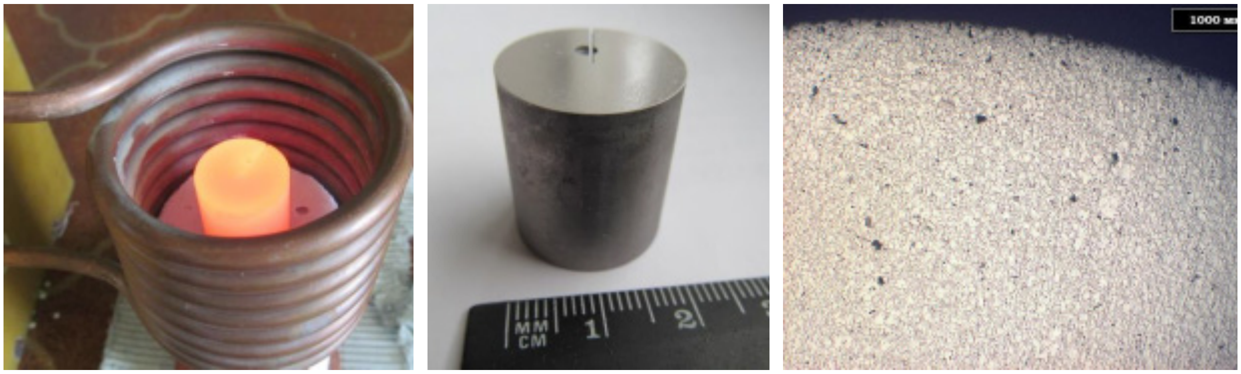


Figure 15. Views of test samples during thermal cycling.

4 Summary

In conclusion, some bullet point observations about this study:

- A casting process does not make it possible to produce billets of Ta_2Be_{17} and $TiBe_{12}$ applicable for items fabrication and manufacture. Best results could be achieved by vacuum hot-pressing of the synthesized powders.
- Synthesis of $TiBe_{12}$ happens at a temperature of 1100°C.
- Synthesis of Ta_2Be_{17} happens at a temperature of 1270°C.
- The UMP JSC process makes it possible to produce items of $TiBe_{12}$ with the density of about 99.9% of TD and Ta_2Be_{17} with the density up to 94.7% of TD.
- Wire EDM and hydro-abrasive cutting could be used effectively for machining the billets of Ta_2Be_{17} and $TiBe_{12}$.

References

[1] P. Kurinskiy. Swelling, thermal desorption, and mechanical properties of Titanium Beryllide with high-dose neutron irradiation. Ph.D. Thesis in Engineering Science, p. 143, Ulyanovsk, 2012.

[2] K.A. Walsh. Beryllium Chemistry and Processing, ASM International, 2009.

[3] I. Papirov (Ed.). Structure and Properties of Beryllium Alloys: Manual. Moscow, "Energoizdat", 1981.

STUDIES FOR PRODUCTION OF BILLETS AND ARTICLES FROM TANTALUM AND TITANIUM BERYLLIDES

E. FRANTS, B. ZORIN, M. KOLMAKOV, S. UDARTSEV, G. FOMINYKH, I. ARINOVA, A. VECHKUTOV

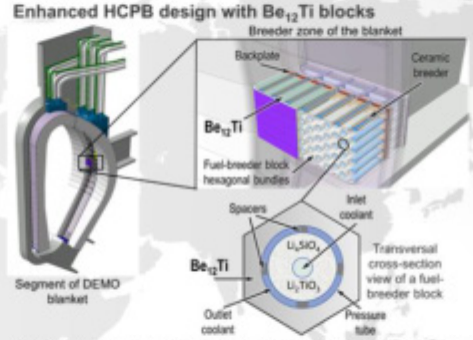
ULBA METALLURGICAL PLANT (UMP), 102, ABAY AV., 070005 UST-KAMENOGORSK, KAZAKHSTAN

$Be_{12}Ti$ – material of neutron multiplier in DEMO reactor

$Be_{12}Ti$ works at higher operating temperatures (900°C) in comparison with Beryllium (650°C)

Has better characteristics to release Tritium in comparison with pure Be

Enhanced HCPB design with $Be_{12}Ti$ blocks



Solid beryllide blocks have been proposed to be used as neutron multiplier in the updated HCPB blanket for the DEMO. The key issue for the implementation of the new breeding blanket design is **the lack of industrial technology** for the production of **massive hexagonal $Be_{12}Ti$ blocks** (Ø144 mm × 150 mm).

$Be_{17}Ta_2$ – material for fabricating high-temp items for aerospace applications

Casting of $Be_{12}Ti$

Casting method makes it possible to produce ingots of significant sizes with larger shrink cavities and cracks.

Powder metallurgy to produce $Be_{12}Ti$ & $Be_{17}Ta_2$ by vacuum hot pressing

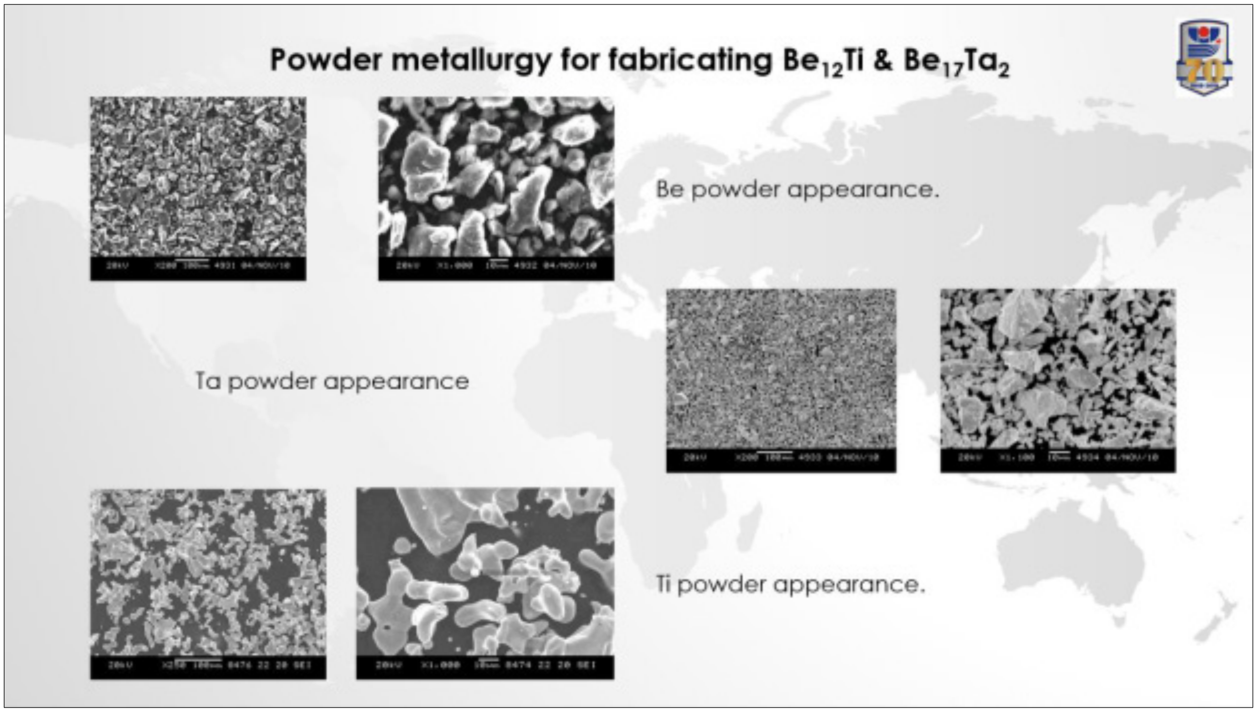
Be powder, PTB-56 grade – standard products by UMP JSC to manufacture items of structural Be

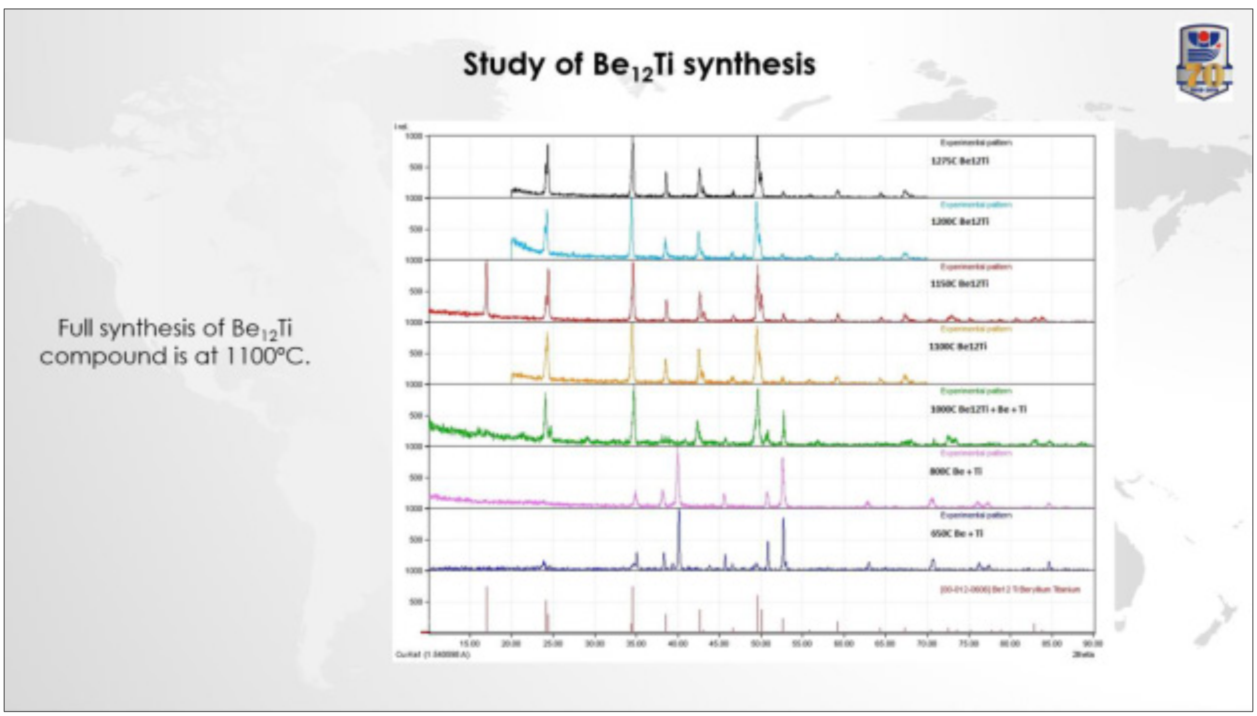
U mass fraction in PTB-56 powder is ≤ 1 ppm

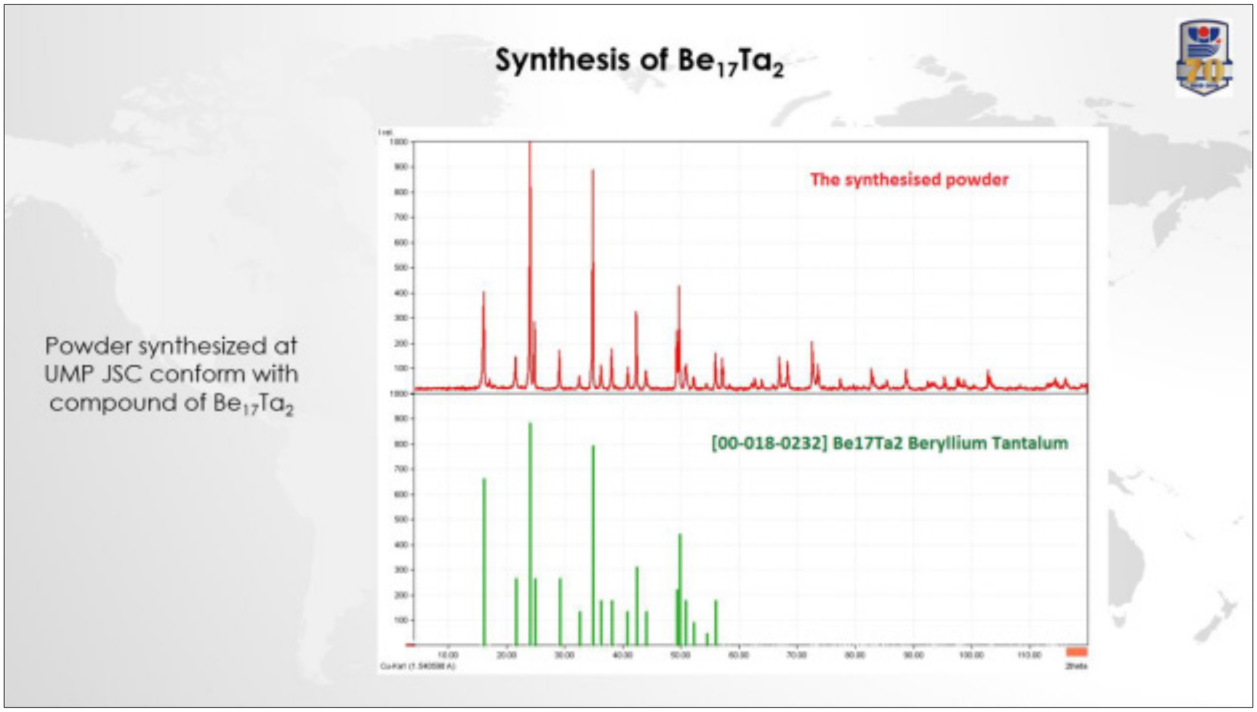
	Chemical composition, ppm												
	Be	Si	Mn	Fe	Mg	Ni	Pb	Al	Cu	C	O	F	Cr
Be	98,93%	250	120	1100	190	180	<50	190	<100	<500	7800	<10	210
Ta	-	<3	<3	3,6	<8	59	-	<20	37	-	--	-	1,5
Ti	-	1000	-	2000	-	2000	-	-	-	500	-	-	-

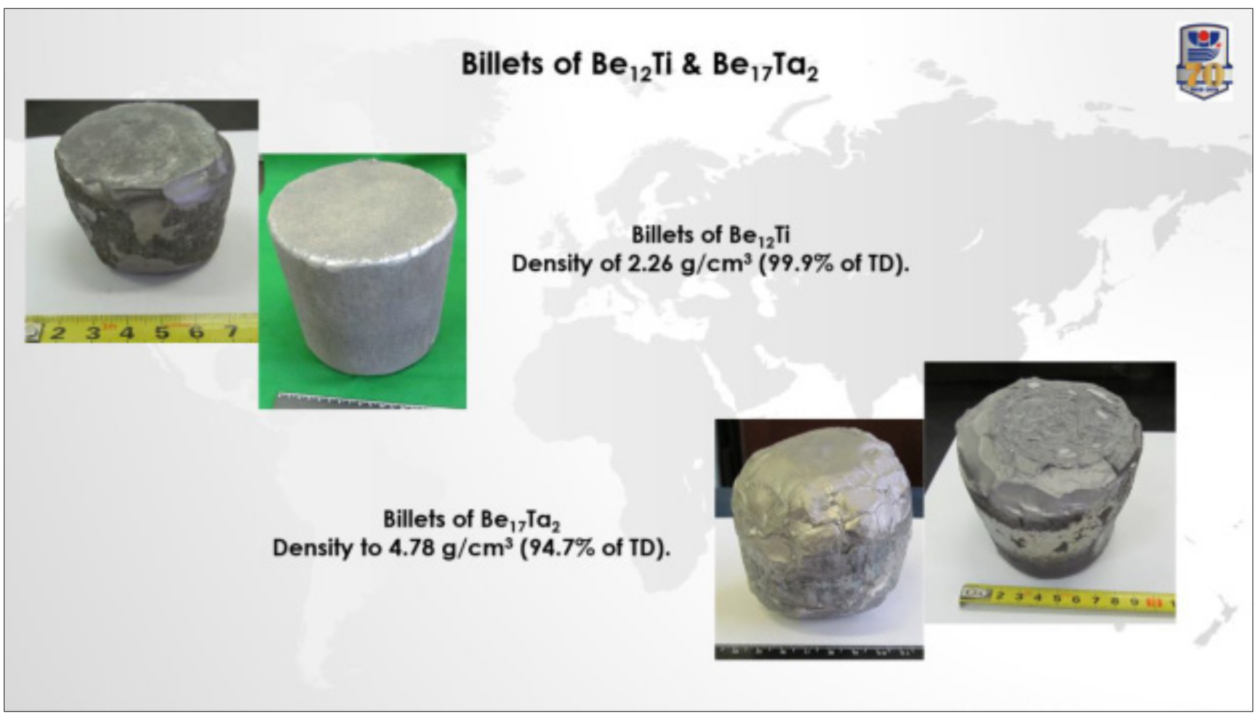
Ta powder of capacitor grade 5A – standard products

	Chemical composition, wt. %										Specific surface, m^2/g	Bulk weight, g/cm^3	After bumping-down bulk weight, g/cm^3
	Ti	Sn	Nb	Zr	Ca	W	Mo	Co	N	H			
Be	-	-	-	-	-	-	-	-	-	-	0.32	0.57	0.98
Ta	<8	<3	<30	<30	<30	<30	<1	<1	-	-	-	4.97	-
Ti	base	-	-	-	-	-	-	-	800	3500	-	1.36	1.62

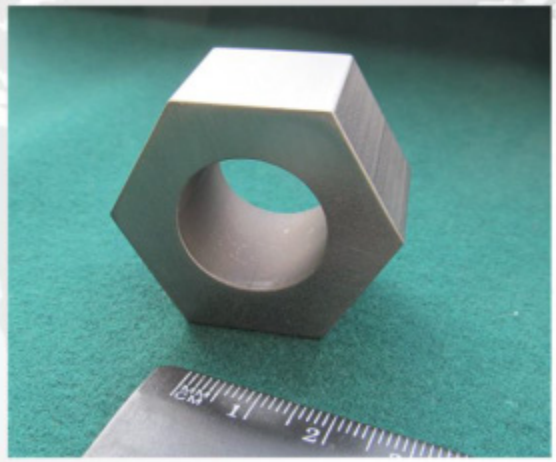

Powder metallurgy for fabricating $Be_{12}Ti$ & $Be_{17}Ta_2$
Be powder appearance.
Ta powder appearance
Ti powder appearance.


Study of $Be_{12}Ti$ synthesis
Full synthesis of $Be_{12}Ti$ compound is at 1100°C.


Synthesis of $Be_{17}Ta_2$
Powder synthesized at UMP JSC conform with compound of $Be_{17}Ta_2$
The synthesised powder
[00-018-0232] Be17Ta2 Beryllium Tantalum


Billets of $Be_{12}Ti$ & $Be_{17}Ta_2$
Billets of $Be_{12}Ti$
Density of 2.26 g/cm³ (99.9% of TD).
Billets of $Be_{17}Ta_2$
Density to 4.78 g/cm³ (94.7% of TD).

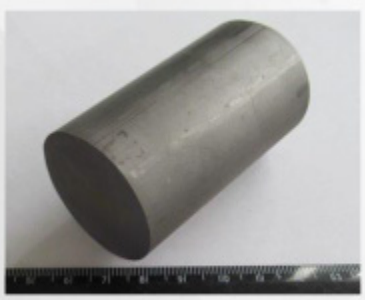
Items of $Be_{12}Ti$



Appearance of $Be_{12}Ti$ item by wire machining.

Items of $Be_{17}Ta_2$

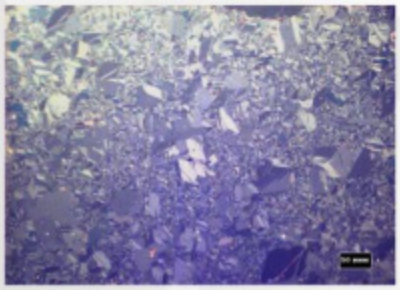
Treatment of billets is only possible by wire machining.

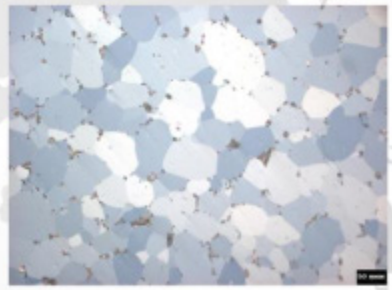


Appearance of $Be_{17}Ta_2$ billets before and after wire machining.

Microstructure and properties of $Be_{12}Ti$ & $Be_{17}Ta_2$

Microstructure of $Be_{17}Ta_2$
Average grain size 59.6 mkm
Micro hardness– 1,152.8 kg/mm²
Electric conductivity at a temperature from 2.6 to 9.5 MS/m depending on the porosity





SUMMARY

- Casting technology does not make it possible to fabricate billets of $Be_{17}Ta_2$ & $Be_{12}Ti$ applicable for items manufacture.
- Synthesis of compounds is at a temperature over than 1100°C.
- UMP JSC process makes it possible to produce $Be_{12}Ti$ items with the density of about 99.9% of TD.
- We produced billets of $Be_{17}Ta_2$ with the density of up to 94.7% of TD.
- For machining billets of $Be_{17}Ta_2$ & $Be_{12}Ti$ specific methods are required as wire machining and hydro-abrasive cutting.

Simple process for mass production of metal Be true sphere

Y. Natori, K. Yonehara, T. Kawakami, and K. Tatenuma

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In order to develop a simple mass-production technology of small true sphere of beryllium metal as neutron multiplier for nuclear fusion reactor, the method to make it directly from its powder by revolving inclined high temperature furnace was evaluated. By the reason that beryllium has a highly chemical activity in a high temperature condition, we have to take notice that beryllium metal easily reacts with different materials. For example, when using the alumina or magnesia ceramics of general material of crucible and furnace tube, beryllium easily reacts and adheres to them at a near beryllium melting point and be converted to beryllium oxide. Considering this fact, it is necessary to develop ceramic materials that are less reactive with beryllium.

In this study, for forming the chemically inactive ceramics surface against metal beryllium at a high temperature range, the coating method of beryllium oxide on alumina ceramics using a silicate binder was developed. By using the alumina tube with a thick coating of beryllium oxide formed by this method, the possibility of simple process mass-production of small true sphere of metal beryllium by revolving inclined high temperature furnace was confirmed.

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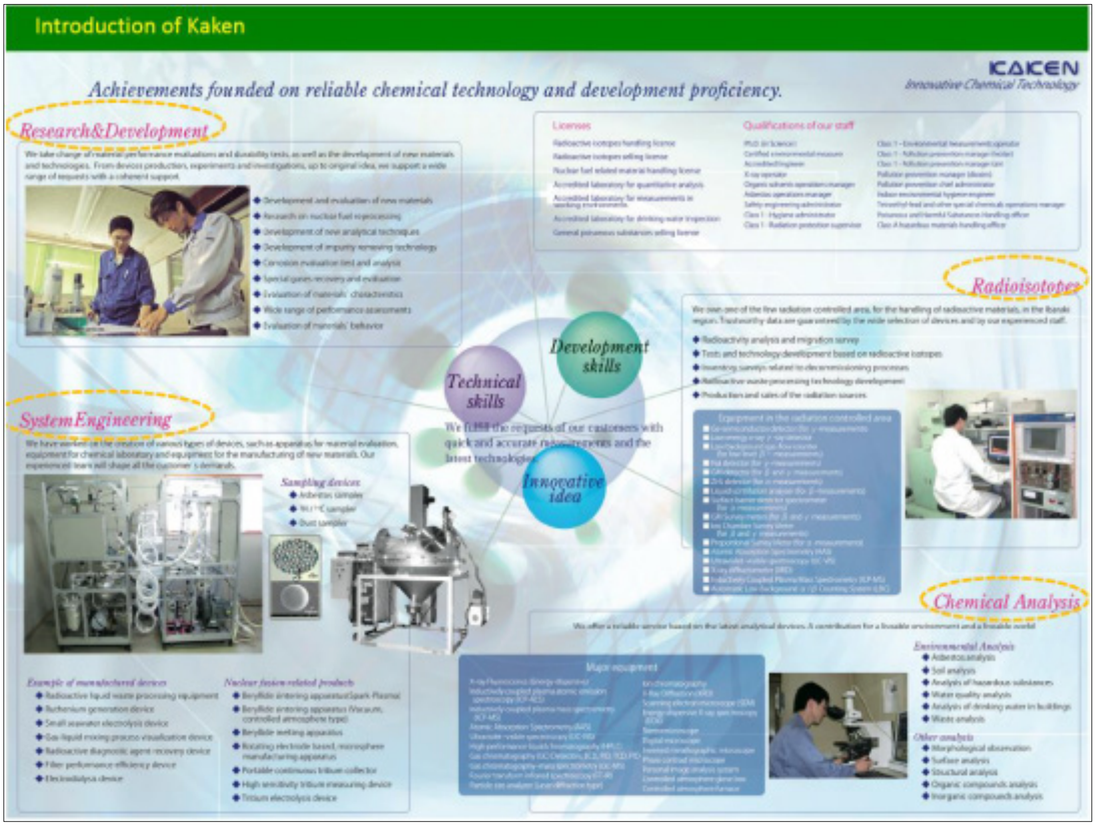
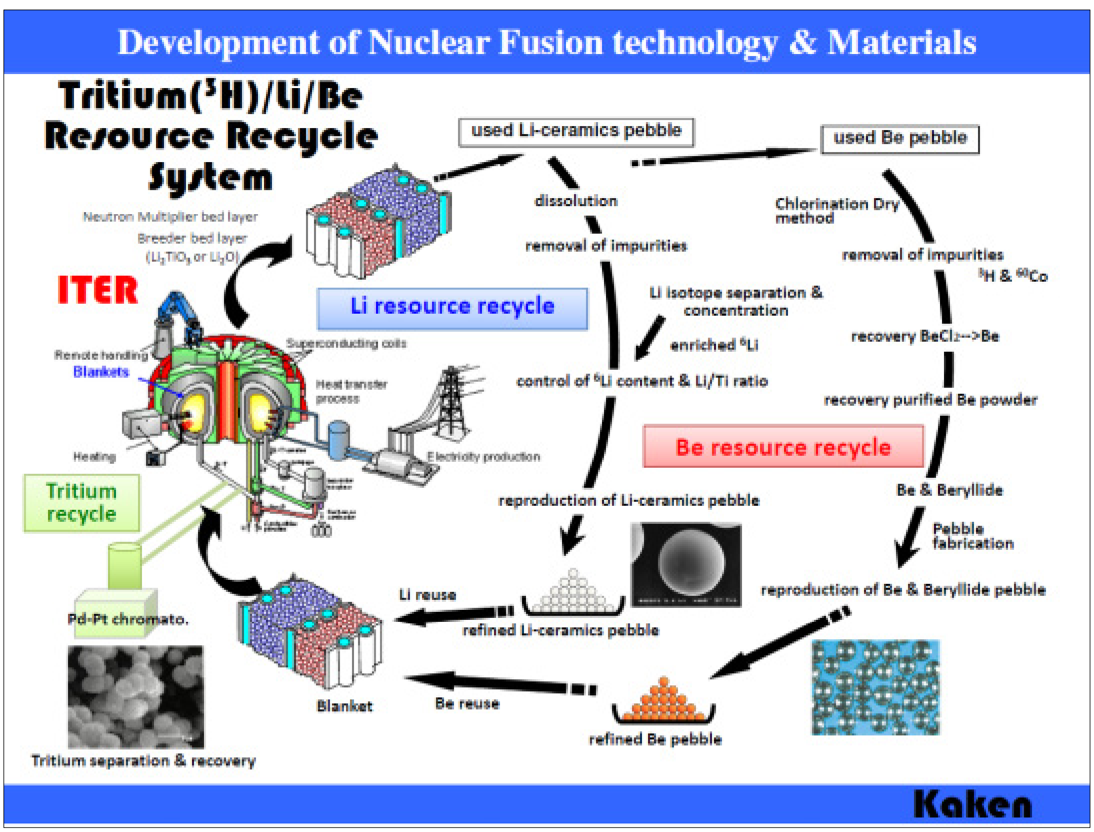

Introduction of Kaken
KAKEN
Innovative Chemical Technology
Achievements founded on reliable chemical technology and development proficiency.
Research&Development
Radioisotopes
Development skills
Technical skills
Innovative idea
SystemEngineering
Chemical Analysis


Development of Nuclear Fusion technology & Materials
Tritium(³H)/Li/Be Resource Recycle System
Neutron Multiplier bed layer
Breeder bed layer
ITER
Remote handling
Blankets
Superconducting coils
Heat transfer process
Electricity production
Heating
Tritium recycle
Pd-Pt chromato.
Tritium separation & recovery
Blanket
Li resource recycle
used Li-ceramics pebble
used Be pebble
dissolution
removal of impurities
Li isotope separation & concentration
enriched ⁶Li
control of ⁶Li content & Li/Ti ratio
reproduction of Li-ceramics pebble
Li reuse
refined Li-ceramics pebble
Chlorination Dry method
removal of impurities
³H & ⁶⁰Co
recovery BeCl₂-->Be
recovery purified Be powder
Be resource recycle
Be & Beryllide
Pebble fabrication
reproduction of Be & Beryllide pebble
Be reuse
refined Be pebble
Kaken

14th International Workshop on Beryllium Technology
Oct. 24-25, 2019, Long Beach, California, USA

KAKEN

Simple process for mass production of metal Be true sphere

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Concept

In order to develop the simple mass-production technology of small true sphere of beryllium metal as neutron multiplier for nuclear fusion reactor, the method to make it directly from its powder by revolving inclined high temperature furnace was evaluated. By the reason that beryllium has a highly chemical activity in a high temperature condition, we have to take notice that beryllium metal easily reacts with different materials. For example, when using an alumina or magnesia ceramics of general material of crucible and furnace tube, beryllium easily reacts and adheres to them at a near beryllium melting point and be converted to beryllium oxide. Considering this fact, it is necessary to develop ceramic materials that are less reactive with beryllium.

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Purpose

Based on an ordinary method for making metal sphere, we confirmed the possibility for mass-producing beryllium sphere with a simple method and process, a high efficiency, a low cost and a small production loss.

Outline

In this study, for forming the chemically inactive ceramics surface against metal beryllium at a high temperature range, the coating method of beryllium oxide on alumina ceramics using a silicate binder was developed. Using the alumina tube with a thick coating of beryllium oxide formed by this method, the possibility of simple process mass-production of small true sphere of metal beryllium by revolving inclined high temperature furnace was confirmed.

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Mass-production of beryllium sphere -1

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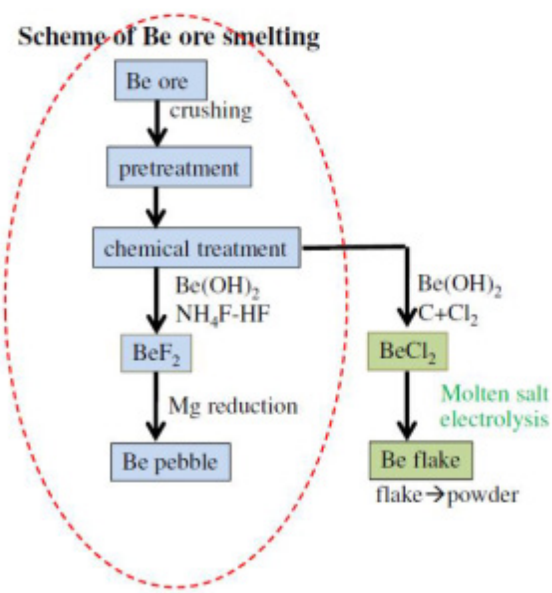
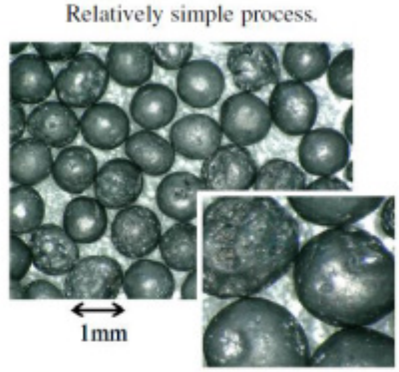
Mg reduction method

Current method for producing Be crude raw metal



Relatively simple process.



Contain impurities and internal void

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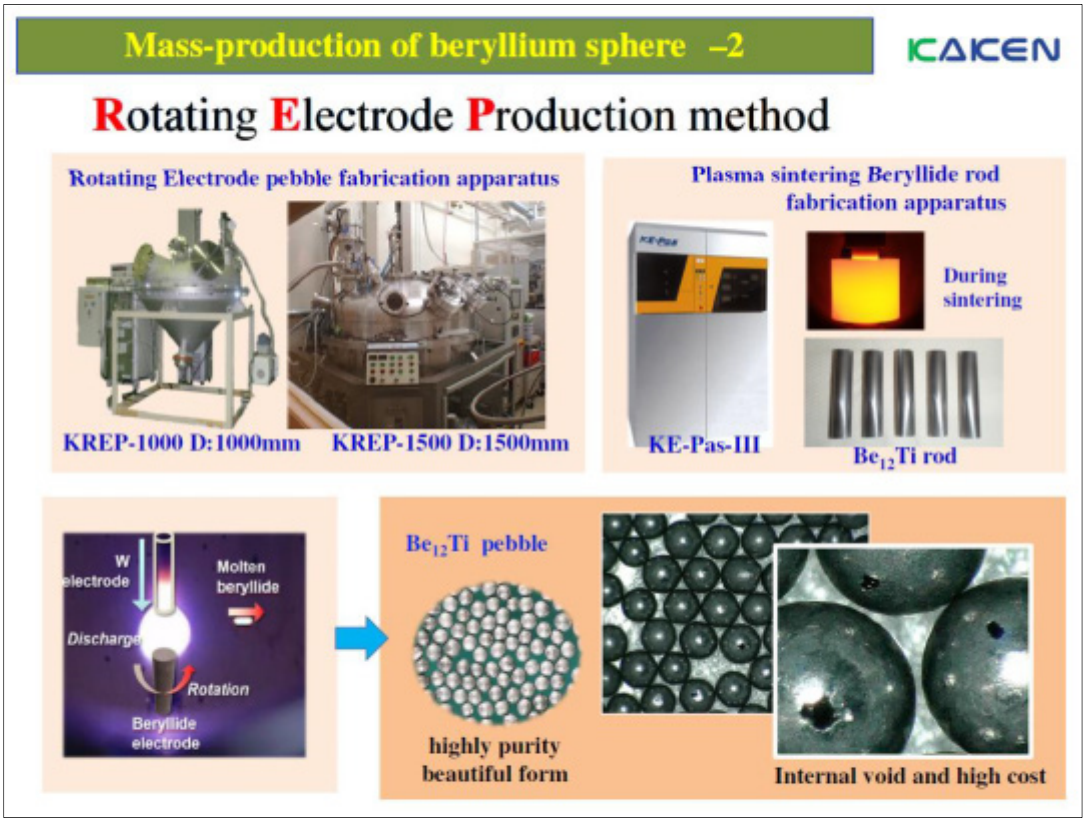
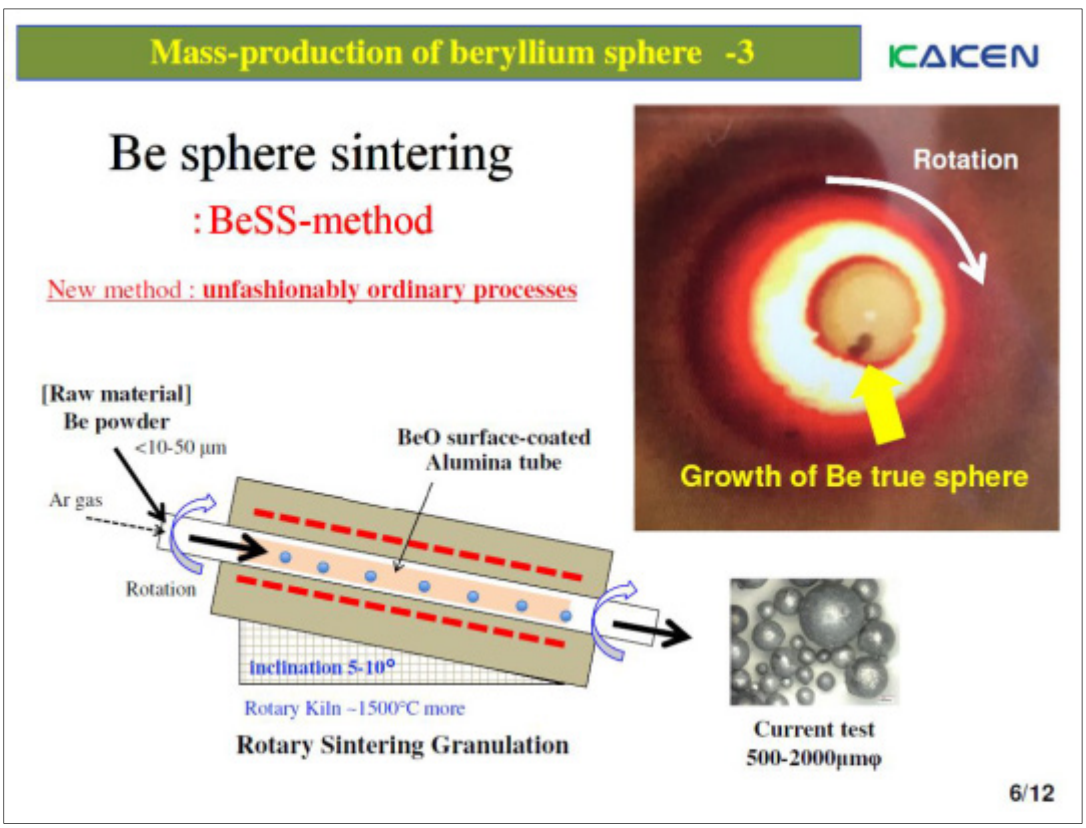

Mass-production of beryllium sphere –2
KAKEN
Rotating Electrode Production method
Rotating Electrode pebble fabrication apparatus
KREP-1000 D:1000mm
KREP-1500 D:1500mm
Plasma sintering Beryllide rod fabrication apparatus
During sintering
KE-Pas-III
$Be_{12}Ti$ rod
W electrode
Molten beryllide
Discharge
Rotation
Beryllide electrode
$Be_{12}Ti$ pebble
highly purity
beautiful form
Internal void and high cost


Mass-production of beryllium sphere -3
KAKEN
Be sphere sintering
: BeSS-method
New method : unfashionably ordinary processes
Rotation
Growth of Be true sphere
[Raw material]
Be powder
<10-50 μm
Ar gas
BeO surface-coated
Alumina tube
Rotation
Inclination 5-10°
Rotary Kiln ~1500°C more
Rotary Sintering Granulation
Current test
500-2000μmφ
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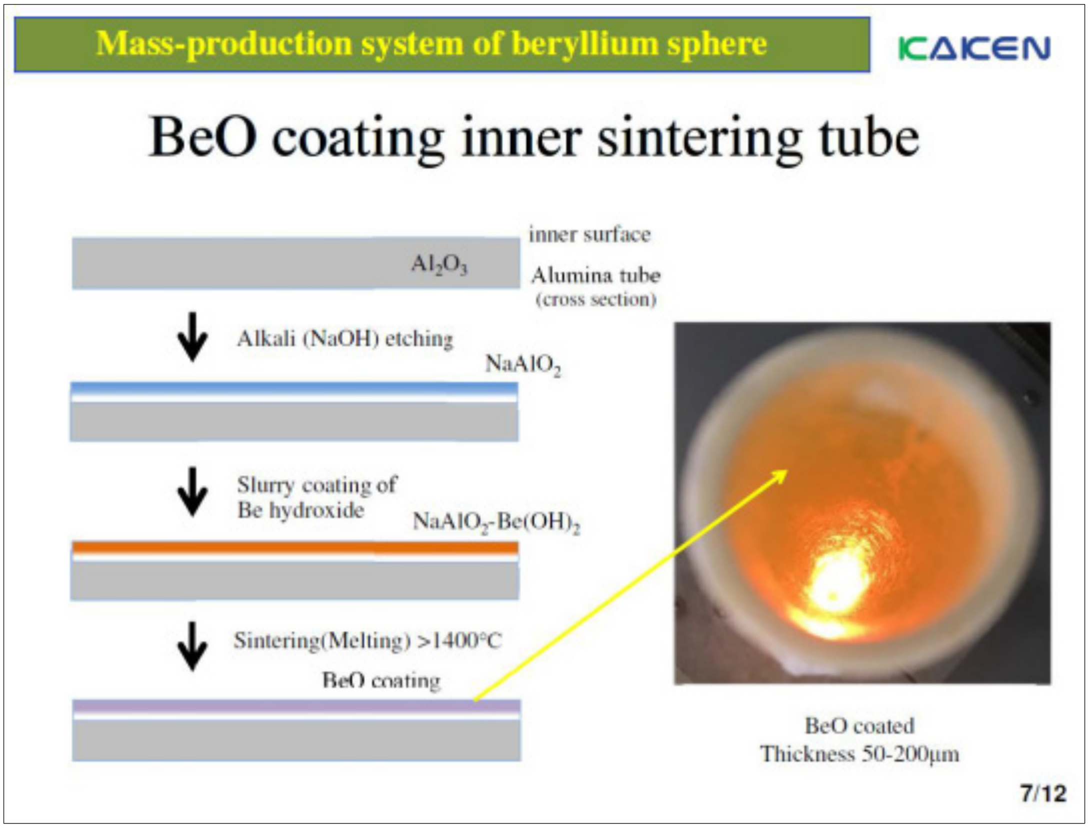
Mass-production system of beryllium sphere
KAKEN
BeO coating inner sintering tube
inner surface
Al_2O_3
Alumina tube
(cross section)
Alkali (NaOH) etching
$NaAlO_2$
Slurry coating of
Be hydroxide
$NaAlO_2$-$Be(OH)_2$
Sintering(Melting) >1400°C
BeO coating
BeO coated
Thickness 50-200μm
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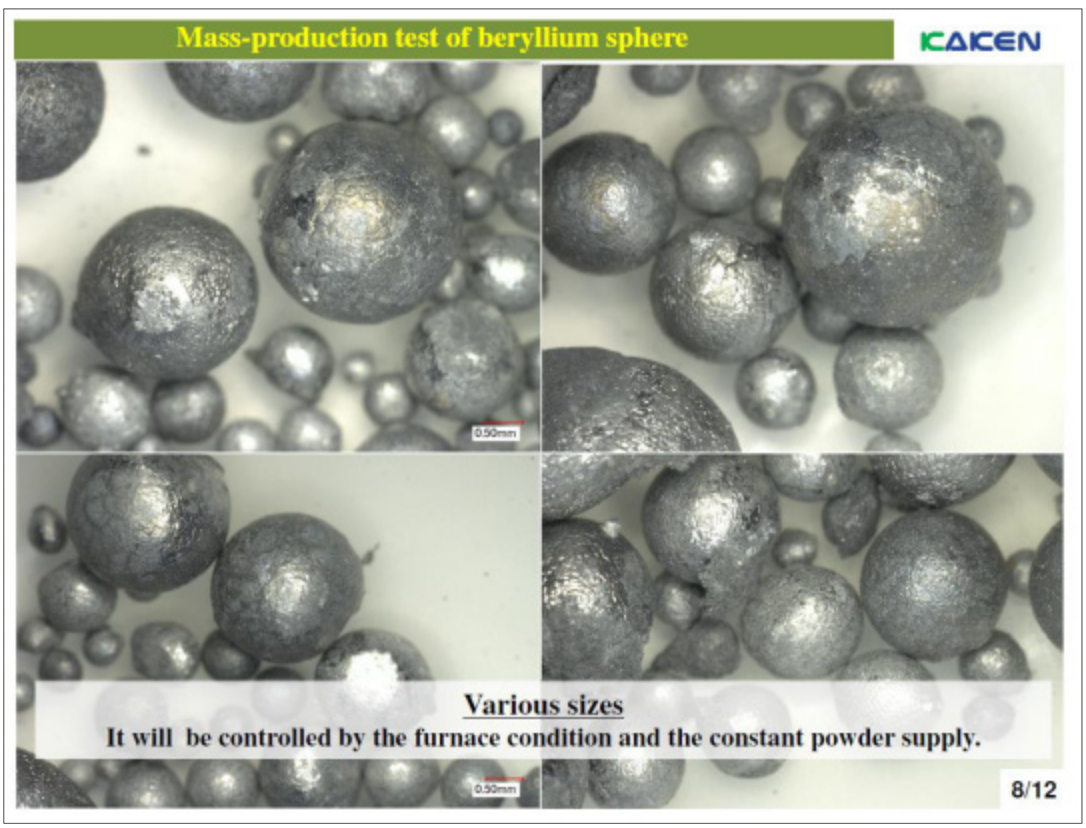
Mass-production test of beryllium sphere
KAKEN
Various sizes
It will be controlled by the furnace condition and the constant powder supply.
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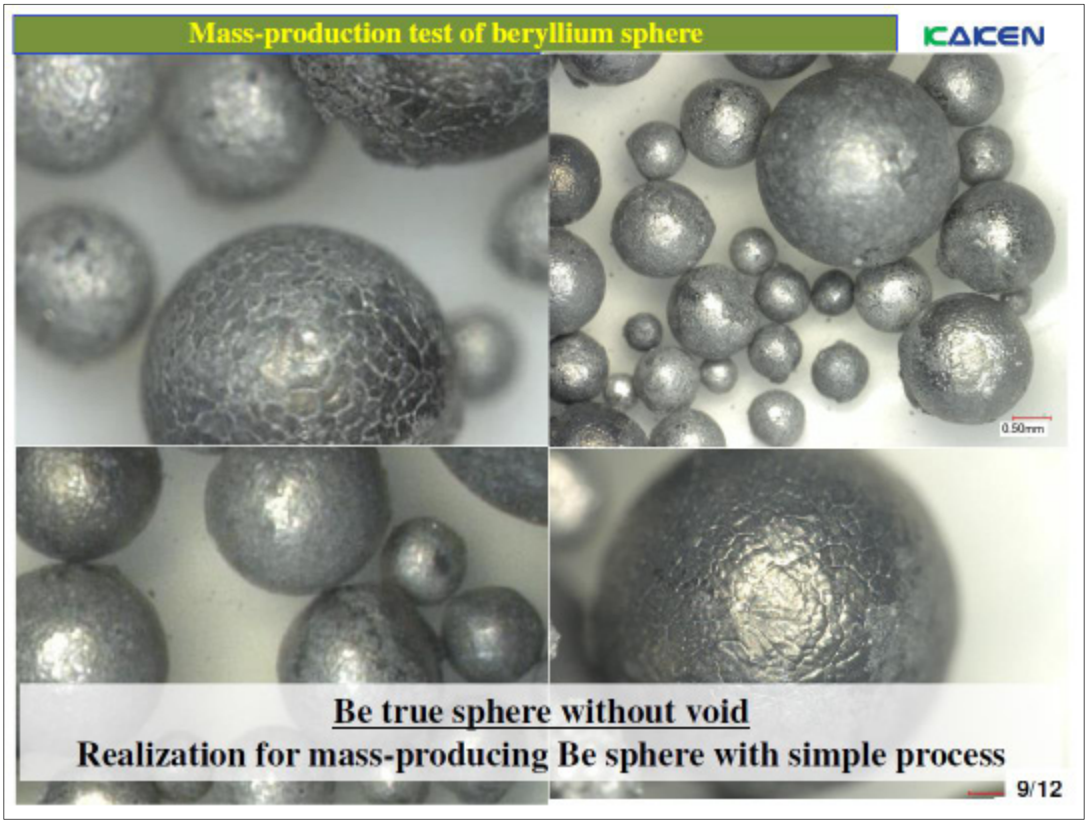
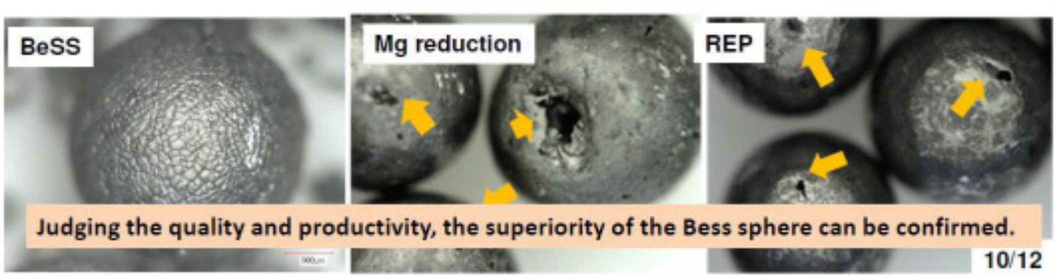


Mass-production test of beryllium sphere KAKEN

Comparison ; Be sphere quality

Production method	BeSS	Mg reduction	REP
Surface shape	Choppy	Rough	Smooth
Spherical degree	> 90 %	70-85 %	> 95 %
Density (g/cc) %TD	1.85 >98-100%TD (Theoretical density)	1.75-1.79 ab. 95-97%TD	1.70-1.78 ab. 92-96%TD
Chemical quality	depend on raw Be powder	?	depend on raw Be powder
Yield (production loss)	>95% (Lossless)	-	<70% (Loss >30%)
Production Process & Controllability	Simple	-	Difficult
Production time	Short <10-30 min	-	Long >1-3 days
Mass-productivity	Easy	-	Difficult
Production Cost	inexpensive	little expensive	expensive



Mass-production system of beryllium sphere

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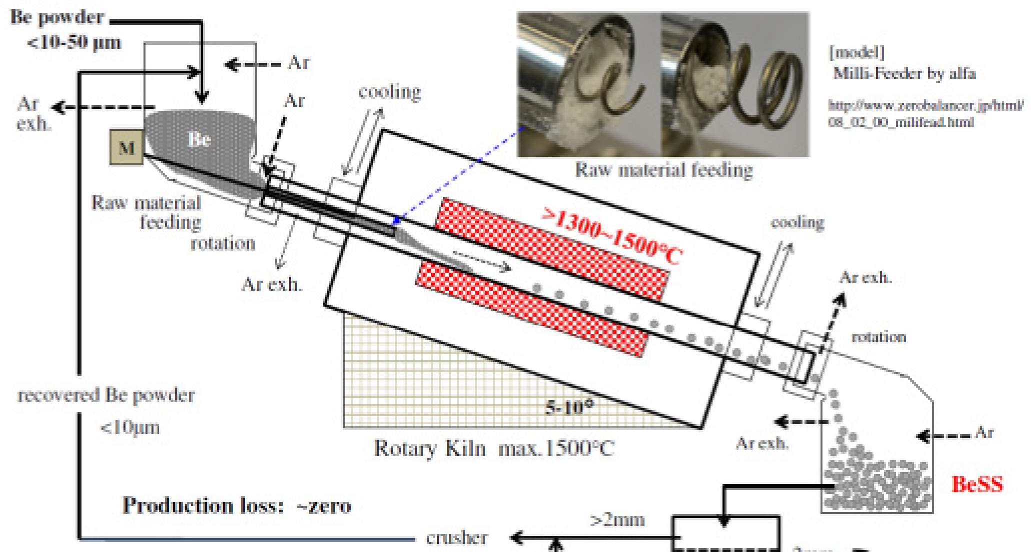

Feature

- **Continuous and large scale production**
- **Simple production process and system**
- **Controllable pebble size**
- **Small or zero loss of raw Be powder**

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Mass-production of beryllium sphere

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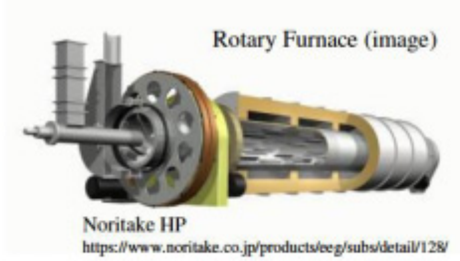
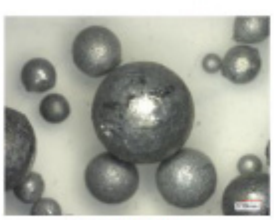
Summary

It is possible to produce beryllium true sphere using slanted rotary furnace with ceramic tube inner-coated by beryllium oxide. By this simple and efficient method, continuous and large scale mass-production of beryllium sphere is possible with a low cost and high density.

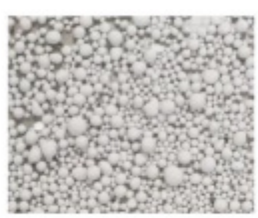


Future Prospect

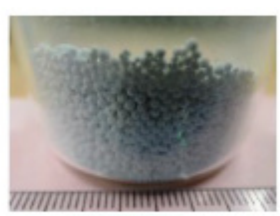
By the similar method, we are also developing the mass-production of tritium breeding material (Li_2TiO_3, Li_2O) pebble, and there is a possibility of mass-productivity of beryllide pebble by a little modification with some chemical treatment.
We will be able to produce and supply of both of neutron multiplier (Be) sphere and tritium breeder (Li compound) sphere with a low cost in the near future.



Be pebble



Li_2TiO_3 pebble



Li_2O pebble

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Session 5: Modeling

Hydrogen Coverage Regimes on (0001) Be Surfaces

C. Stihl (KIT, Germany) et al.

Hydrogen coverage regimes on (0001) Be surfaces

Christopher Stihl and Pavel V. Vladimirov

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Beryllium is a proposed neutron-multiplier material in future fusion devices like ITER and DEMO. During operation of such devices, considerable accumulation of helium and, most importantly, radioactive tritium is expected in beryllium due to neutron-induced transmutation. As large tritium inventories are considered a safety issue for fusion applications, substantial experimental efforts have been made to investigate microstructural features serving as tritium traps in neutron-irradiated beryllium.

At elevated temperatures and high neutron fluences, the most prominent change of microstructure is the emergence of gas bubbles filled with helium and tritium. Bubbles inside grains typically form oblate prisms with hexagonal bases lying on (0001) beryllium surfaces and covered by a considerable amount of tritium. As these bubbles are effective traps for dissolved tritium, they exert decisive influence on the effective tritium retention and release behavior of beryllium. Thus, a reliable model capable of predicting tritium inventory as well as retention and release behavior even under accidental conditions will have to comprise an accurate understanding of processes involving tritium at such bubble surfaces. Many further experiments aiming to find atomic processes that collectively govern the rise of such phenomena were done. Typically, exposure to a hydrogen isotope gas or implantation of hydrogen isotopes are typical measures to introduce controlled amounts of hydrogen isotopes into the sample.

This work is dedicated to the understanding of such experiments by means of atomic processes modelled based on ab-initio methods. In that regard, two hydrogen coverage regions below and above ratios of ^{1}H/Be are established at the surface. Coverages below 1 H/Be can be considered a low-coverage regime. Within this regime, the dynamics of hydrogen isotopes on the surface can be considered by lattice gas models as they are also well established in the scope of surface catalysis. In particular, kinetic lattice Monte Carlo methods in conjunction with energy estimation schemes like cluster expansions and appropriately constructed rate equations allow for the simulation of desorption experiments after exposure to hydrogen isotope gases. Such experiments are found to be closely associated with this low hydrogen coverage regime due to self-limiting adsorption processes even in the case of exposure to atomic hydrogen. The high coverage regime beyond 1 He/Be, in contrast, is closely related to hydrogen isotope implantation. As there are no self-limiting processes in these circumstances, hydrogen coverages >1 H/Be can easily be attained, given the total implantation fluence is sufficient to saturate trap sites within the bulk of the beryllium samples. We found that under these circumstances, hydrogen diffusing towards a sufficiently pre-covered surface readily engages in reconstruction processes of the surface leading to the emergence of tetrahedral building blocks of BeH_2 attached to the surface.

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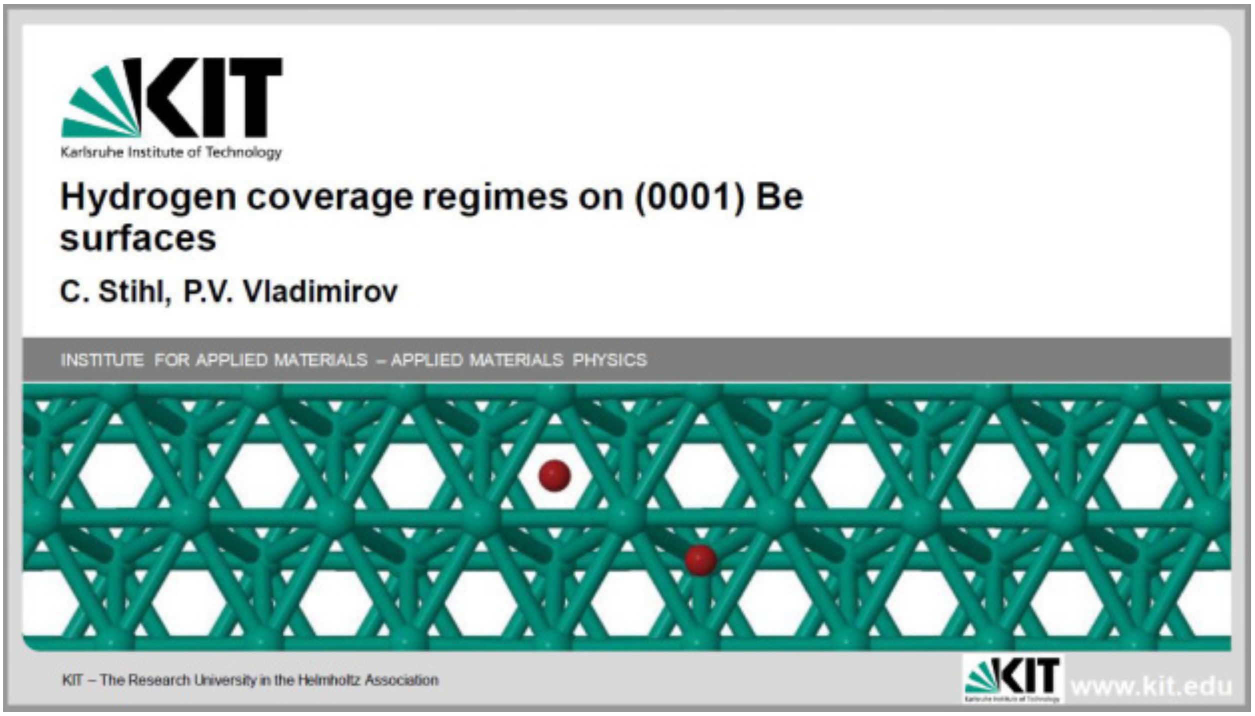
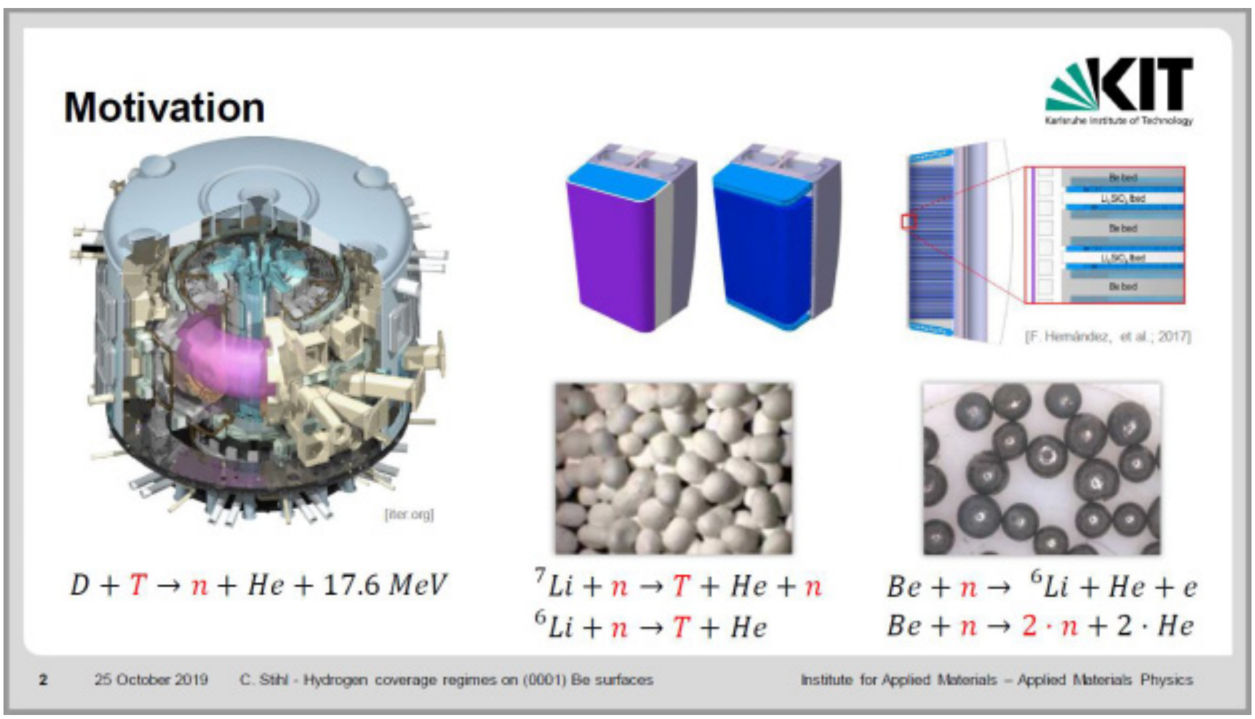
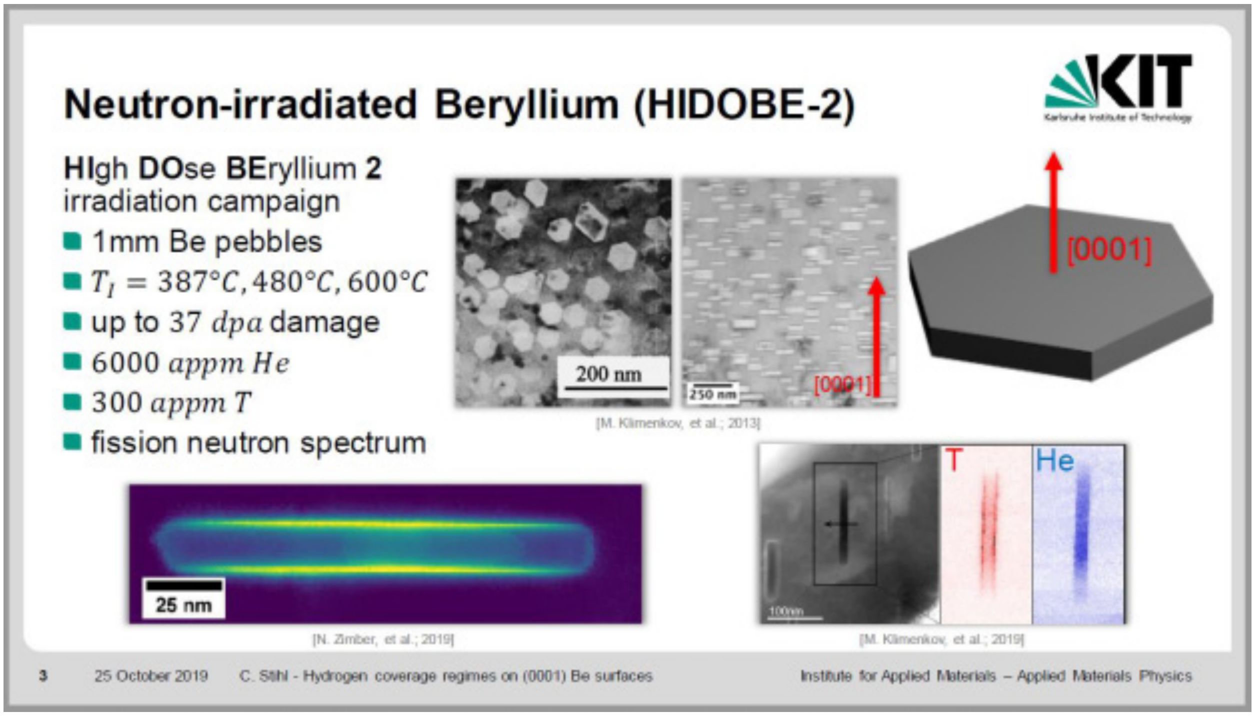
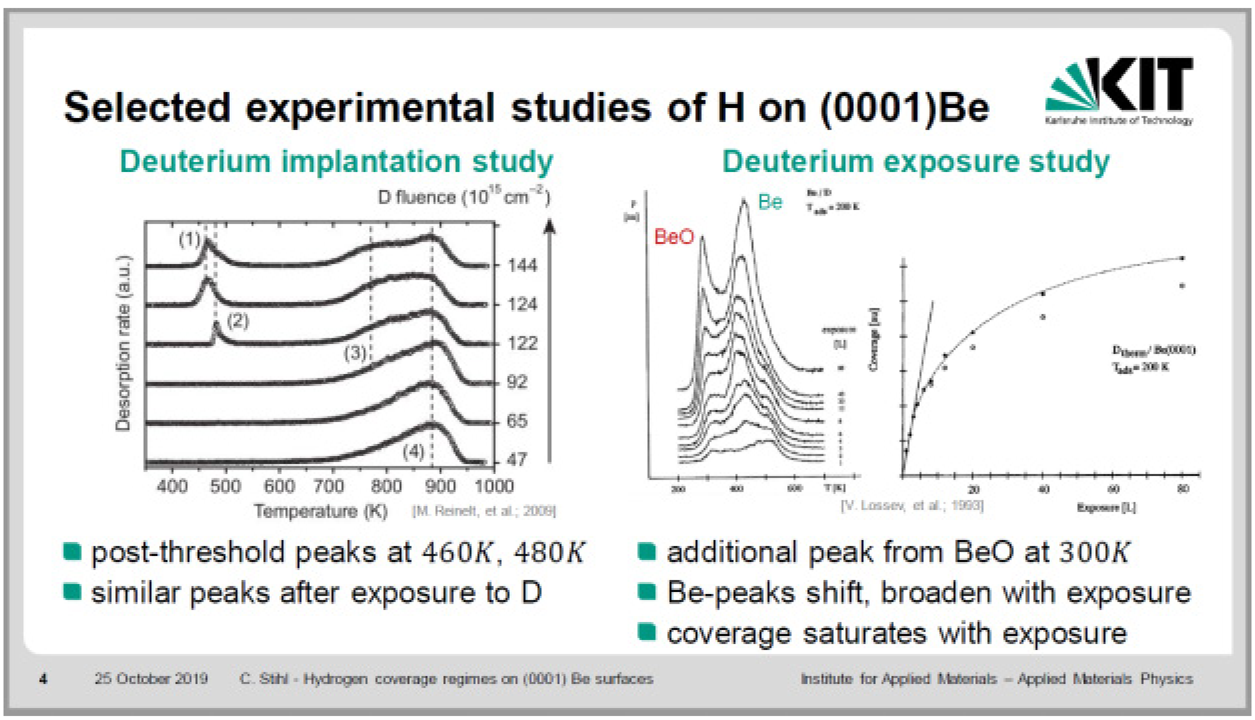
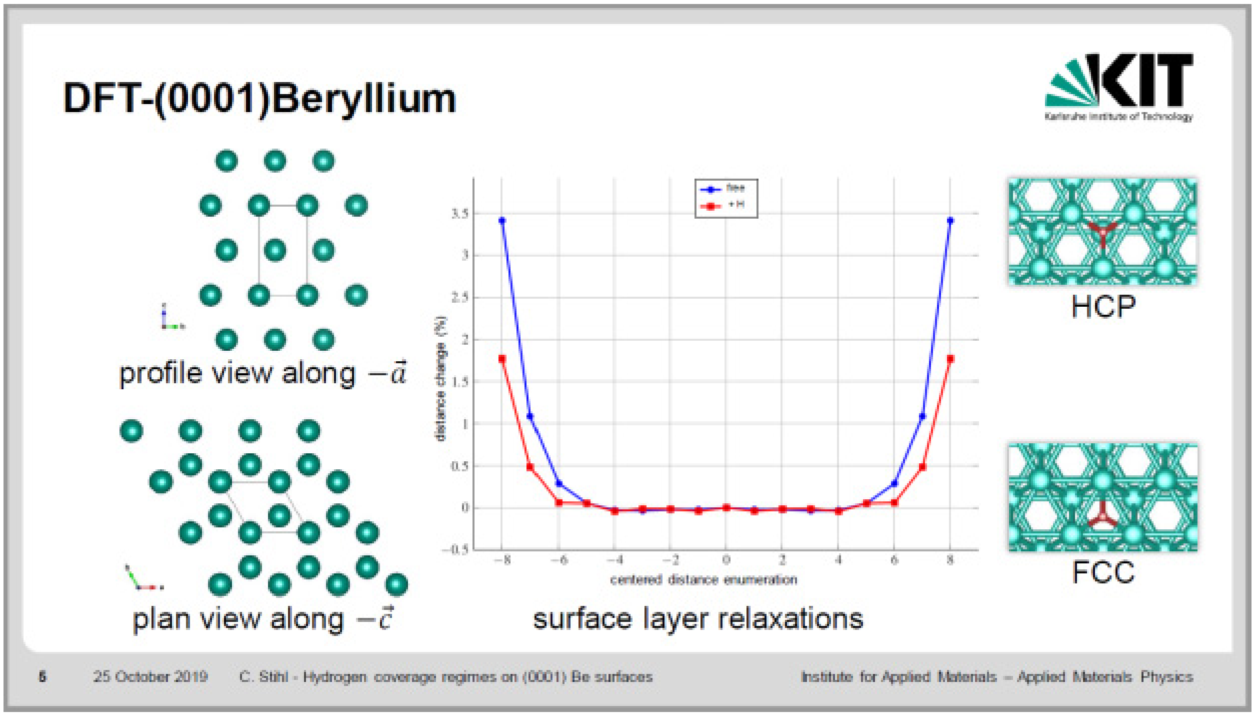

KIT
Karlsruhe Institute of Technology
Hydrogen coverage regimes on (0001) Be surfaces
C. Stihl, P.V. Vladimirov
INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS
KIT – The Research University in the Helmholtz Association
www.kit.edu


Motivation
KIT
Karlsruhe Institute of Technology
[iter.org]
[F. Hernández, et al.; 2017]
$D + T \rightarrow n + He + 17.6\ MeV$
$^{7}Li + n \rightarrow T + He + n$
$^{6}Li + n \rightarrow T + He$
$Be + n \rightarrow {}^{6}Li + He + e$
$Be + n \rightarrow 2 \cdot n + 2 \cdot He$
2
25 October 2019
C. Stihl - Hydrogen coverage regimes on (0001) Be surfaces
Institute for Applied Materials – Applied Materials Physics


Neutron-irradiated Beryllium (HIDOBE-2)
KIT
Karlsruhe Institute of Technology
HIgh DOse BEryllium 2 irradiation campaign
1mm Be pebbles
$T_I = 387°C, 480°C, 600°C$
up to 37 dpa damage
6000 $appm\ He$
300 $appm\ T$
fission neutron spectrum
200 nm
250 nm
[0001]
[0001]
[M. Klimenkov, et al.; 2013]
25 nm
[N. Zimber, et al.; 2019]
T
He
100nm
[M. Klimenkov, et al.; 2019]
3
25 October 2019
C. Stihl - Hydrogen coverage regimes on (0001) Be surfaces
Institute for Applied Materials – Applied Materials Physics


Selected experimental studies of H on (0001)Be
KIT
Karlsruhe Institute of Technology
Deuterium implantation study
D fluence (10^{15} cm^{-2})
Desorption rate (a.u.)
(1)
(2)
(3)
(4)
144
124
122
92
65
47
400 500 600 700 800 900 1000
Temperature (K)
[M. Reinelt, et al.; 2009]
Deuterium exposure study
BeO
Be
[V. Lossev, et al.; 1993]
Exposure [L]
Coverage [au]
post-threshold peaks at $460K$, $480K$
similar peaks after exposure to D
additional peak from BeO at $300K$
Be-peaks shift, broaden with exposure
coverage saturates with exposure
4
25 October 2019
C. Stihl - Hydrogen coverage regimes on (0001) Be surfaces
Institute for Applied Materials – Applied Materials Physics


DFT-(0001)Beryllium
KIT
Karlsruhe Institute of Technology
profile view along $-\vec{a}$
plan view along $-\vec{c}$
distance change (%)
centered distance enumeration
surface layer relaxations
HCP
FCC
5
25 October 2019
C. Stihl - Hydrogen coverage regimes on (0001) Be surfaces
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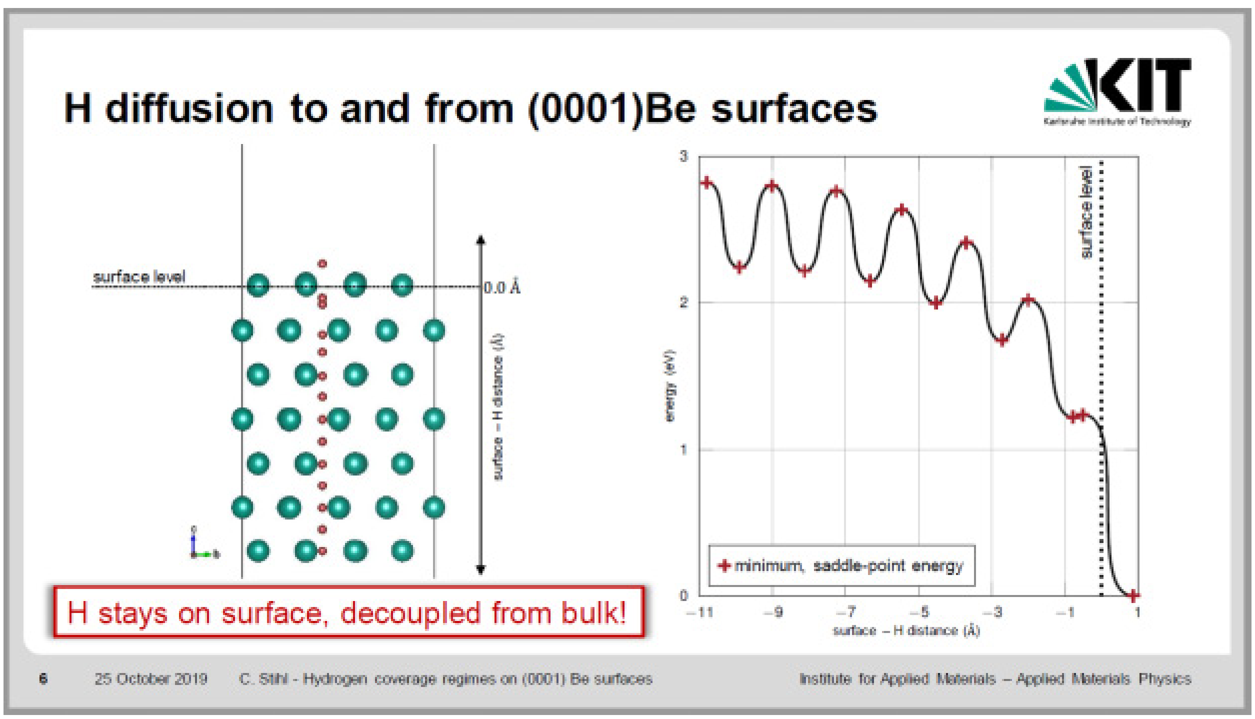
H diffusion to and from (0001)Be surfaces
KIT
Karlsruhe Institute of Technology
surface level
0.0 Å
surface – H distance (Å)
H stays on surface, decoupled from bulk!
energy (eV)
surface level
minimum, saddle-point energy
surface – H distance (Å)
6
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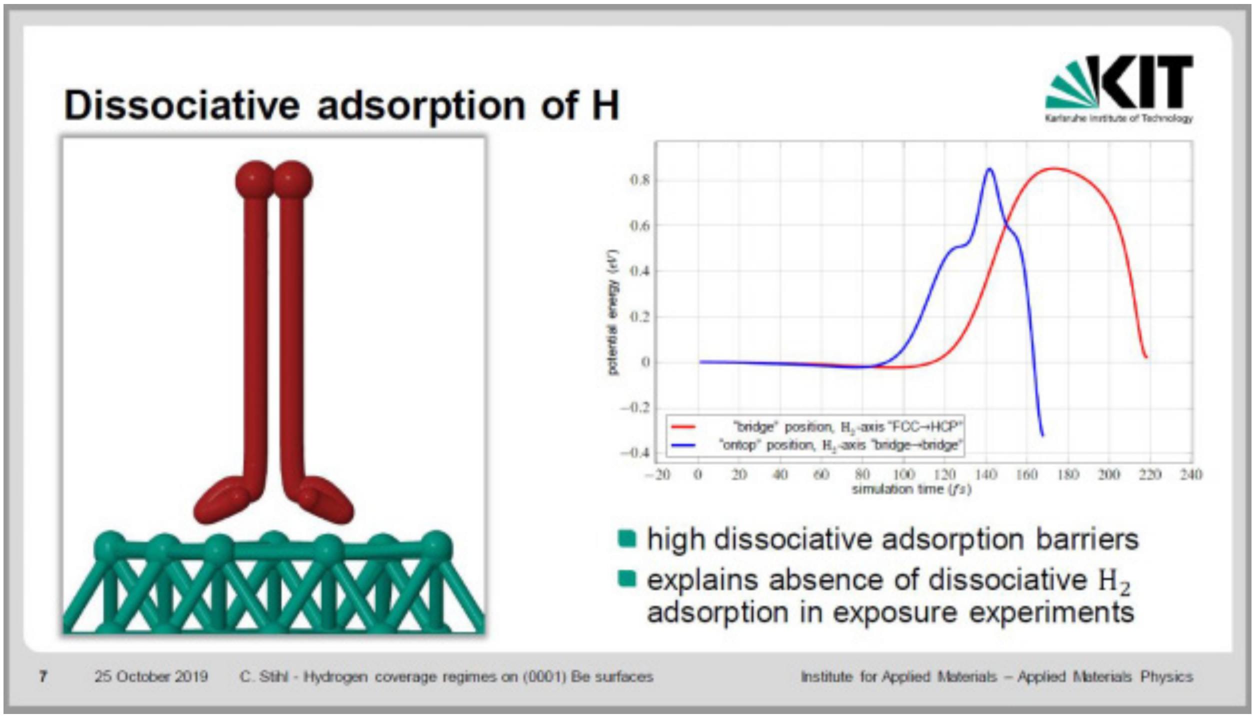
Dissociative adsorption of H
KIT
Karlsruhe Institute of Technology
potential energy (eV)
simulation time (fs)
"bridge" position, H$_2$-axis "FCC→HCP"
"ontop" position, H$_2$-axis "bridge→bridge"
high dissociative adsorption barriers
explains absence of dissociative H$_2$ adsorption in exposure experiments
7
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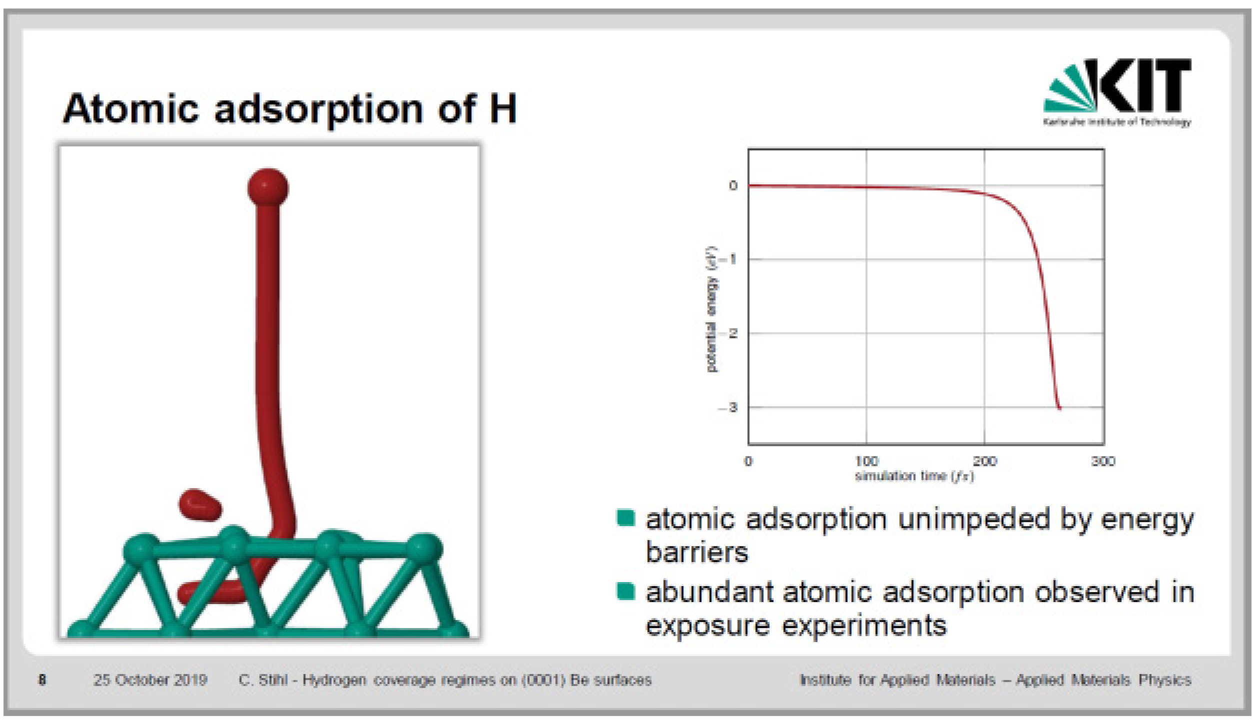
Atomic adsorption of H
KIT
Karlsruhe Institute of Technology
potential energy (eV)
simulation time (fs)
atomic adsorption unimpeded by energy barriers
abundant atomic adsorption observed in exposure experiments
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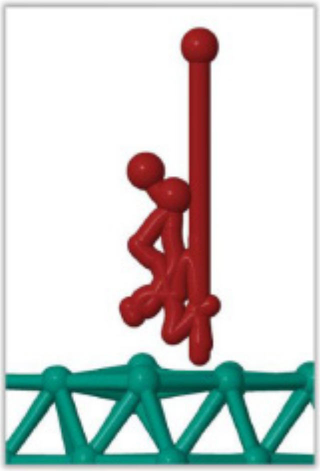
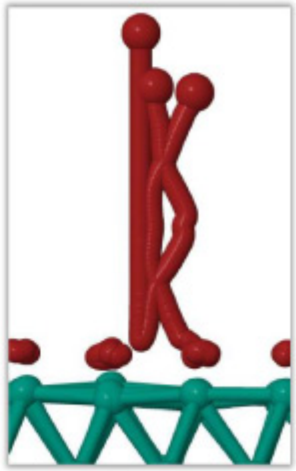
Effective desorption of H





- H impinging at occupied adsorption site induces associative desorption
- H impinging in vicinity of adsorbed H induces associative desorption
- inherent coverage limitation even with atomic exposure

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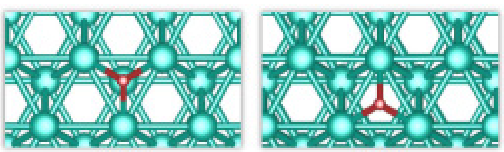
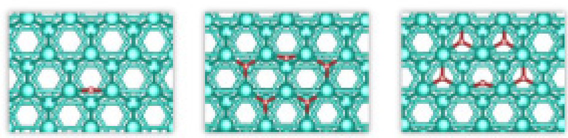
Capturing surface dynamics

- effectively incorporate surface dynamics on adsorption site lattice



- at given coverage, adsorbate layer evolves by surface diffusion hops between directly adjacent adsorption sites



PROCESS	FCC→HCP	HCP→FCC
free	0.18 eV	0.21 eV
add. HCP	0.31 eV	0.07 eV
add. FCC	0.03 eV	0.28 eV

- fast surface diffusion quickly disperses H accumulations due to *repulsion*

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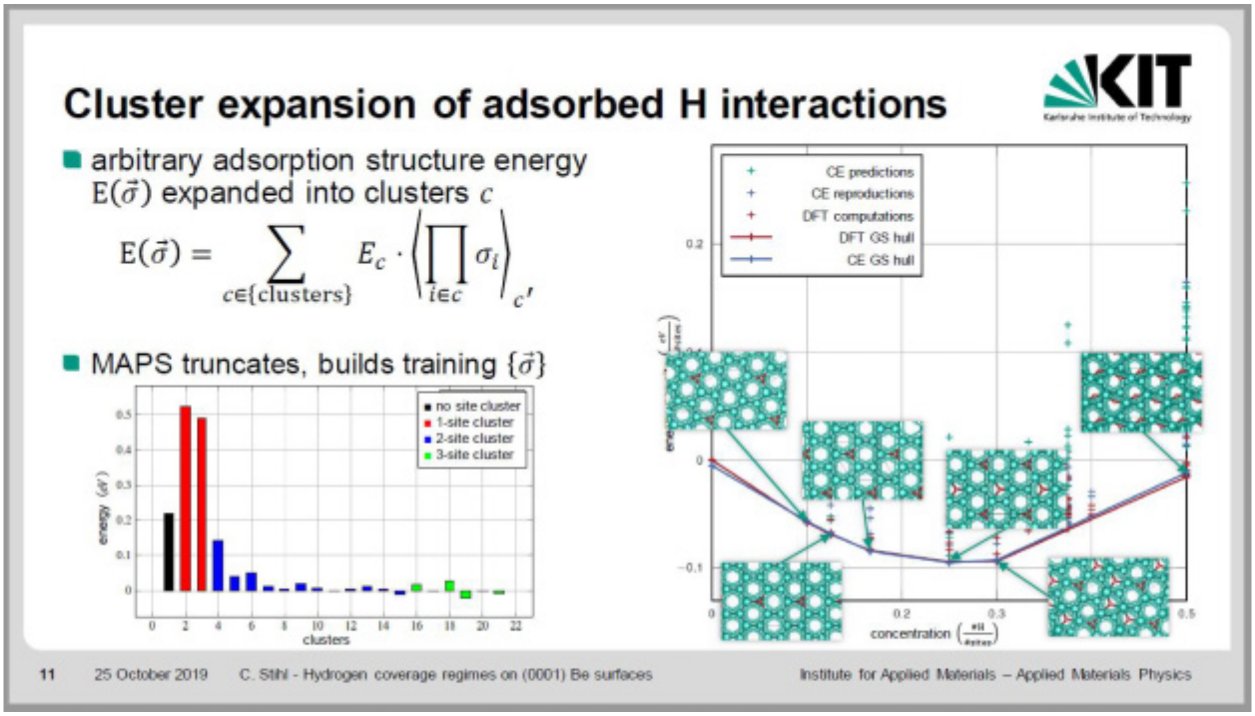
Cluster expansion of adsorbed H interactions
arbitrary adsorption structure energy $E(\vec{\sigma})$ expanded into clusters c
$E(\vec{\sigma}) = \sum_{c \in \{clusters\}} E_c \cdot \left\langle \prod_{i \in c} \sigma_i \right\rangle_{c'}$
MAPS truncates, builds training $\{\vec{\sigma}\}$
no site cluster
1-site cluster
2-site cluster
3-site cluster
energy (eV)
clusters
CE predictions
CE reproductions
DFT computations
DFT GS hull
CE GS hull
concentration
11
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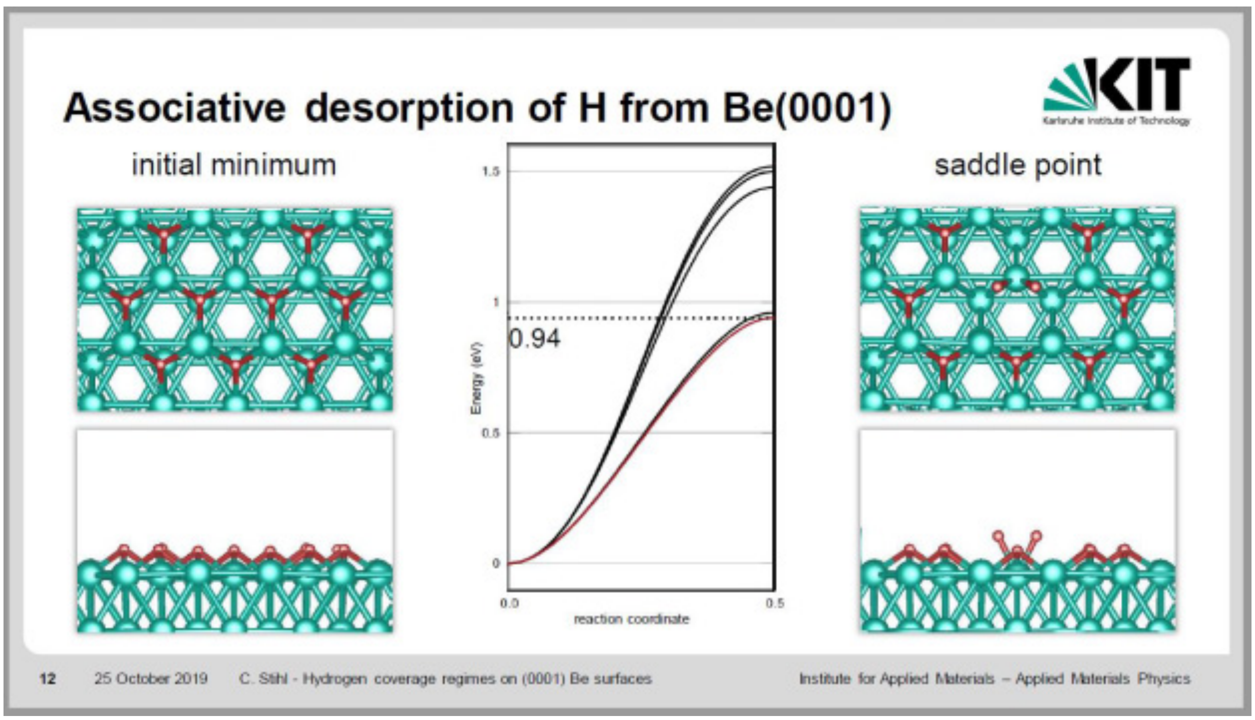
Associative desorption of H from Be(0001)
initial minimum
saddle point
0.94
Energy (eV)
reaction coordinate
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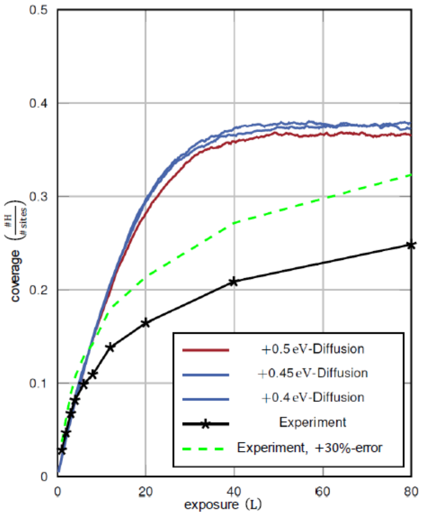
KMC simulation of exposure experiments



- diffusion slowed down, enabling simultaneous simulation of slower ad- and desorption
- little impact of large slowdown differences indicate diffusion still fast enough
- linear coverage uptake during small exposures properly reproduced
- discrepancy most likely from additional desorption processes incorporating other features of the surface, e.g. oxygen or kinks
- coverage saturation from desorption events induced by atomic impingement only
- exposure experiments thus inherently associated with low to intermediate coverages

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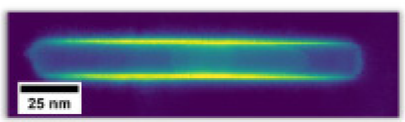
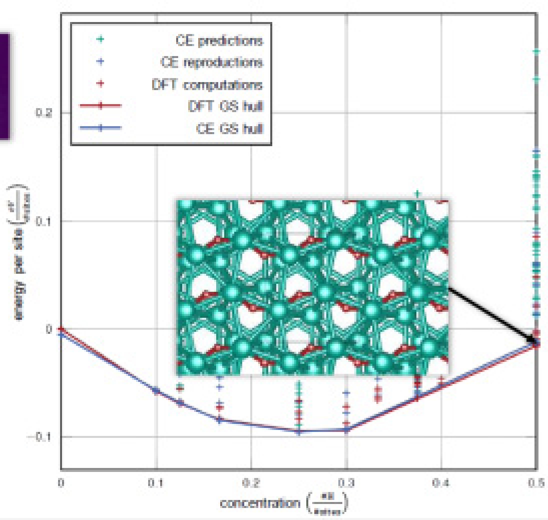
Reconsidering (0001)Be as bubble surfaces



[N. Zimber, et al., 2019]



- oxygen rare and thus most likely not decisive for dynamics at bubble surface in contrast to exposure experiments
- surface coverage limitations inherent to exposure experiments do not apply as T produced in bulk Be.
- surface dynamics of bubble surfaces thus likely in different coverage regime than exposure experiments

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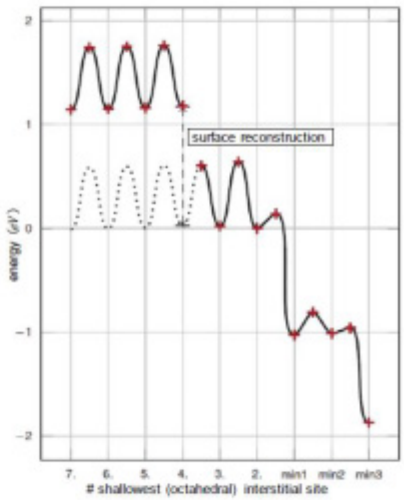
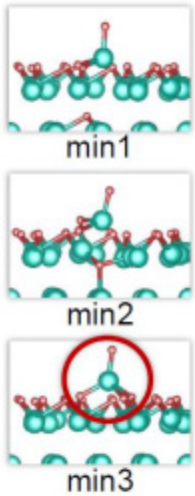
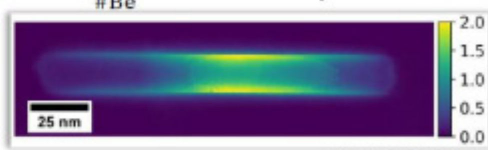
(0001)Be at high coverages





- tetrahedral H-Be building blocks of BeH_2 emerged on surface
- after H diffusion from bulk to surface with $c = 0.5\frac{\#H}{\#sites} = 1\frac{\#H}{\#Be}$
- $1\frac{\#H}{\#Be} < c \leq 2\frac{\#H}{\#Be}$ at surface of bubbles in experiment?

$\frac{\#H}{\#Be}$ from EELS spectra



[N. Zimber, et al.; 2019]

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Conclusions

- exposure experiments closely associated with low surface coverages due to inherently self-limiting adsorption processes
- bubble surface concentrations not subject to this limitation as T is perpetually produced within material
- dynamics at bubble surface most likely in high coverage regimes
- oxygen mostly absent at bubble surfaces, thus not decisive for dynamics
- models may miss results of exposure experiments while being well suited for bubble surfaces

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Beryllium Lifetime Analysis for Research and Test Reactors

Gary L. Solbrekken[1], Cezary Bojanowski[2], Zachary Pfitzner[1], and Nickie Peters[3]

[1]*Mechanical and Aerospace Engineering, University of Missouri*
[2]*Nuclear Engineering Division, Argonne National Laboratory*
[3]*University of Missouri Research Reactor (MURR)*

Beryllium is a highly effective neutron-reflecting structure that is commonly used in research and test reactors. In the case of the reflecting ring at the University of Missouri Research Reactor (MURR), an age-caused fracture could impact the operation of the control blades and the safe operation of the reactor. Moreover, it is a costly item that requires a multi-year lead-time to finance. Reflecting structures in other reactors have a similar concern. Therefore, it is important to forecast the expected life of beryllium structures to extract as much useful life, yet not degrade to the point where the component fails mechanically due to radiation damage. This work will describe the process that is being developed to estimate the expected life of beryllium components subjected to operational radiation in research and test reactors. There is a single reflector fracture in MURR's history that is serving as a single reference point to evaluate the efficacy of the technique.

Monte Carlo simulations are built for the reactor core and the surrounding reflecting, coolant, and irradiation positions. The output of these simulations is the gas production and gamma heating tallies for the operational fluence over a period that extends beyond the expected lifetime of the reflector. That lifetime is on the order of 8 years with simulations being conducted out to 13 years in the case of MURR. The spatial distribution of fluence, gas production, and gamma ray absorption within the beryllium reflector structure provides a corresponding estimate of thermal/mechanical property changes, swelling, and heat-generation rate. The changes in mechanical properties of beryllium are estimated from a limited number of published studies in the open literature.

Using the modified thermal/mechanical properties and the gamma heating profile at various points in time, a static stress analysis is carried out on the MURR reflector structure to determine the location and magnitude of the maximum stress. The analysis is carried out using the commercial finite element code, ABAQUS. The maximum principal stress is then compared with the ultimate stress value to determine if there is a likelihood of ductile failure. At the same time, the fracture toughness is used as a multiplicative factor on the maximum principal stress to evaluate the critical crack length to check the potential for a brittle failure. The critical crack length is then compared to expected native cracks in the reflector structure. The proposed paper will review the analysis process, including the Monte Carlo simulations, the ABAQUS finite element models, and the property variations used. The limited availability of material property data on irradiated beryllium of the type used in MURR and other research/test reactors will be discussed.

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MATERIAL MANAGEMENT AND MINIMIZATION
CONVERT, REMOVE, DISPOSE

Beryllium Lifetime Analysis for Research and Test Reactors

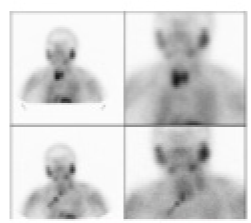
October 25, 2019
14th IEA International Workshop on Beryllium Technology, Long Beach, CA

Gary L. Solbrekken[1], Cezary Bojanowski[2], Walid Mohammad[2], Zachary Pfitzner[1], and Nickie Peters[3]

[1]Department of Mechanical and Aerospace Engineering, University of Missouri
[2]Nuclear Science and Engineering Division, Argonne National Laboratory
[3]University of Missouri Research Reactor (MURR)

Why do we care?

- MURR is the only US producer of the medical isotopes I-131, Mo-99, Lu-177, and Sm153
  - Both therapy and diagnostic procedures
  - Any adverse impact to the MURR production schedule would disrupt the isotope supply chain to the medical community
- MURR is evaluating a change from highly-enriched uranium (HEU) fuel to low-enriched uranium (LEU) U-10Mo monolithic fuel
  - Performance cannot be adversely affected
    - Core neutronic and thermal-hydraulic behavior completed
  - Structural components need to be evaluated for safety
    - Beryllium reflector is an example
- Preliminary MCNP analysis of beryllium reflector suggests that converting from HEU to LEU could:
  - Reduce gamma heating by about 20%
  - Increase swelling caused by gas production by about 10%



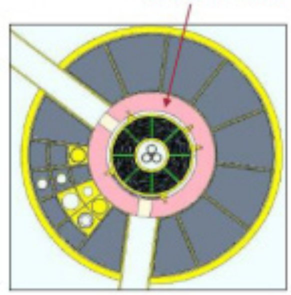
Sager S, Shafipour H, Asa S, Yılmaz S, Teksöz S, Onsel C - Indian journal of endocrinology and metabolism (2014)

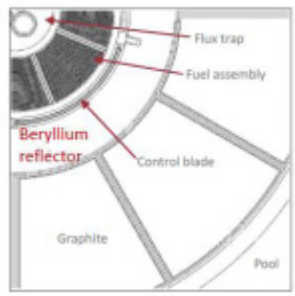
How is overall life (replacement schedule) impacted?

Current MURR Core Basic Reactor Parameters (HEU)

- MURR is a pressurized, heterogeneous, reflected, open pool-type, which is light-water moderated and cooled
- Maximum power – 10 MW_{th}
- Peak flux in center test hole – 6.0E14 n/cm²-s
- Core – 8 fuel assemblies (775 grams of U-235/assembly)
- Forced primary coolant flow rate – 3,750 gpm (237 lps)
- Forced pool coolant flow rate – 1,200 gpm (76 lps)
- Primary coolant temps – **120 °F (49 °C) inlet, 136 °F (58 °C) outlet**
- Primary coolant system pressure – 85 psia (586 kPa)
- Pool coolant temps – **100 °F (38 °C) inlet, 106 °F (41 °C) outlet**
- Preliminary thermal analysis indicates that the temperature in Be reflector is below **212 °F (100 °C)**
- **Low temperature and high neutron flux experienced by Be reflectors in research and test reactors create particularly challenging conditions (swelling due to immobile gases, high embrittlement, no annealing effect)**

Beryllium reflector





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Objective and Assumptions

- **Does the Be reflector replacement schedule need to be modified using the LEU core compared to the HEU core?**
- Replacement is dictated by mechanical failure (critical crack length) due to stresses in material exceeding mechanical integrity limits
  - 2 sources contribute to total stress – gamma heating and gas production swelling
- Current (historical) failure theory for MURR HEU core Be reflector
  - $\sigma_\gamma + \sigma_{swell} = \sigma_{total} \geq \sigma_{UTS,Be\ new}$
  - σ_{swell} is linearly scaled by fluence at single failure with HEU core (not directly modeled)
- Evaluate **RELATIVE** state of reflector for LEU vs. HEU
  - Establish new σ_γ profile based on MCNP estimate of q_γ'''
  - Estimate σ_{swell} by converting He gas production into a swelling term
  - Estimate changes in properties during irradiation based on literature and survey of data at reactors (MURR, HFIR, and ATR)

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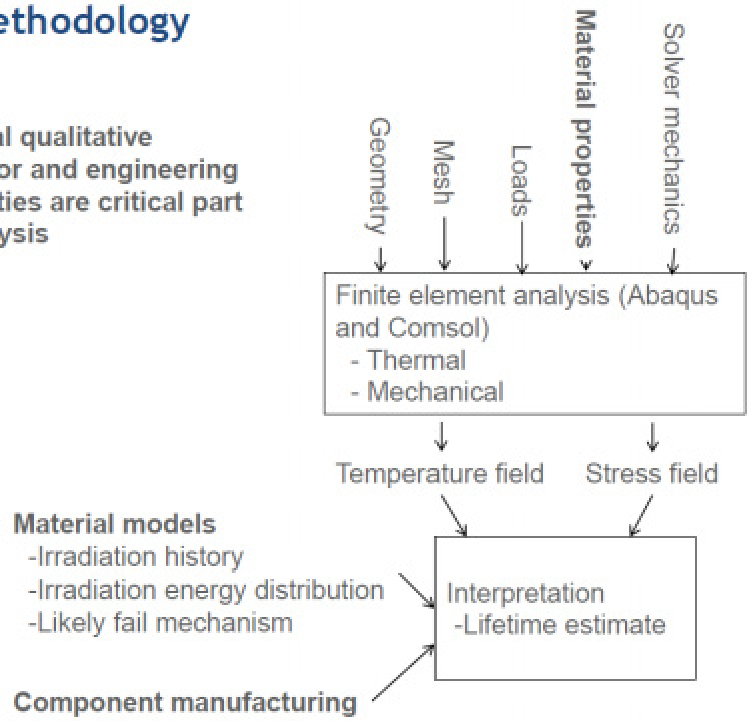
Methodology

Material qualitative behavior and engineering properties are critical part of analysis



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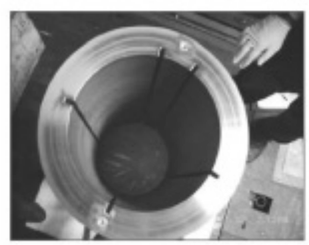
MURR Be Reflector Geometry

- The MURR Be reflector is a cylindrical sleeve located around the reactor pressure vessel at the height of the reactor core
- Its dimensions are:
  - Height: 37 inch (94 cm)
  - Outer diameter: 19 inch (48.3 cm)
  - Thickness: 2.71 in (6.9 cm)
- The reflector is made of **S-200FH** grade Be
- The reflector rests on a supporting structure without any constraints for expansion (except for two pin grooves)
- Inner spacer grooves are about 0.82-inch-deep and 0.375-inch-wide, occupied by spacers that establish channels for control blades



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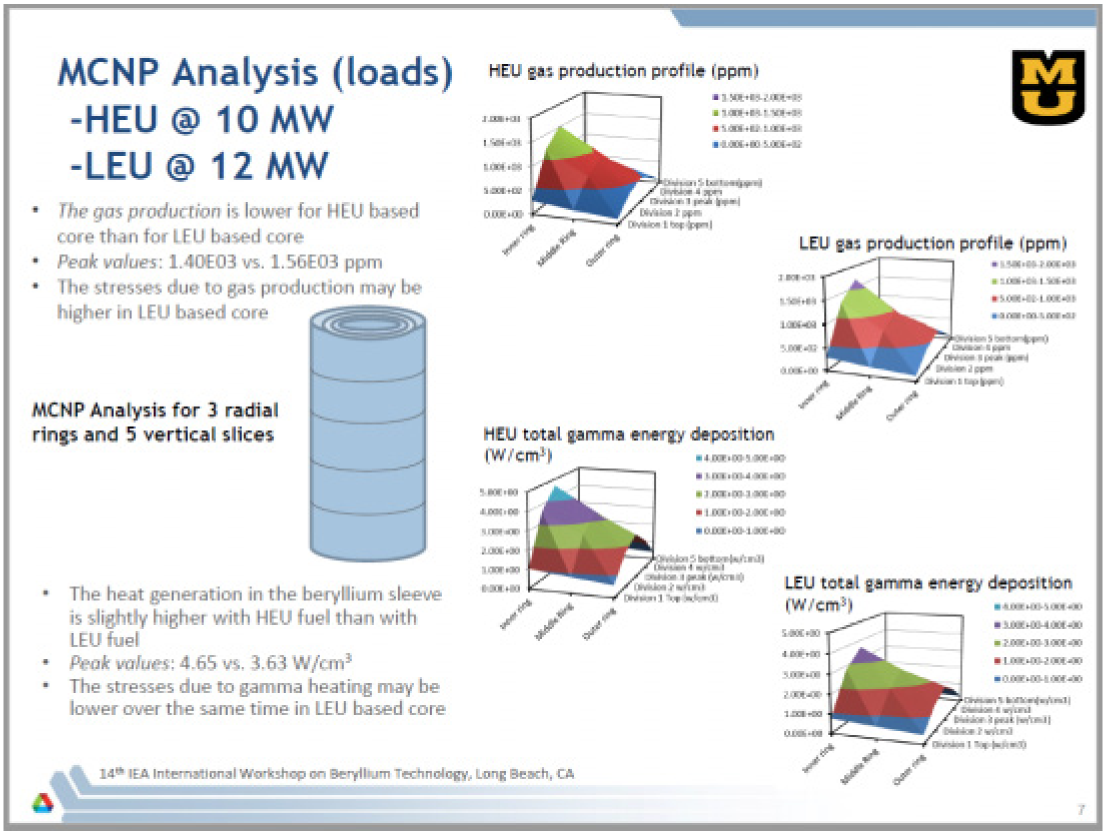
MCNP Analysis (loads)
-HEU @ 10 MW
-LEU @ 12 MW
The gas production is lower for HEU based core than for LEU based core
Peak values: 1.40E03 vs. 1.56E03 ppm
The stresses due to gas production may be higher in LEU based core
MCNP Analysis for 3 radial rings and 5 vertical slices
The heat generation in the beryllium sleeve is slightly higher with HEU fuel than with LEU fuel
Peak values: 4.65 vs. 3.63 W/cm³
The stresses due to gamma heating may be lower over the same time in LEU based core
HEU gas production profile (ppm)
LEU gas production profile (ppm)
HEU total gamma energy deposition (W/cm³)
LEU total gamma energy deposition (W/cm³)
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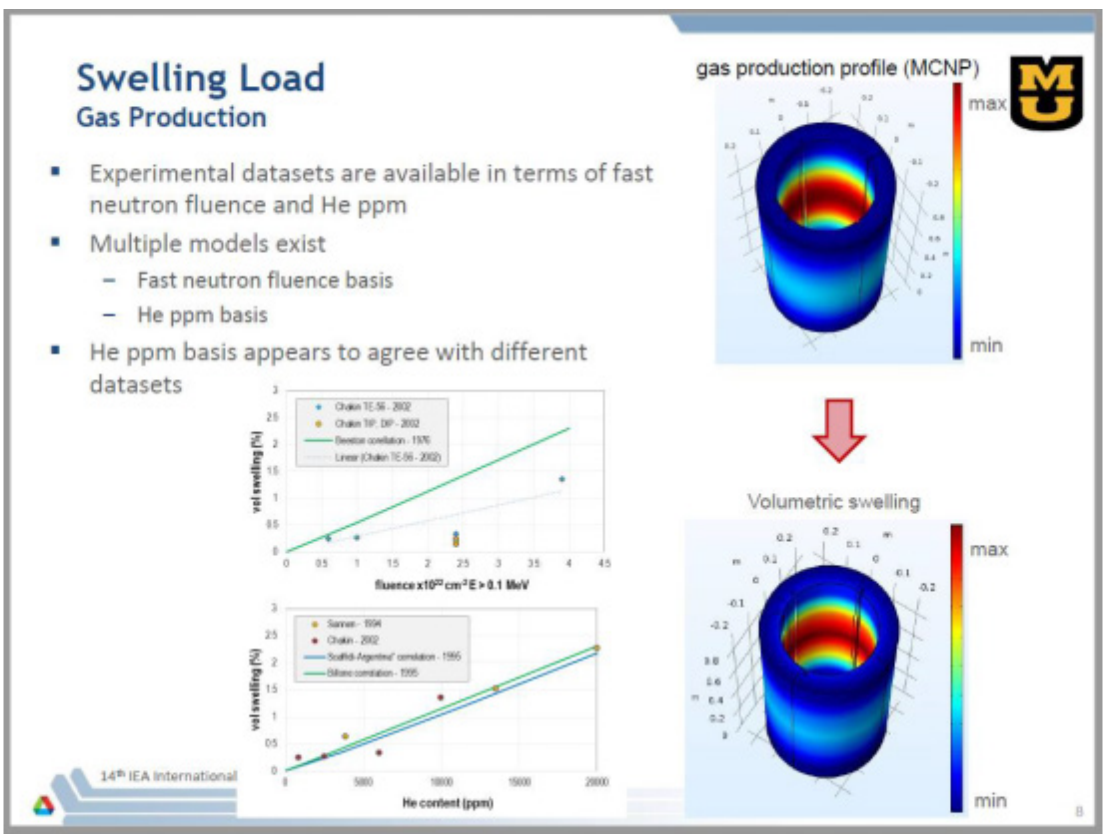
Swelling Load
Gas Production
Experimental datasets are available in terms of fast neutron fluence and He ppm
Multiple models exist
– Fast neutron fluence basis
– He ppm basis
He ppm basis appears to agree with different datasets
gas production profile (MCNP)
max
min
Volumetric swelling
max
min
vol swelling (%)
fluence x10²² cm⁻² E > 0.1 MeV
He content (ppm)
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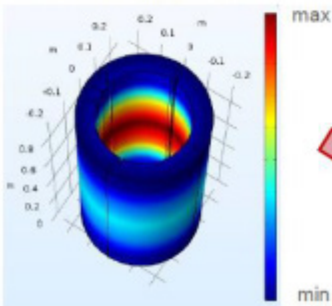
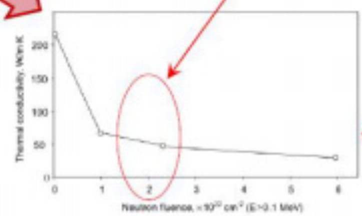
Gamma Heating Load - Thermal Expansion

Thermal Conductivity Irradiation Dependence

Fast neutron fluence - MCNP



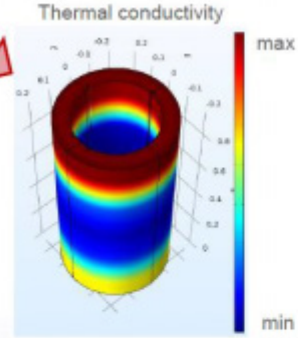
Fast neutron fluence range for current replacement schedule



"Influence of high dose neutron irradiation of thermal conductivity of Be" - Syslov, Chakin - 2002

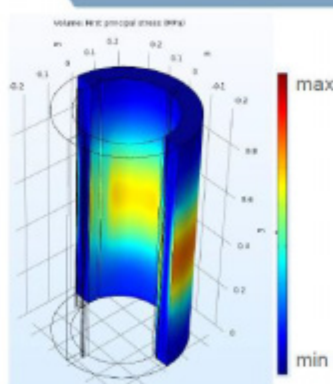
Very sparse data availability

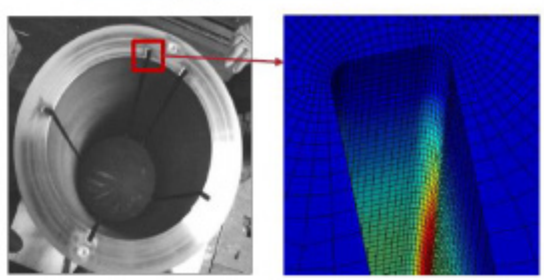
Thermal conductivity

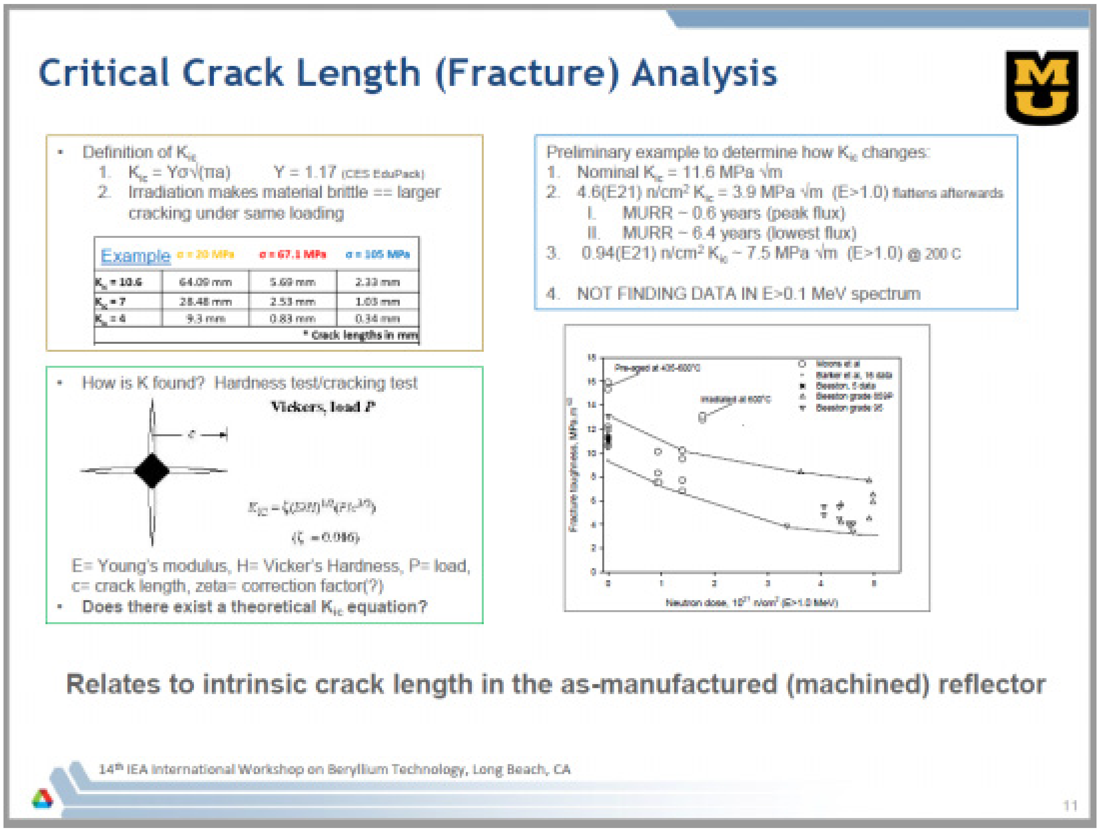


FE analysis

- Temperature and stress field obtained
- Currently calculations are performed with updated geometry, material and swelling models
- The fine geometry features act as stress concentrators and detailed models are needed
- Interpretation of results
  - UTS?
  - Critical crack length?






Critical Crack Length (Fracture) Analysis
• Definition of K_{Ic}
1. $K_{Ic} = Y\sigma\sqrt{(\pi a)}$ Y = 1.17 (CES EduPack)
2. Irradiation makes material brittle == larger cracking under same loading
Example σ = 20 MPa σ = 67.1 MPa σ = 105 MPa
K_{Ic} = 10.6 | 64.09 mm | 5.69 mm | 2.33 mm
K_{Ic} = 7 | 28.48 mm | 2.53 mm | 1.03 mm
K_{Ic} = 4 | 9.3 mm | 0.83 mm | 0.34 mm
* Crack lengths in mm
• How is K found? Hardness test/cracking test
Vickers, load P
c
$K_{IC} = \zeta(E/H)^{1/2}(P/c^{3/2})$
$(\zeta = 0.016)$
E= Young's modulus, H= Vicker's Hardness, P= load, c= crack length, zeta= correction factor(?)
• Does there exist a theoretical K_{Ic} equation?
Preliminary example to determine how K_{Ic} changes:
1. Nominal K_{Ic} = 11.6 MPa √m
2. 4.6(E21) n/cm² K_{Ic} = 3.9 MPa √m (E>1.0) flattens afterwards
I. MURR ~ 0.6 years (peak flux)
II. MURR ~ 6.4 years (lowest flux)
3. 0.94(E21) n/cm² K_{Ic} ~ 7.5 MPa √m (E>1.0) @ 200 C
4. NOT FINDING DATA IN E>0.1 MeV spectrum
Relates to intrinsic crack length in the as-manufactured (machined) reflector
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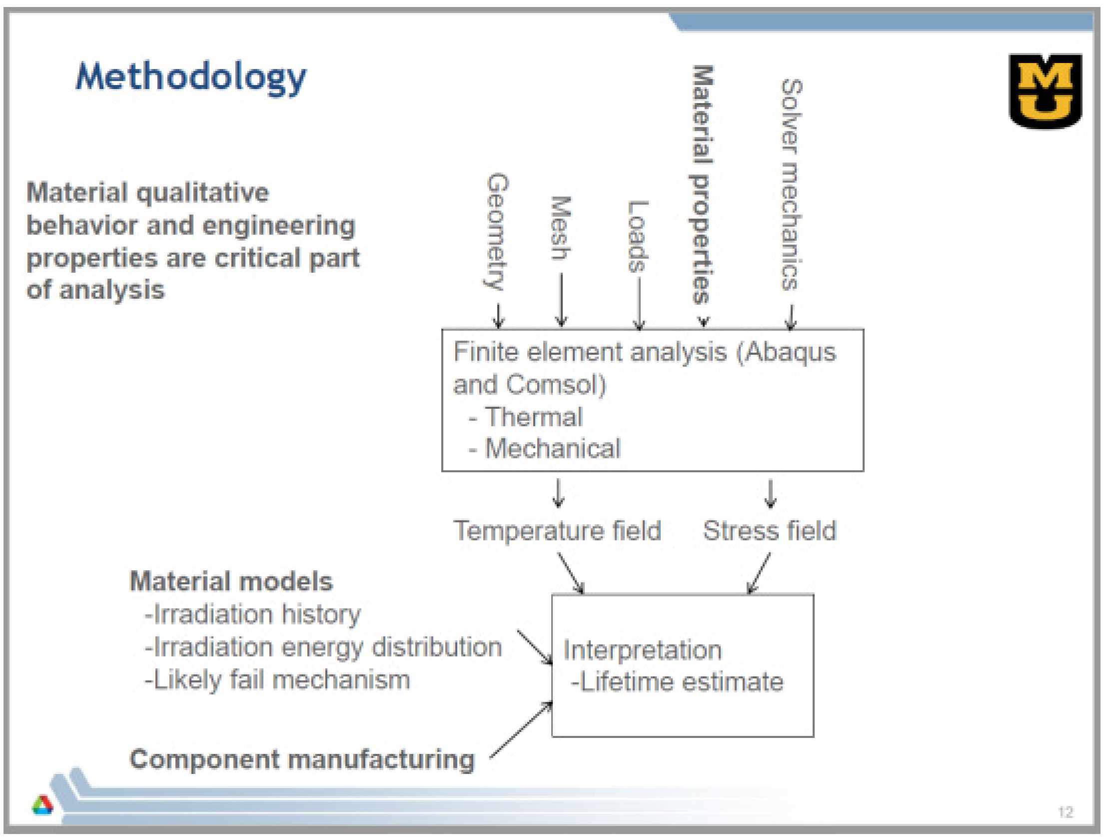
Methodology
Material qualitative behavior and engineering properties are critical part of analysis
Geometry
Mesh
Loads
Material properties
Solver mechanics
Finite element analysis (Abaqus and Comsol)
- Thermal
- Mechanical
Temperature field
Stress field
Material models
-Irradiation history
-Irradiation energy distribution
-Likely fail mechanism
Interpretation
-Lifetime estimate
Component manufacturing
12

Conclusions

- It is critical to ensure the ongoing, safe operation of MURR and other research and test reactors before making ANY operational change
  - Beryllium reflector is one such key component
- New methodology needed to evaluate replacement lifetime, based on mechanical failure analysis
- Neutron irradiation induces property changes that increase stresses
  - Property data mostly indexed to fast fluence
    - DPA better to capture full energy spectrum?
    - Standard model for MC simulation of beryllium?
  - Thermal and swelling gradients drive crack potential
  - Intrinsic cracks and embrittlement reduce critical crack length
    - Intrinsic crack characterization for fabrication operations needed for comparison
- Without clear model calibration data, only relative scaling of failure potential is possible
  - Fracture or yield?
  - DPA or He ppm indexed properties desired
- Are we missing other modeling 'best practices'?

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Acknowledgement

This work was sponsored at Argonne and U. Missouri by the U.S. Department of Energy, Office of Material Management and Minimization Office of Conversion in the U.S. National Nuclear Security Administration Office of Defense Nuclear Nonproliferation under Contract DE-AC02-06CH11357

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Valence electron structures of the beryllides using soft X-ray emission spectroscopy

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[2]*Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan*
[3]*Fusion Energy Research and Development Directorate, National Institutes for Quantum and Radiological Science and Technology, 2-166 Obuchi, Omotedate, Rokkasho, Aomori, 039-3212, Japan*

Beryllium-rich intermetallics (beryllides) have been developed as an advanced neutron multiplier for DEMO fusion reactors. Previous studies reported that the beryllides of $Be_{12}Ti$ and $Be_{12}V$ have advantages in oxidation resistance at elevated temperatures and tolerance against fast neutron irradiation compared with metallic Be. Toward an efficient mass production of neutron multiplier pebbles, additionally, $Be_{13}Zr$ beryllides has been developed as an alternative material because $Be_{13}Zr$ does not exhibit a peritectic reaction during granulation while having a low neutron absorption property. Electron structure can be altered by addition of transition metal such as Ti, V, and Zr, however, the change in the beryllides has rarely studied experimentally.

This study aims to verify valence electron structures of the beryllides experimentally using soft x-ray emission spectrometer (SXES) attached to an electron probe micro-analyzer (EPMA). The SXES covers a low energy range (50–210 eV) including Be–K_α (~110eV) with ultra-high energy resolutions as good as 0.22eV. The plasma-sintered beryllide specimens of $Be_{12}Ti$, $Be_{12}V$, and $Be_{13}Zr$ fabricated in the beryllium facility of Quantum and Radiological Science and Technology (QST) were used for the EPMA/SXES analysis. The Be–K_α spectra of $Be_{12}Ti$, $Be_{12}V$, and $Be_{13}Zr$ showed changes of valence electron structures with shoulder (or peak) at the Fermi edges due to repopulation of electrons associated with the crystallographic structural change. The experimental spectra were directly compared with density of states (DOSs) obtained by density functional theory (DFT) calculations. The Be–K_α spectra by the SXES agreed well with the calculated Be 2p states from the bulk of $Be_{12}Ti$, $Be_{12}V$, and $Be_{13}Zr$, although the photon energy was underestimated as large as 10%.

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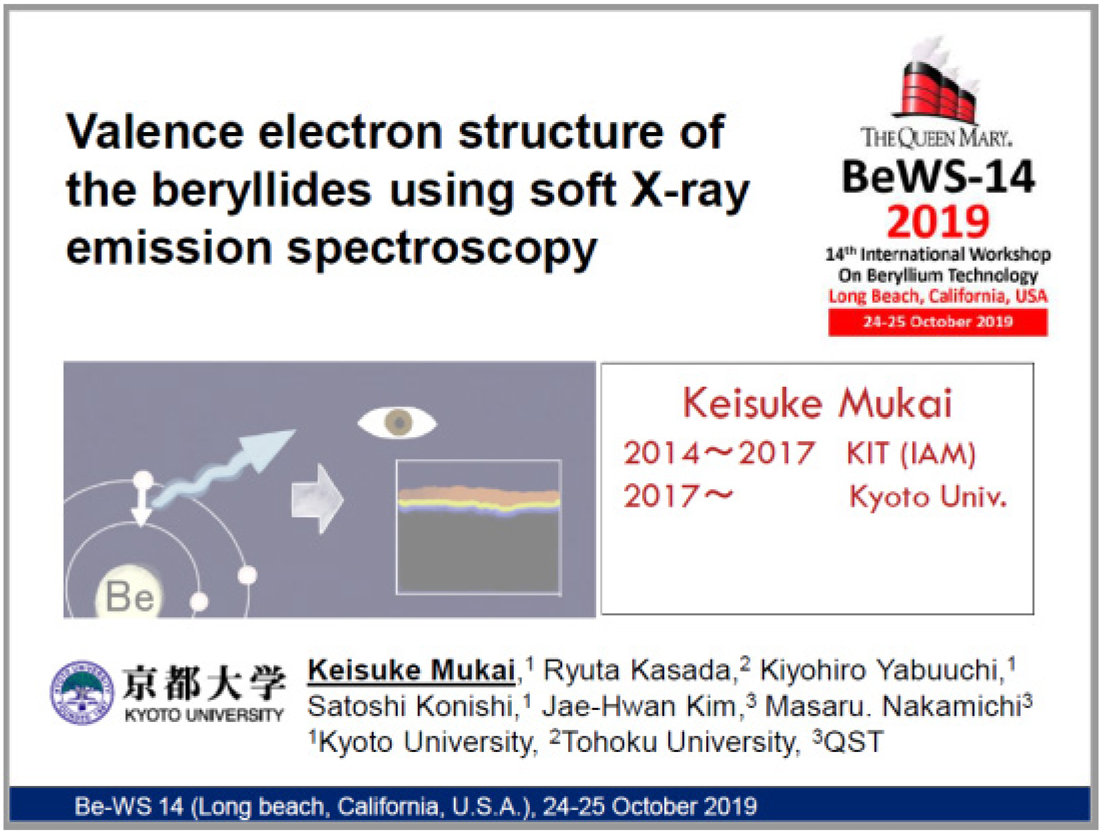
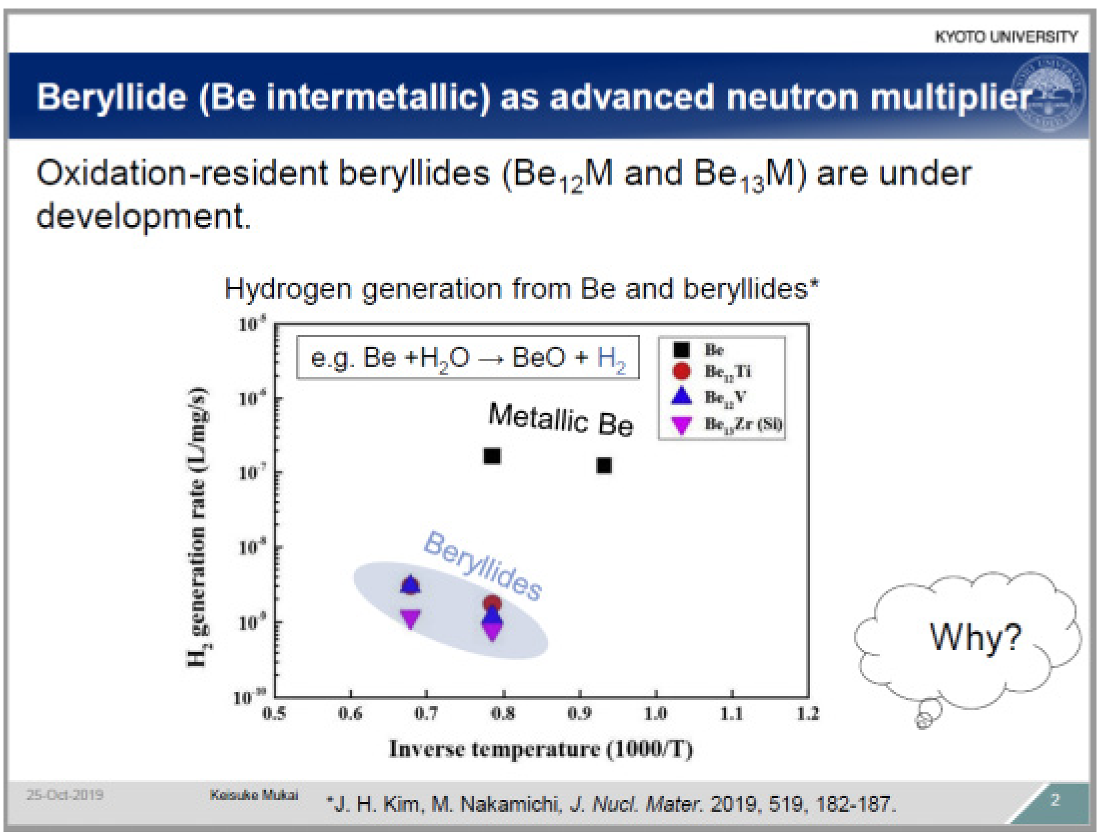

Valence electron structure of the beryllides using soft X-ray emission spectroscopy
THE QUEEN MARY
BeWS-14
2019
14th International Workshop On Beryllium Technology
Long Beach, California, USA
24-25 October 2019
Be
Keisuke Mukai
2014～2017 KIT (IAM)
2017～ Kyoto Univ.
京都大学
KYOTO UNIVERSITY
Keisuke Mukai,1 Ryuta Kasada,2 Kiyohiro Yabuuchi,1
Satoshi Konishi,1 Jae-Hwan Kim,3 Masaru. Nakamichi3
1Kyoto University, 2Tohoku University, 3QST
Be-WS 14 (Long beach, California, U.S.A.), 24-25 October 2019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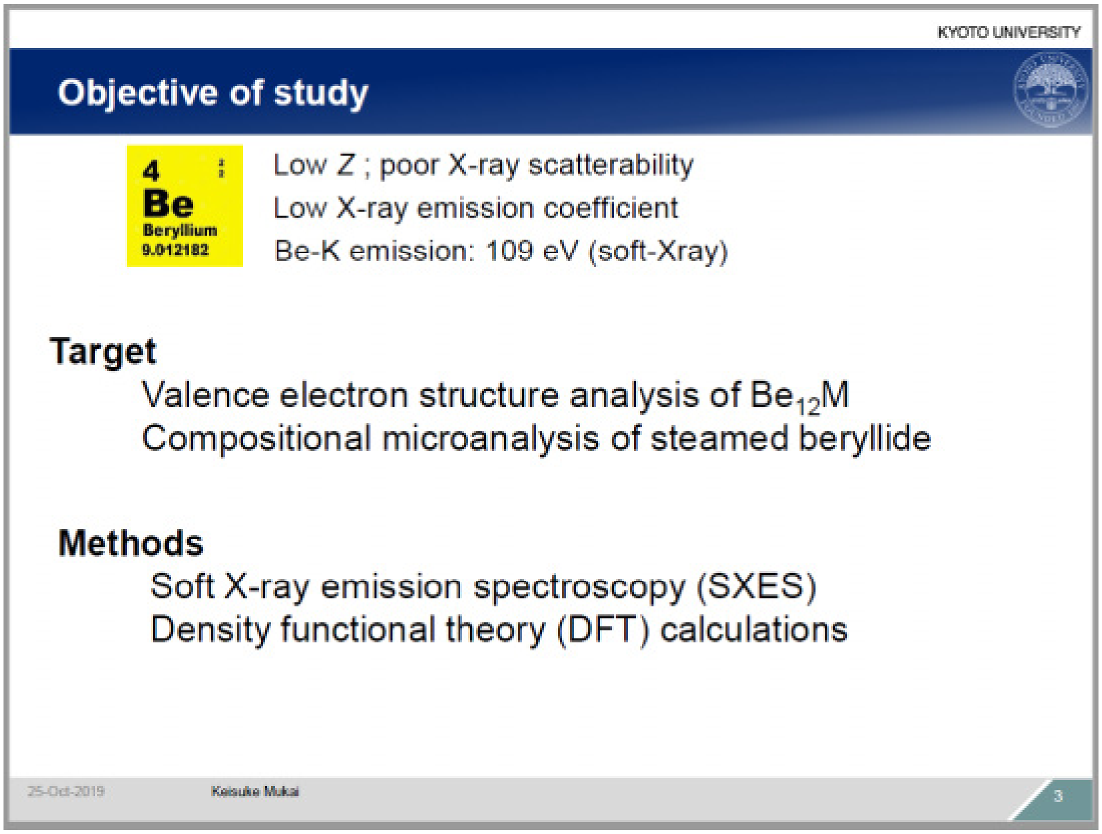
Beryllide (Be intermetallic) as advanced neutron multiplier
Oxidation-resident beryllides (Be12M and Be13M) are under development.
Hydrogen generation from Be and beryllides*
e.g. Be +H2O → BeO + H2
Be
Be12Ti
Be12V
Be13Zr (SI)
Metallic Be
Beryllides
H2 generation rate (L/mg/s)
Inverse temperature (1000/T)
Why?
25-Oct-2019
Keisuke Mukai
*J. H. Kim, M. Nakamichi, J. Nucl. Mater. 2019, 519, 182-187.
2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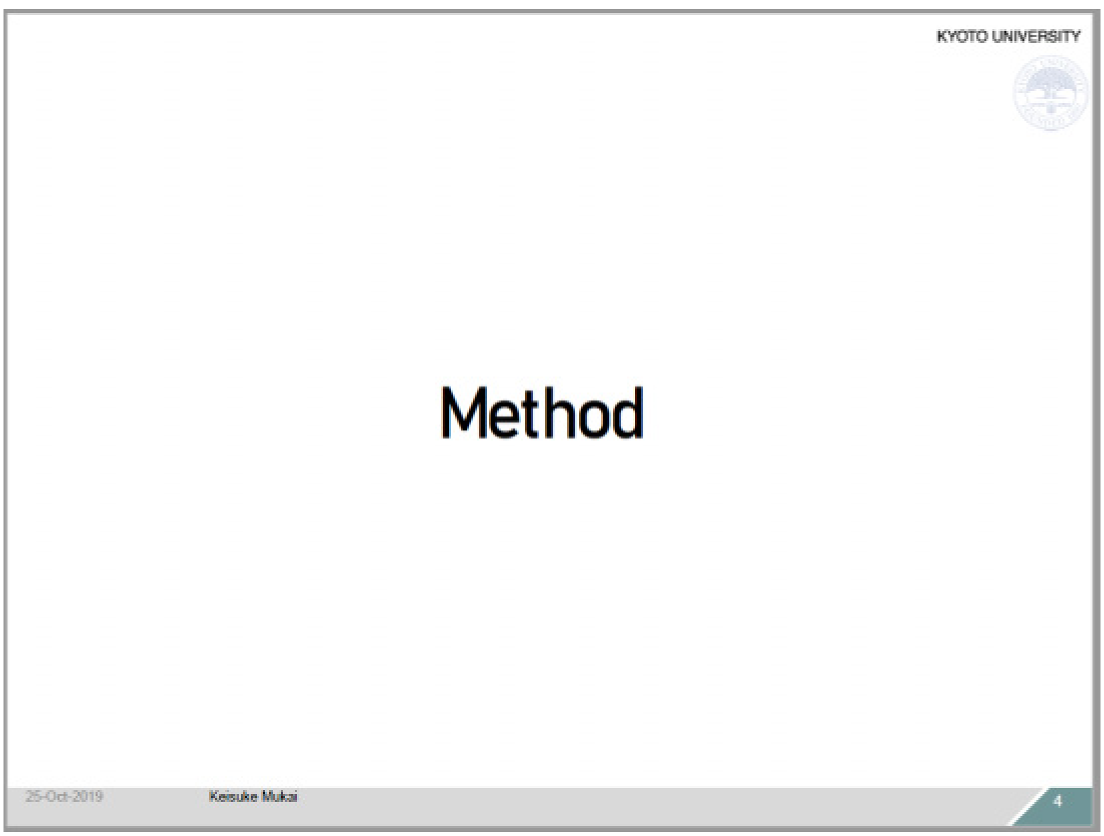
Objective of study
4
Be
Beryllium
9.012182
Low Z ; poor X-ray scatterability
Low X-ray emission coefficient
Be-K emission: 109 eV (soft-Xray)
Target
Valence electron structure analysis of Be12M
Compositional microanalysis of steamed beryllide
Methods
Soft X-ray emission spectroscopy (SXES)
Density functional theory (DFT) calculations
25-Oct-2019
Keisuke Mukai
3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Method
25-Oct-2019
Keisuke Mukai
4

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Be, beryllide, and beryllia samples

Bulk samples	Method and conditions
Be	Hot press
BeO	Plasma sintering*; 1430 °C, 15 min
Be−Ti & Be−V beryllides	Plasma sintering*; 1000 °C, 20 min
steamed $Be_{12}V$	Single-phase $Be_{12}V$ was oxidized at 1000 °C, 24h, 15%H_2O/Ar

All of the samples were prepared at QST Rokkasho
by Dr. J.H. Kim & Dr. M. Nakamichi

*M. Nakamichi, J.H. Kim, K. Yonehara, *Fusion Eng. Des.* 88 (2013) 611−615.

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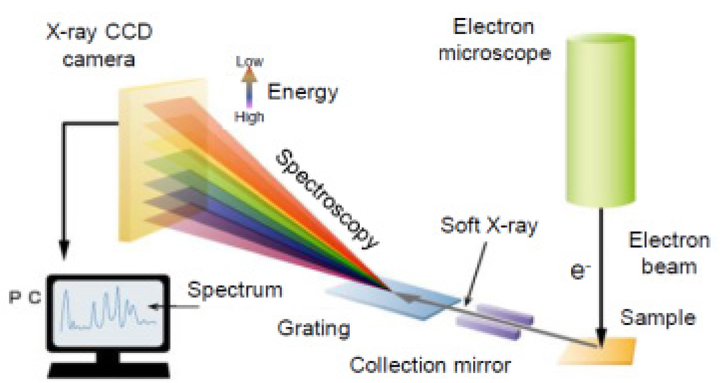
EPMA with Soft X-ray emission spectrometer (SXES)



SXES by JEOL is attached to electron probe micro analyzer (EPMA) JXA-8500F.

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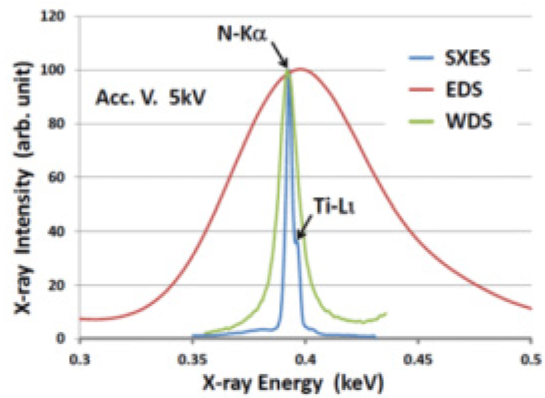
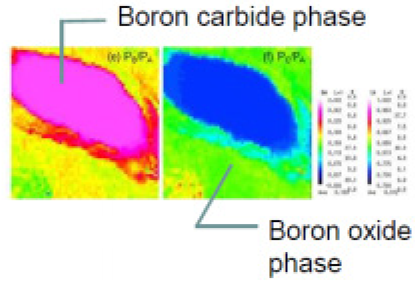
Advantages of the SXES

Energy resolution*



Chemical state mapping;

B_4C after steam oxidation (1250 °C, 30 min)**



Low energy range 50～200 eV covers Li-Kα (54 eV) and Be-Kα (110 eV)
High energy resolution (0.22 eV @Al-L) enables chemical state mapping**

*JEOL web site
**R. Kasada, Y. Ha, T. Higuchi, K. Sakamoto, *Sci. Rep.* 2016, 6, 25700.

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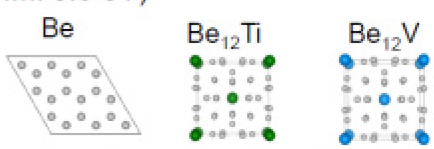
Theoretical Be-K emission by DFT calculations

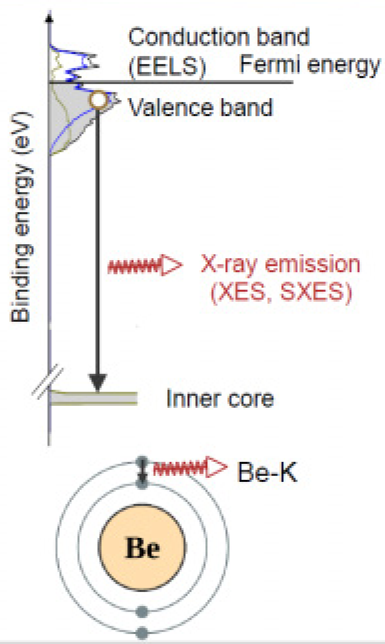
Projected DOS by VASP code (PBE-GGA functionals)

Be: $1s^2$, $2s^2$, O: $2s^22p^4$, V: $3s^23p^63d^44s^1$

- Be metal (3×3×3 supercell, 54 atoms)
- BeO (2×2×2 supercell, 32 atoms)
- $Be_{12}Ti$ (unit cell, 26 atoms)
- $Be_{12}V$ (unit cell, 26 atoms)

Convoluted of Be-PDOS with Gaussian (FWHM: 0.6 eV)





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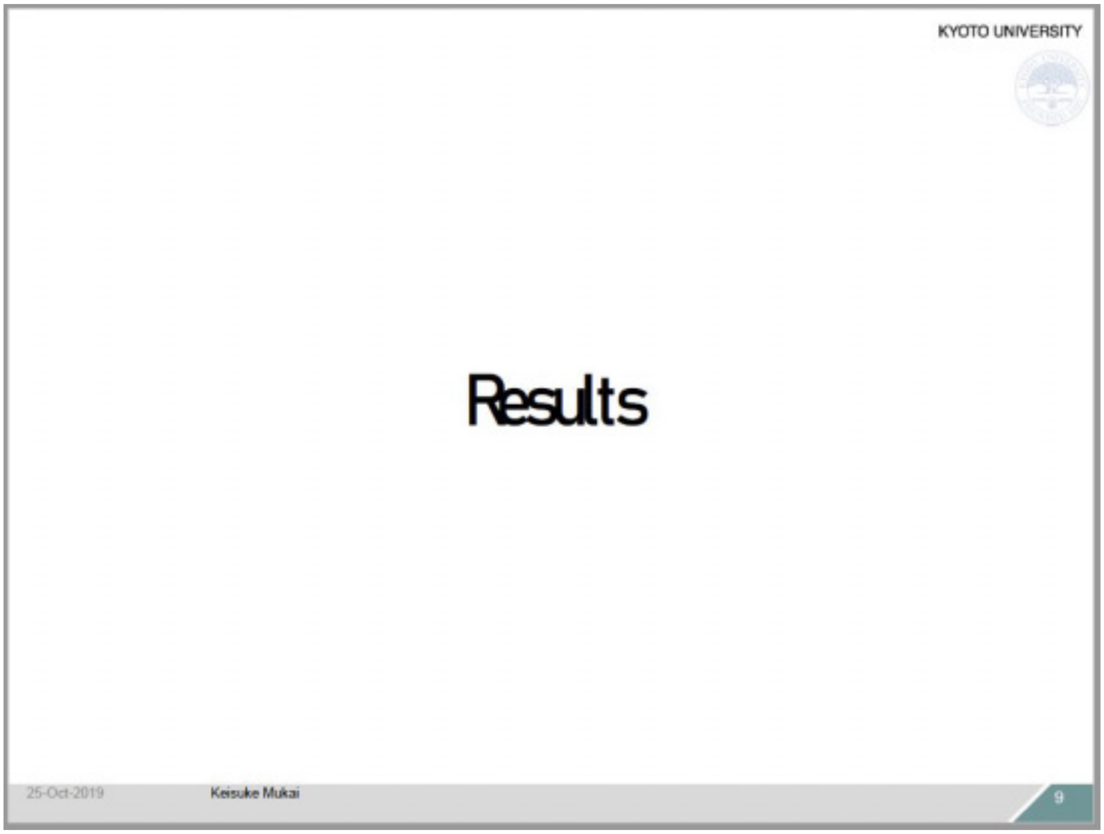
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Results
25-Oct-2019
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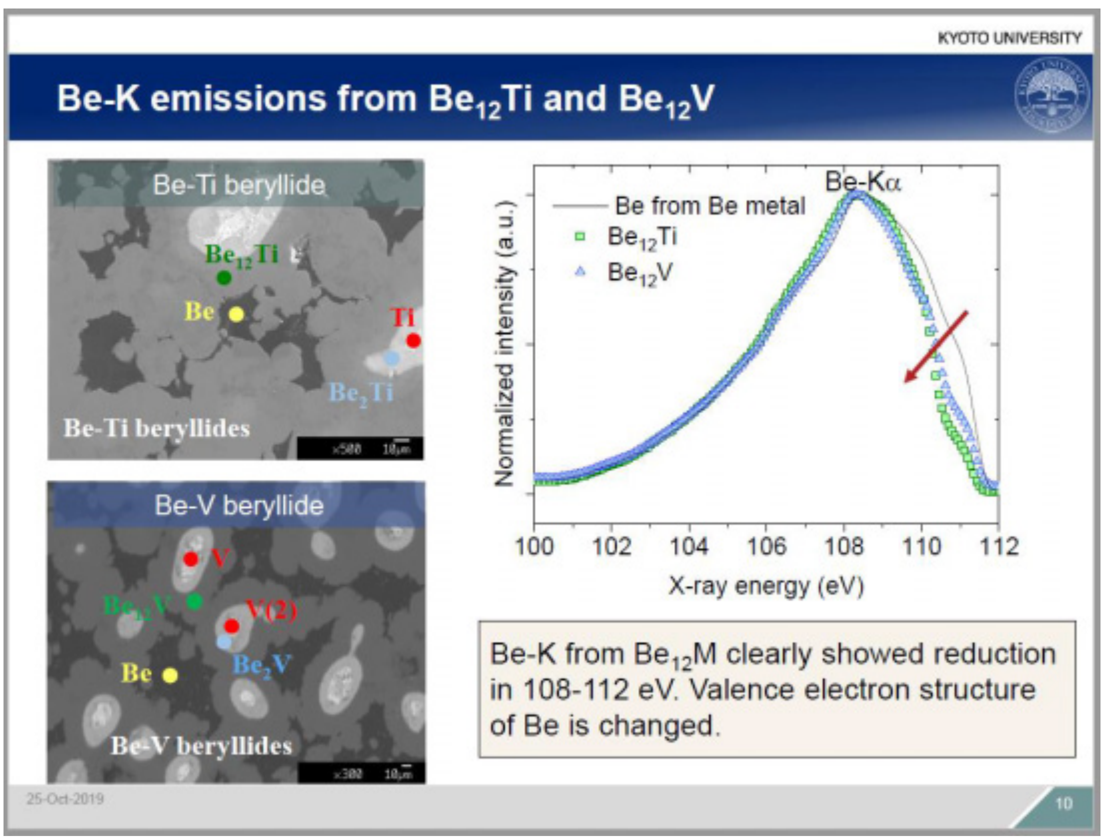
9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Be-K emissions from $Be_{12}Ti$ and $Be_{12}V$
Be-Ti beryllide
$Be_{12}Ti$
Be
Ti
Be_2Ti
Be-Ti beryllides
Be-V beryllide
V
$Be_{12}V$
V(2)
Be
Be_2V
Be-V beryllides
Be-Kα
Be from Be metal
$Be_{12}Ti$
$Be_{12}V$
Normalized intensity (a.u.)
100
102
104
106
108
110
112
X-ray energy (eV)
Be-K from $Be_{12}M$ clearly showed reduction in 108-112 eV. Valence electron structure of Be is changed.
25-Oct-2019
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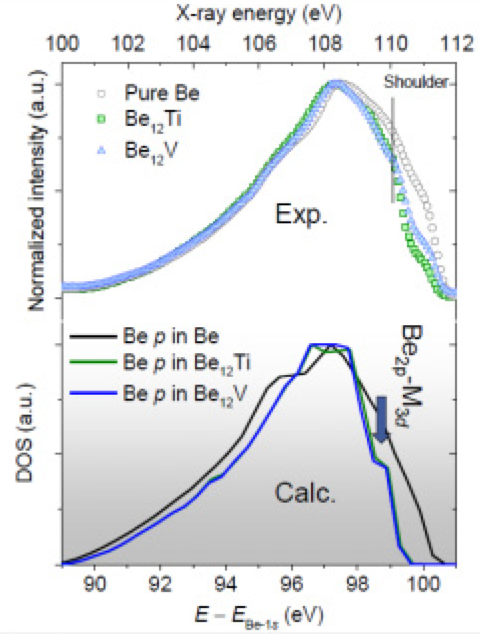
Comparison between Be-Kα by SXES and DFT calc.



- Be 2p state was convoluted with Gaussian (FWHM: 0.6 eV) and compared with SXES spectra.
- SXES spectra were consistent with Be 2p electron structures with 10% of underestimation in energy.
- Shoulder shows coverency between 2p state of Be and 3d of V/Ti.

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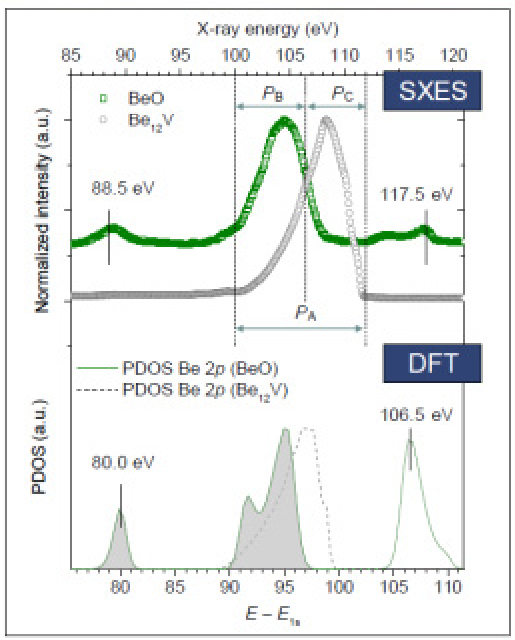
Be-K emissions from BeO and $Be_{12}V$

A large chemical shift (~4 eV) was observed in BeO because of 1s core level shift.

P_A: 100 ~ 112 eV
P_B: 100~106.5 eV
P_C: 106.5 ~ 112 eV



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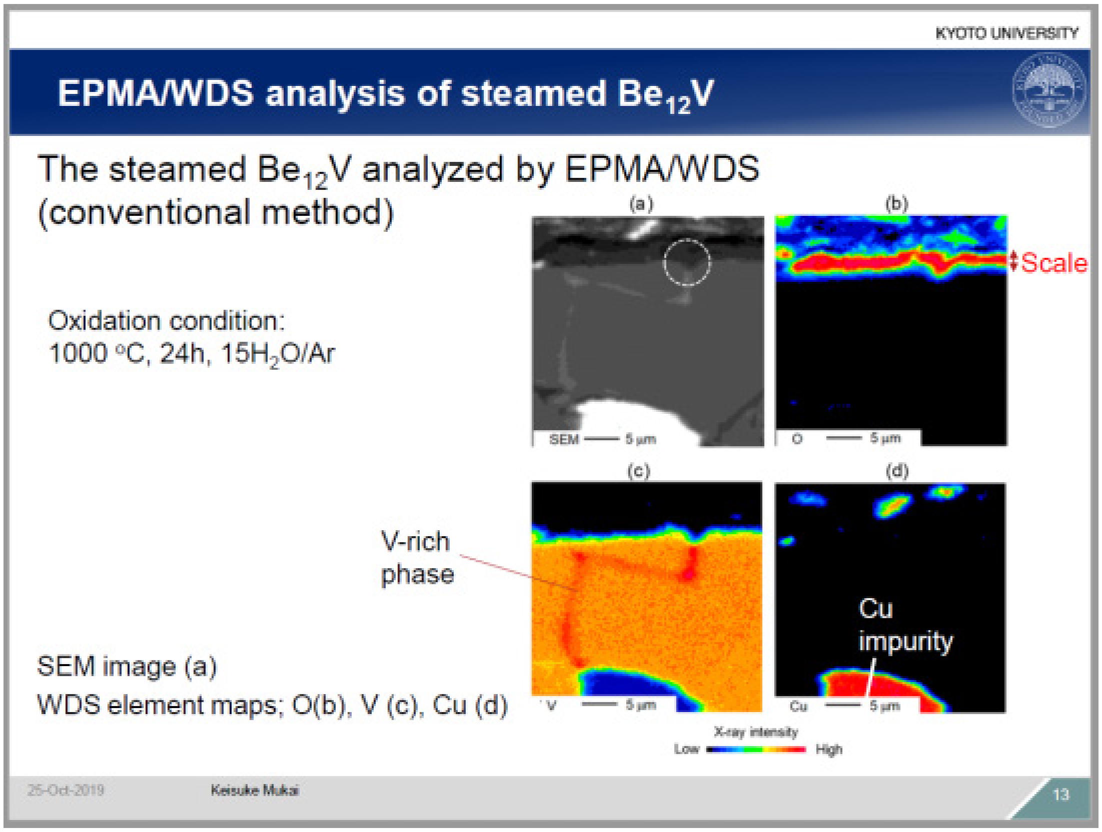
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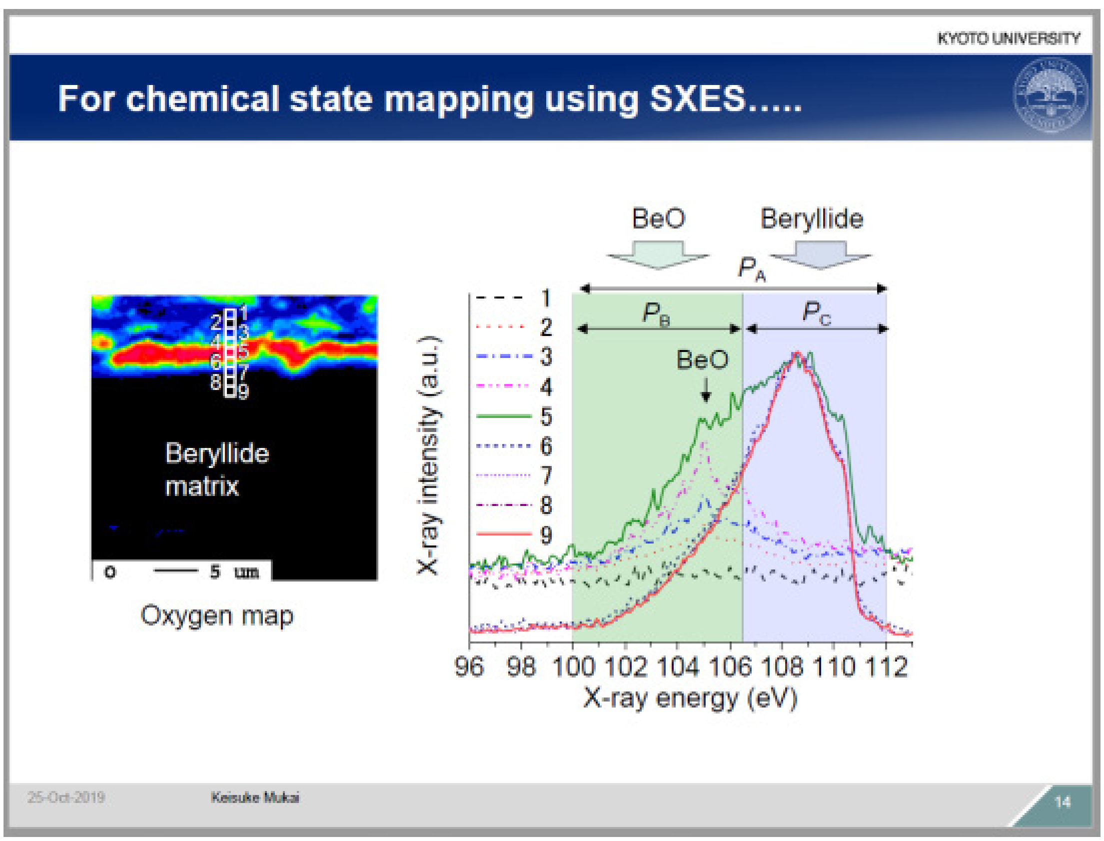
EPMA/WDS analysis of steamed Be12V
The steamed Be12V analyzed by EPMA/WDS (conventional method)
Oxidation condition:
1000 °C, 24h, 15H2O/Ar
(a)
(b)
(c)
(d)
Scale
SEM 5 µm
O 5 µm
V-rich phase
Cu impurity
V 5 µm
Cu 5 µm
SEM image (a)
WDS element maps; O(b), V (c), Cu (d)
X-ray intensity
Low
High
25-Oct-2019
Keisuke Mukai
13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For chemical state mapping using SXES.....
BeO
Beryllide
PA
PB
PC
BeO
1
2
3
4
5
6
7
8
9
X-ray intensity (a.u.)
96 98 100 102 104 106 108 110 112
X-ray energy (eV)
Beryllide matrix
O 5 um
Oxygen map
25-Oct-2019
Keisuke Mukai
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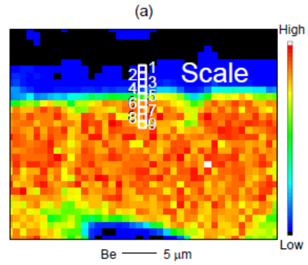
Analysis of steamed $Be_{12}V$ by SXES

40×32 pixels (1pixel: 1 μm×1 μm, 30 s/pixel) by SXES.

Element mapping

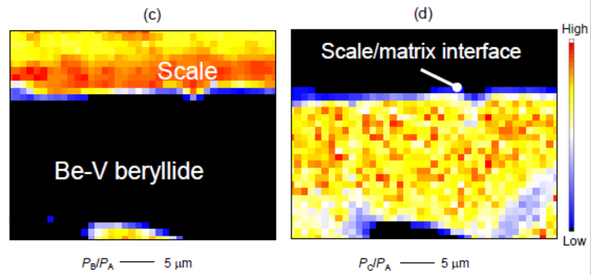
P_A: Be element map



Chemical state mappings

P_B/P_A: BeO map (left)

P_C/P_A: Beryllide map (right)



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Conclusion

- ✓ Valence electron structure of Be in the Be-compounds were successfully analyzed from Be-K emission using SXES.
- ✓ Using the large chemical shift (~4 eV) in BeO, chemical state mappings in steamed $Be_{12}V$ was successfully visualized
- ✓ Influence of valence electron structure on chemical reactivity will be investigated in future work.

Publication

K. Mukai, R. Kasada, K. Yabuuchi, S. Konishi, J. H. Kim, M. Nakamichi, *ACS Applied Energy Materials* **2019**, 2, 2889-2895

Acknowledgement:
This work is supported by the Joint Usage/Research Program on Zero-Emission Energy Research, Institute of Advanced Energy, Kyoto University (ZE29A-12, ZE30A-23)

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TEM analytical study and *ab initio* simulation of impurities in beryllium

P. Vladimirov, N. Zimber, and M. Klimenkov

Karlsruhe Institute of Technology, 76131 Karlsruhe, Germany

Impurities and their spatial distribution significantly affect mechanical properties of beryllium and microstructural evolution under neutron irradiation.

In this work we investigate interaction of naturally occurring impurities (Al, Fe and Si) with vacancies using analytical TEM and modelling based on the density functional theory. TEM studies have revealed abundant formation of Al-Fe-Be precipitates, formation of complex multiple phase precipitates, homogeneous segregations of elements to grain boundaries as well as Al-Fe-Be phase precipitation along dislocations. All precipitates are richly decorated with helium bubbles which are smaller in size than typical bubbles inside grains. Precipitates-free and helium-bubble-free zones were observed along grain boundaries.

These experimental observations have inspired us to simulate interaction of impurities with vacancies as well as to calculate their migration barriers. These simulations were performed using the simulation program package VASP enhanced with the VTST extensions for the nudge elastic band (NEB) calculation of migration paths.

Our simulations revealed strong attraction of Al and Si to vacancies, while Fe, unexpectedly, repels vacancies. This fact should also have implications on the mechanism of the solute atom diffusion: Al and Si might diffuse as a vacancy-solute atom complex going along vacancy gradient, while unbound Fe should rather move against it (so called inverse Kirkendall effect). Using the NEB method, we have calculated the migration barriers for these solutes if they migrate by the vacancy-exchange mechanism. For both iron and aluminum, diffusion barriers are similar for the jumps within and outside of basal plane suggesting their isotropic diffusion within beryllium matrix.

On the contrary, the diffusion barrier of silicon is approximately two times lower than that of iron and aluminum within basal plane but is comparable with that when jumping out of basal plane. As it was shown by us previously, helium diffusion is also highly anisotropic. These facts imply a consequence for the width of zones denuded from precipitates and helium bubbles. Due to the nearly isotropic diffusion of Al and Fe the width of zones denuded in Al-Fe-Be precipitates does not depend on the orientation of grains with respect to the grain boundary. In contrast, anisotropic helium diffusion should result in variation of the width of bubble-denuded zones depending on the inclination angle of the basal plane to the grain boundary. It seems that our preliminary experimental results support this hypothesis.

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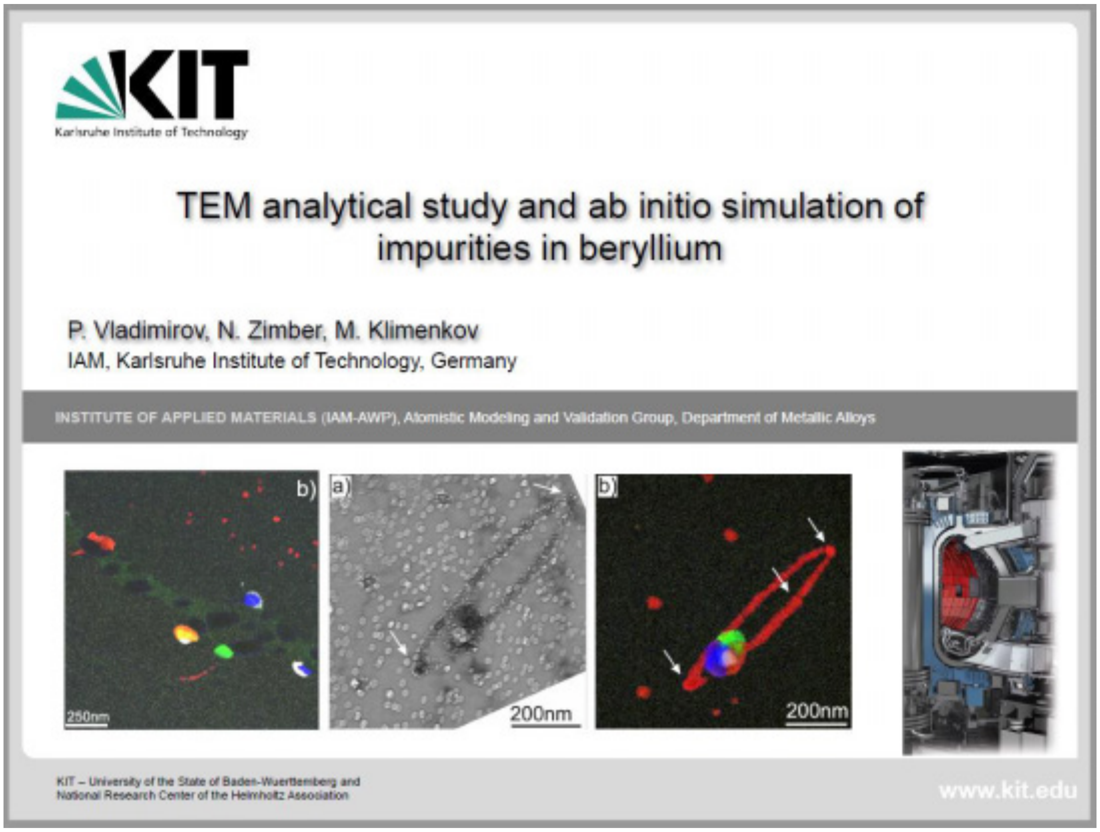
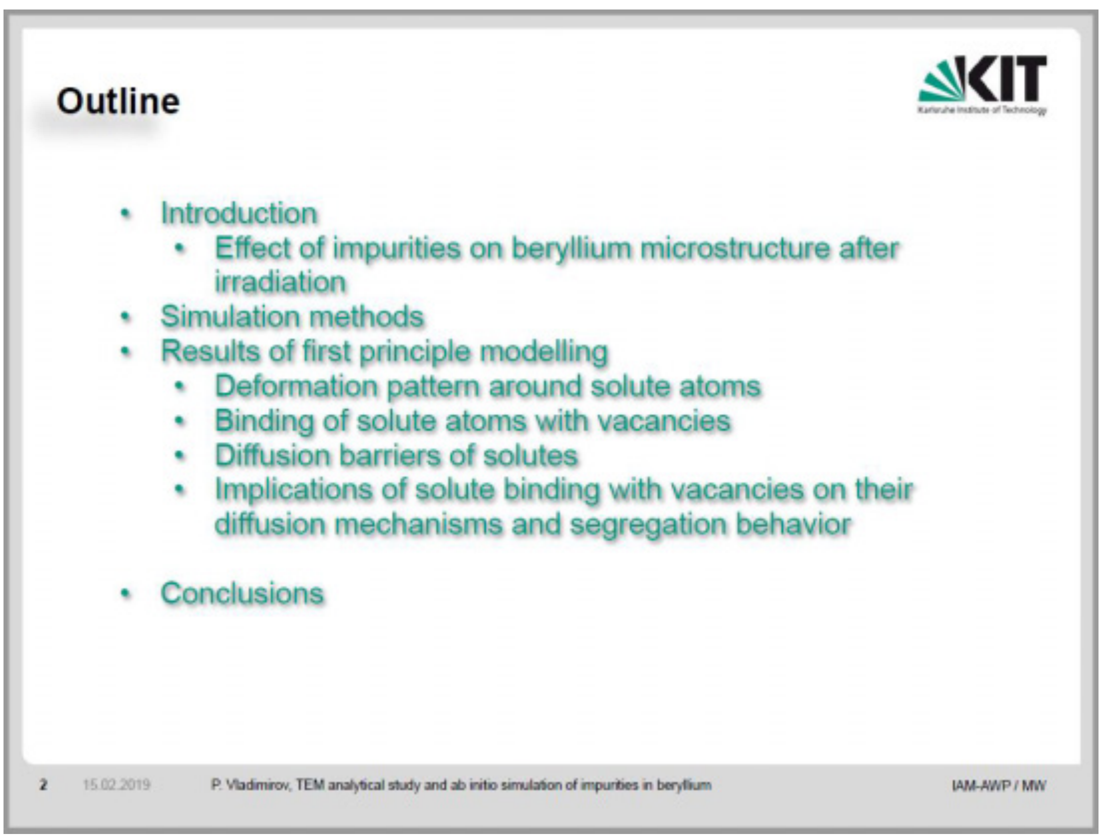
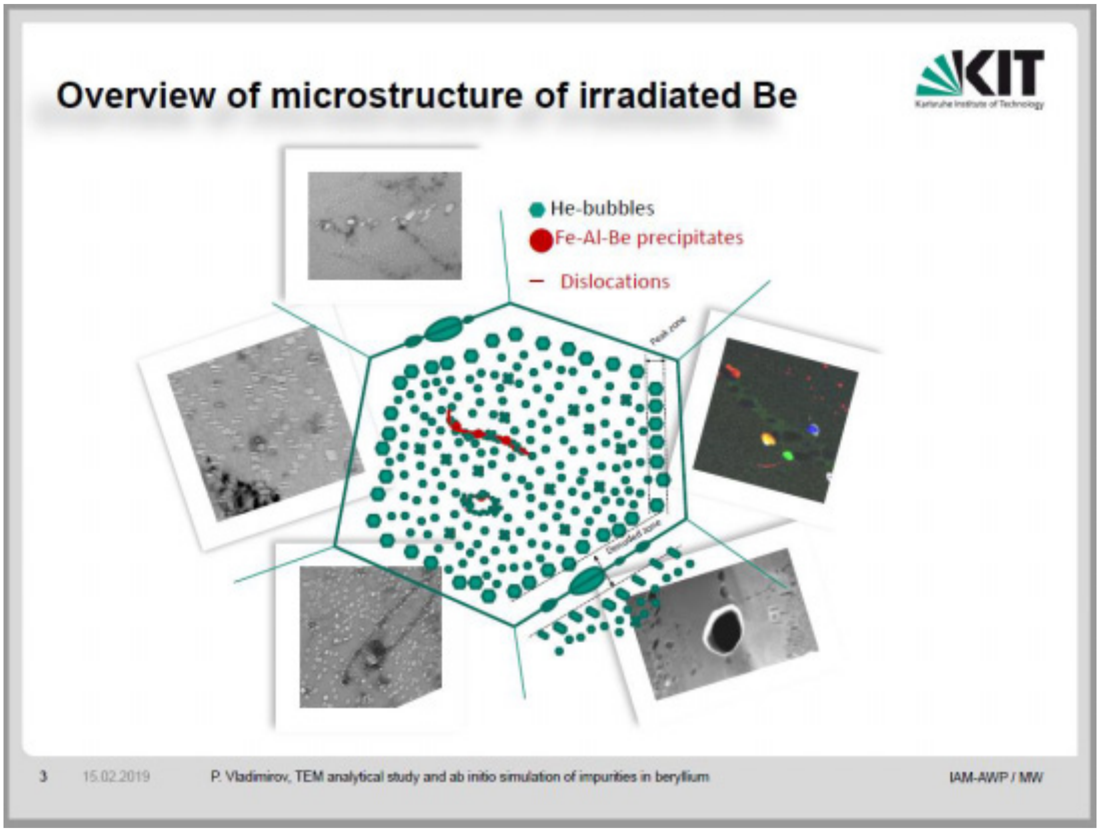
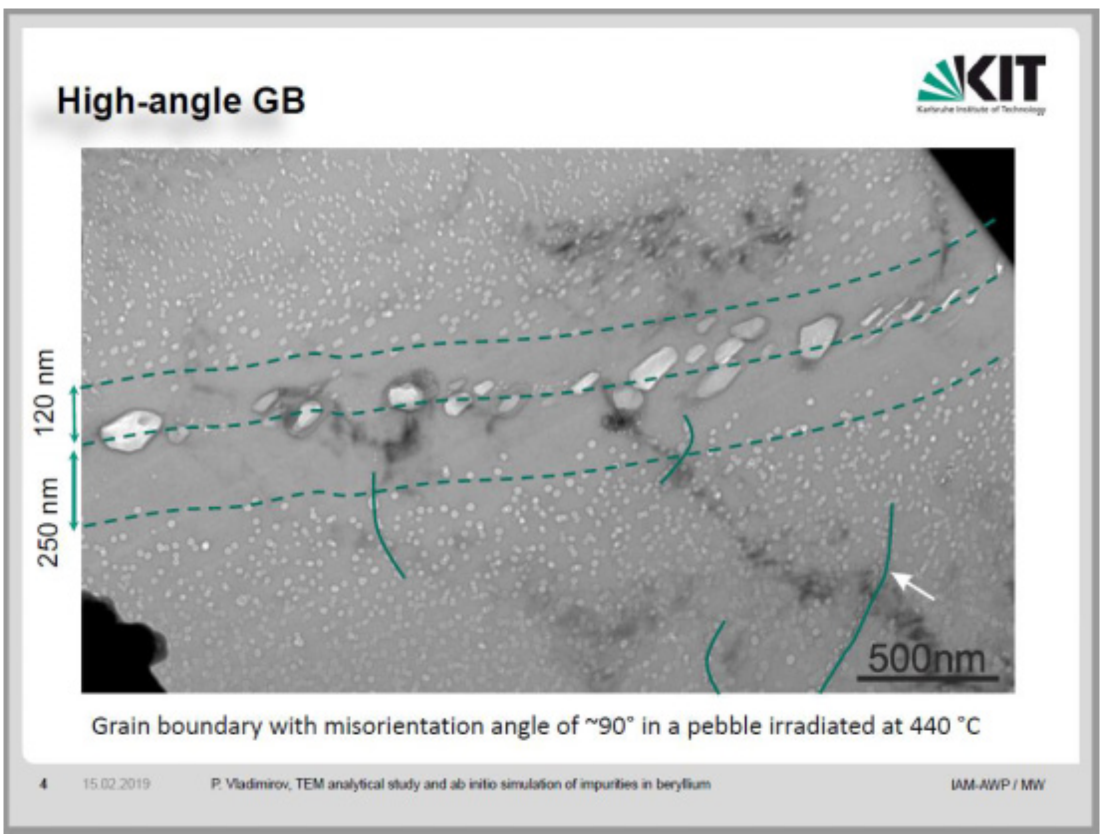
GERMANY

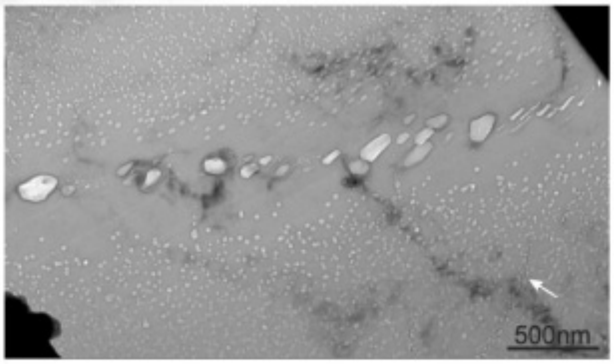

Karlsruhe Institute of Technology
TEM analytical study and ab initio simulation of impurities in beryllium
P. Vladimirov, N. Zimber, M. Klimenkov
IAM, Karlsruhe Institute of Technology, Germany
INSTITUTE OF APPLIED MATERIALS (IAM-AWP), Atomistic Modeling and Validation Group, Department of Metallic Alloys
b)
250nm
a)
200nm
b)
200nm
KIT – University of the State of Baden-Wuerttemberg and National Research Center of the Helmholtz Association
www.kit.edu

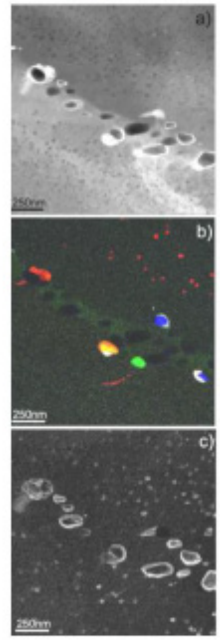

Outline
Karlsruhe Institute of Technology
• Introduction
• Effect of impurities on beryllium microstructure after irradiation
• Simulation methods
• Results of first principle modelling
• Deformation pattern around solute atoms
• Binding of solute atoms with vacancies
• Diffusion barriers of solutes
• Implications of solute binding with vacancies on their diffusion mechanisms and segregation behavior
• Conclusions
2
15.02.2019
P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium
IAM-AWP / MW


Overview of microstructure of irradiated Be
KIT
He-bubbles
Fe-Al-Be precipitates
Dislocations
3
15.02.2019
P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium
IAM-AWP / MW


High-angle GB
KIT
120 nm
250 nm
500nm
Grain boundary with misorientation angle of ~90° in a pebble irradiated at 440 °C
4
15.02.2019
P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium
IAM-AWP / MW

High-angle GB

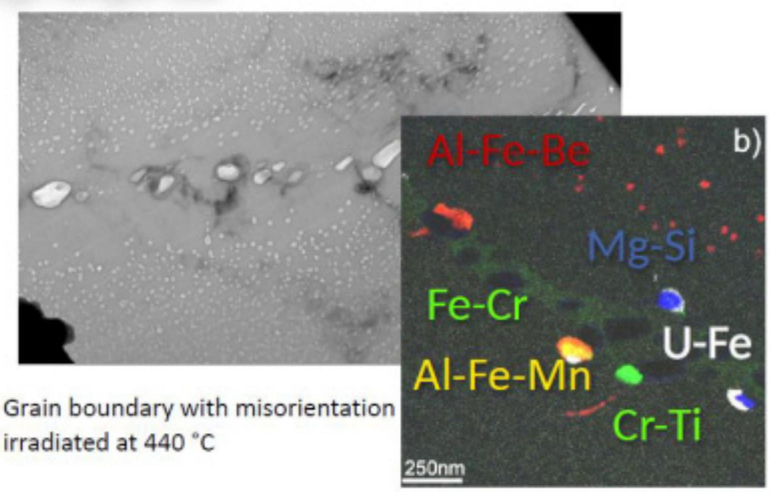


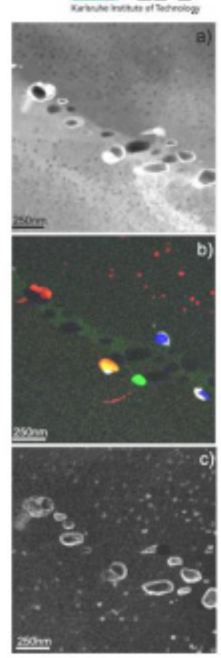


Grain boundary with misorientation angle of ~90° in a pebble irradiated at 440 °C

5 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW

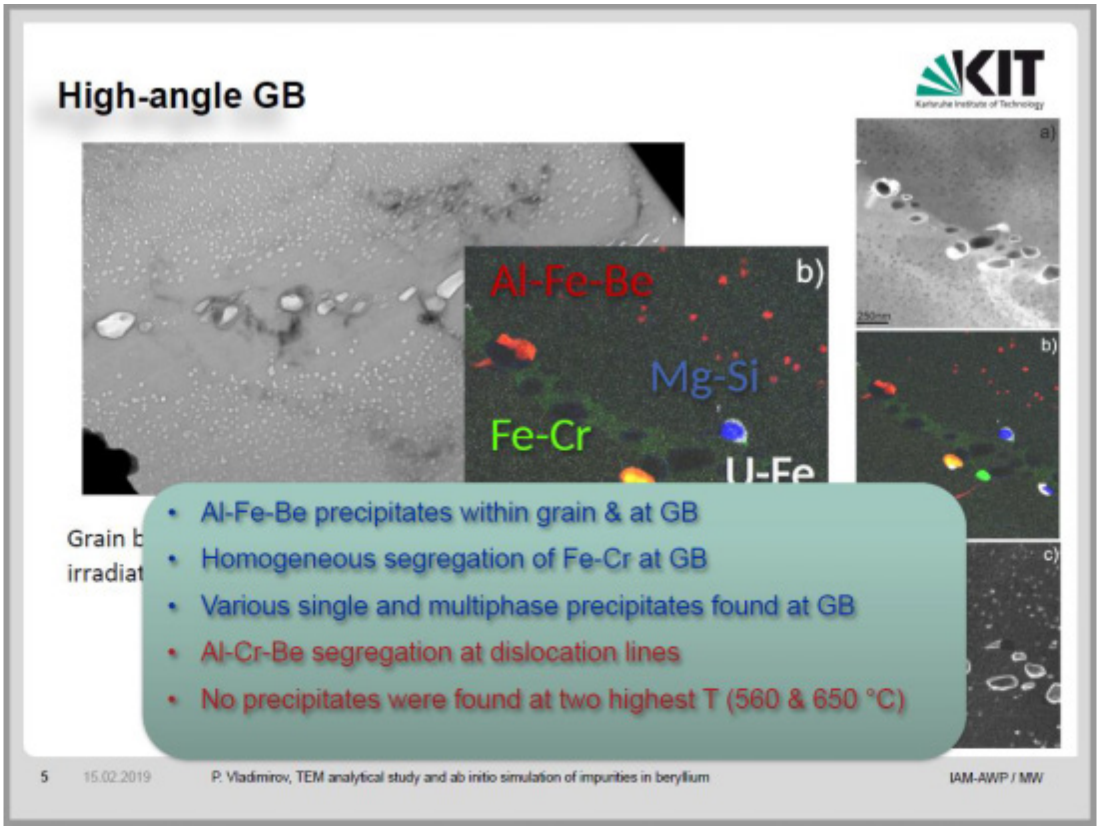
High-angle GB

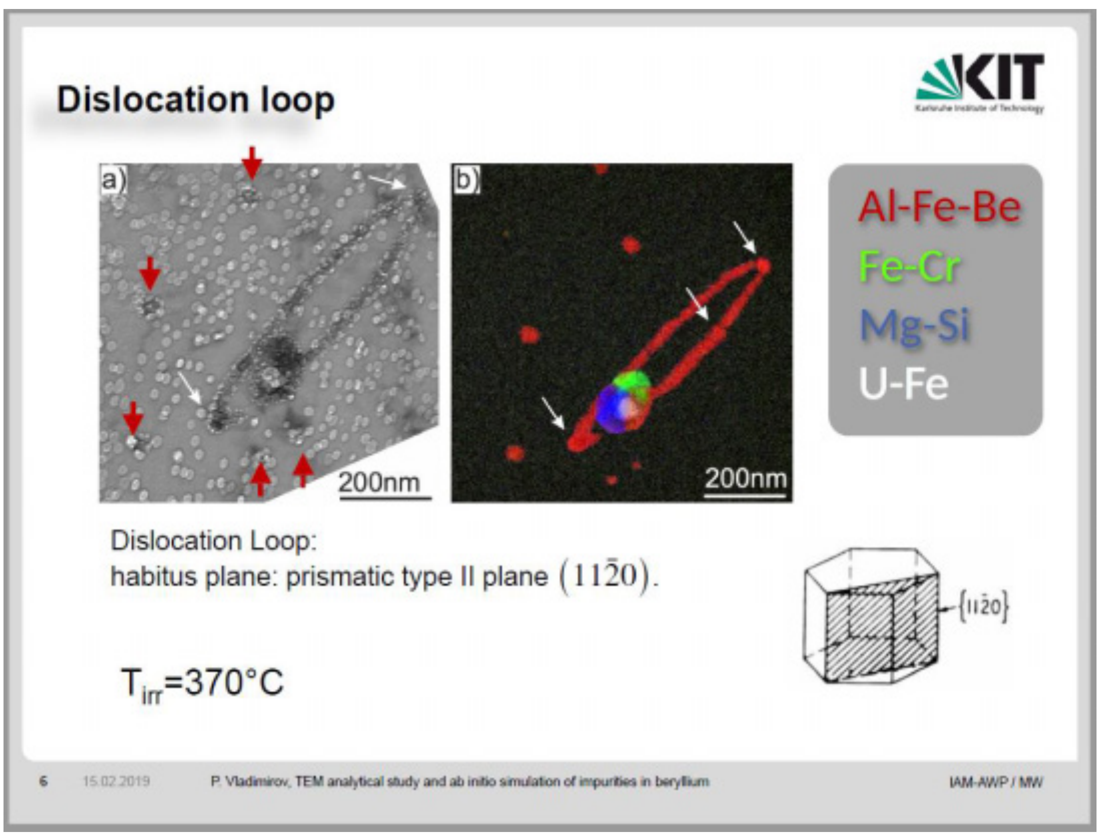


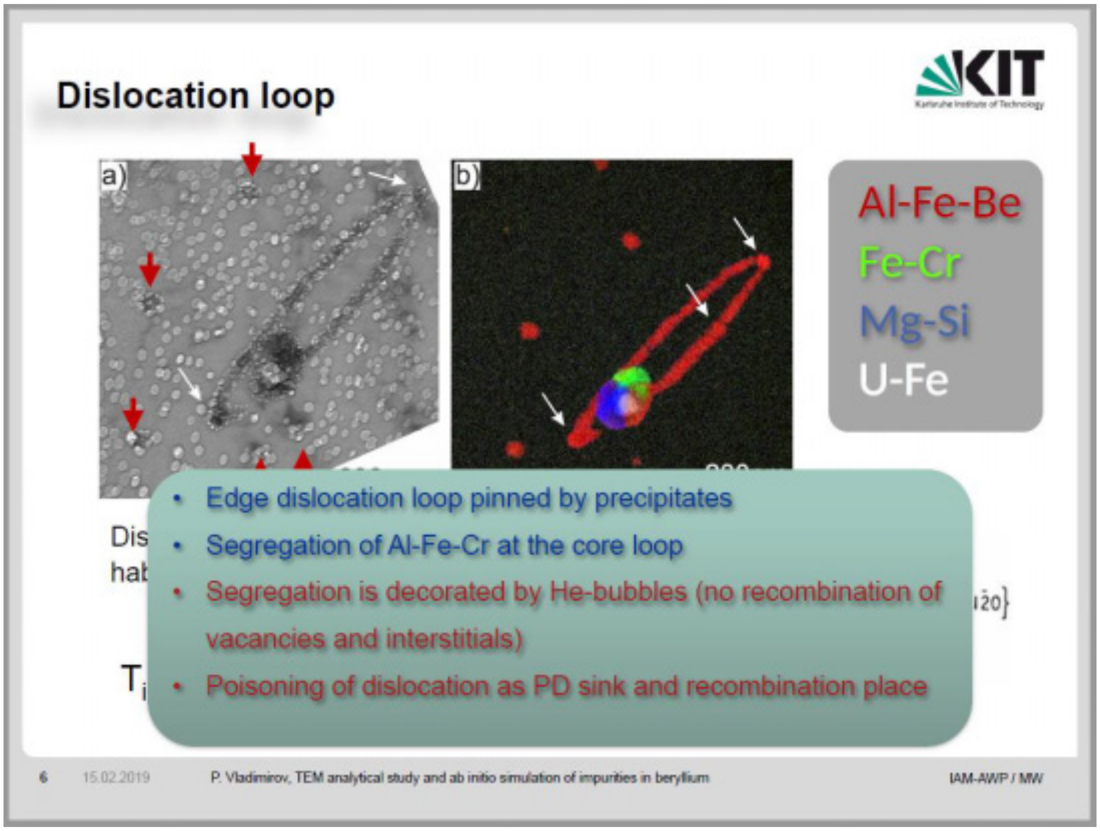


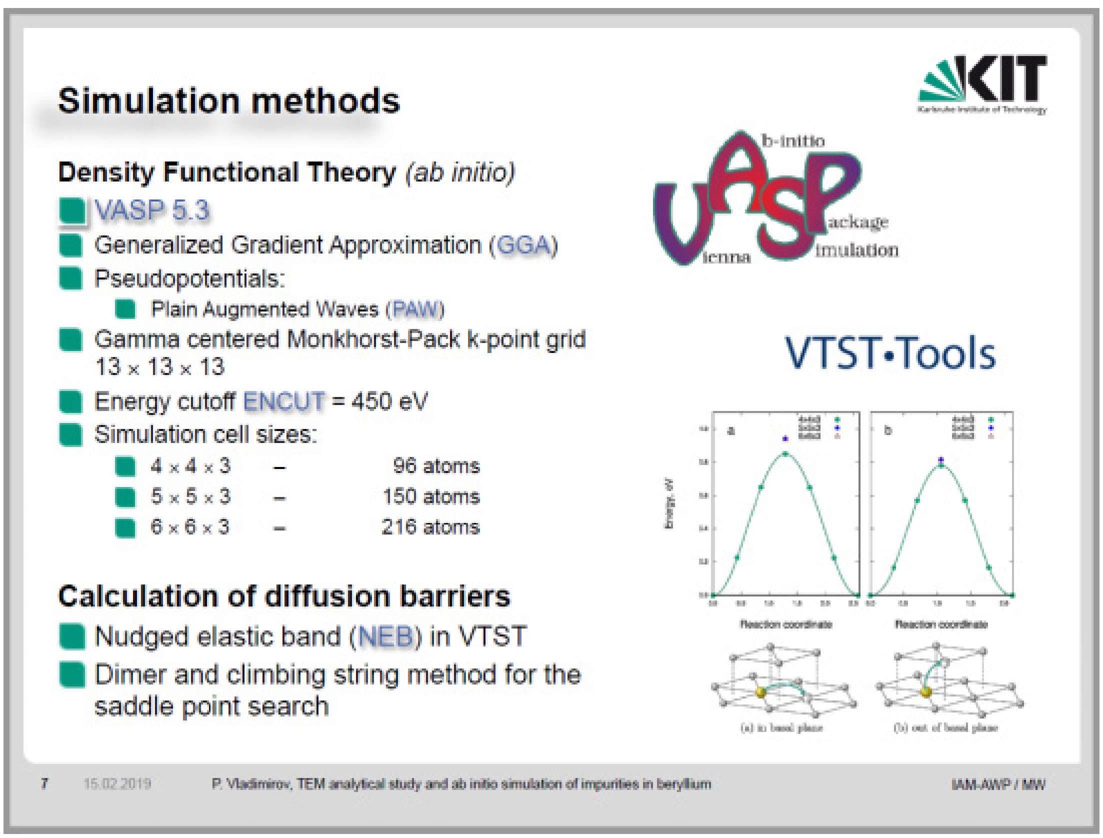
Grain boundary with misorientation
irradiated at 440 °C

5 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW

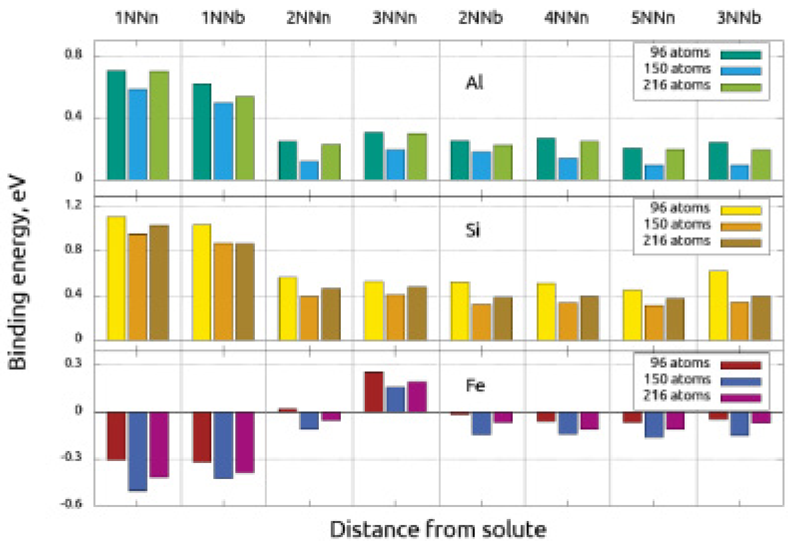

High-angle GB
KIT
a)
Al-Fe-Be
b)
Mg-Si
Fe-Cr
U-Fe
b)
c)
Grain b
irradiat
• Al-Fe-Be precipitates within grain & at GB
• Homogeneous segregation of Fe-Cr at GB
• Various single and multiphase precipitates found at GB
• Al-Cr-Be segregation at dislocation lines
• No precipitates were found at two highest T (560 & 650 °C)
5 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW

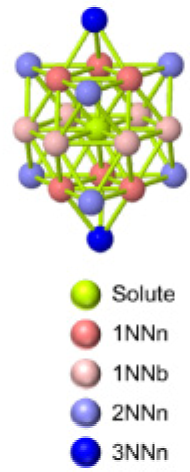

Dislocation loop
KIT
a)
200nm
b)
200nm
Al-Fe-Be
Fe-Cr
Mg-Si
U-Fe
Dislocation Loop:
habitus plane: prismatic type II plane $(11\bar{2}0)$.
$\{11\bar{2}0\}$
T_{irr}=370°C
6 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW


Dislocation loop
KIT
a)
b)
Al-Fe-Be
Fe-Cr
Mg-Si
U-Fe
• Edge dislocation loop pinned by precipitates
• Segregation of Al-Fe-Cr at the core loop
• Segregation is decorated by He-bubbles (no recombination of vacancies and interstitials)
• Poisoning of dislocation as PD sink and recombination place
6 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW


Simulation methods
KIT
Density Functional Theory (ab initio)
VASP 5.3
Generalized Gradient Approximation (GGA)
Pseudopotentials:
Plain Augmented Waves (PAW)
Gamma centered Monkhorst-Pack k-point grid 13 × 13 × 13
Energy cutoff ENCUT = 450 eV
Simulation cell sizes:
4 × 4 × 3 – 96 atoms
5 × 5 × 3 – 150 atoms
6 × 6 × 3 – 216 atoms
Calculation of diffusion barriers
Nudged elastic band (NEB) in VTST
Dimer and climbing string method for the saddle point search
VASP
b-initio
ackage
imulation
ienna
VTST•Tools
Reaction coordinate
Reaction coordinate
7 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW

Solute-vacancy interaction





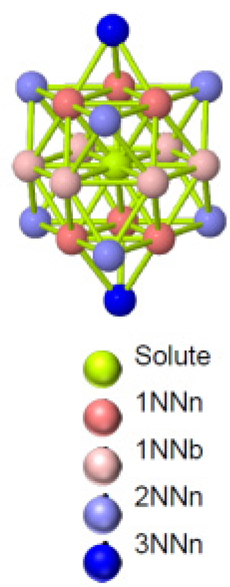
8 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW

Deformation around substitutional impurities

Atom	Radius*, pm	1NNn	1NNb	2NNn	3NNn
He	-	+1.7%	+3.9%	-1.6%	-0.7%
Be	105				
Si	110	2.6%	2.8%	0.3%	0.3%
Al	125	+3.9%	+3.6%	-0.3%	+0.3%
Fe	140	-0.04%	+0.06%	-0.6%	+0.3%

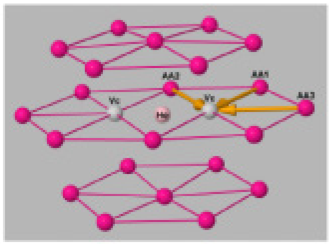
*Empirically measured atomic radii,
https://en.wikipedia.org/wiki/Atomic_radius#Empirically_measured_atomic_radius

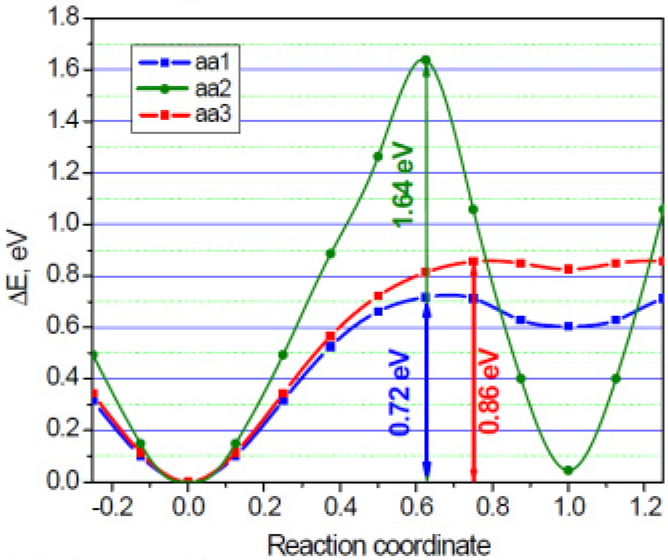
- He, Al, and Si act in Be as oversized solutes
- Fe induce very small deformations in Be



9 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW

He_1V_2 in-basal-plane migration path

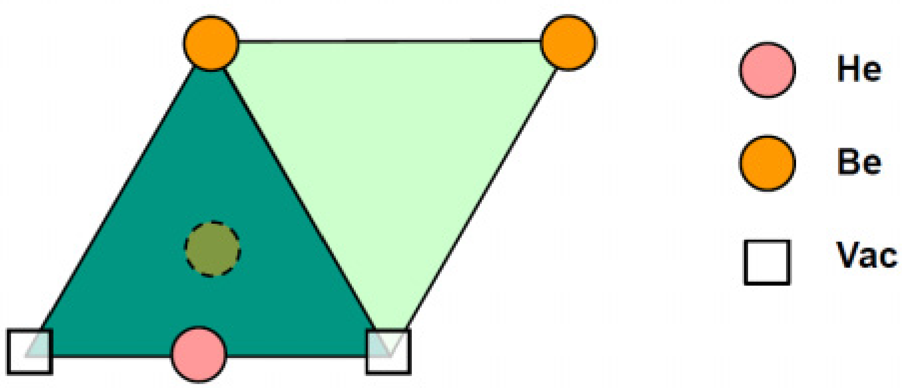




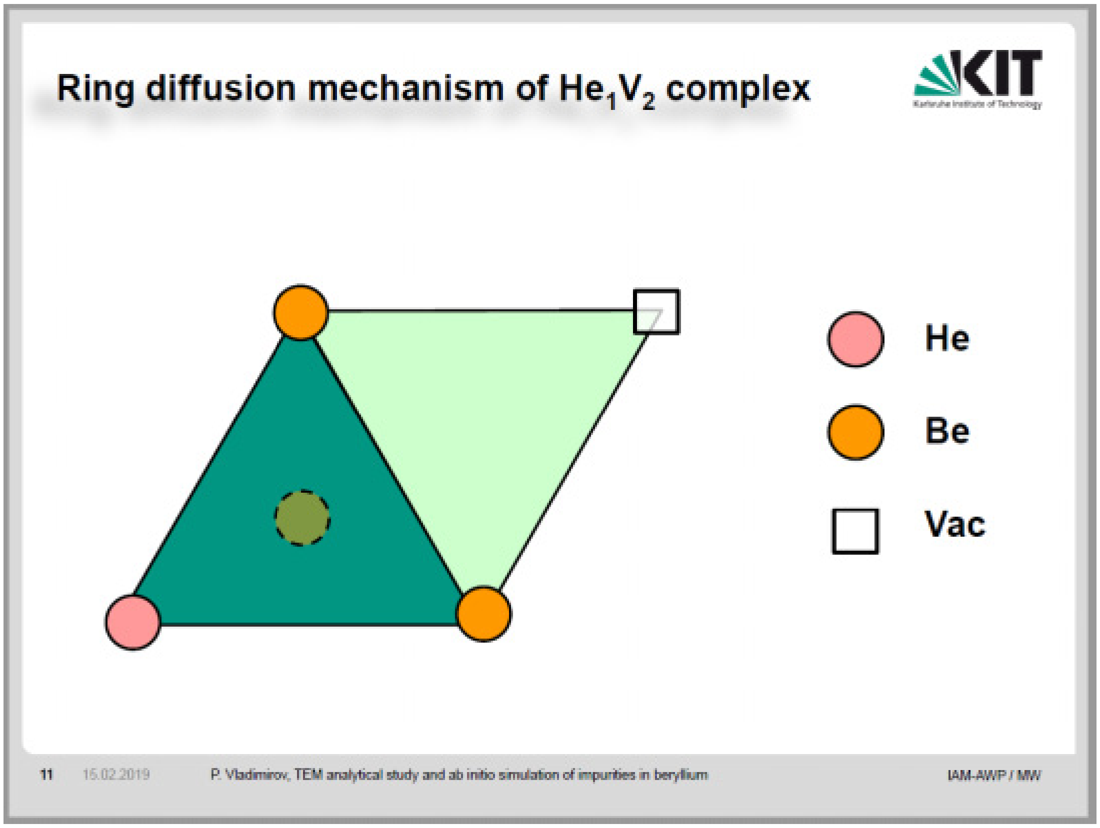
P.V. Vladimirov, A. Moeslang, *Ab initio static and molecular dynamics studies of helium behavior in beryllium*, J. Nucl. Mater. **442**(1–3) (2013) S694-S698

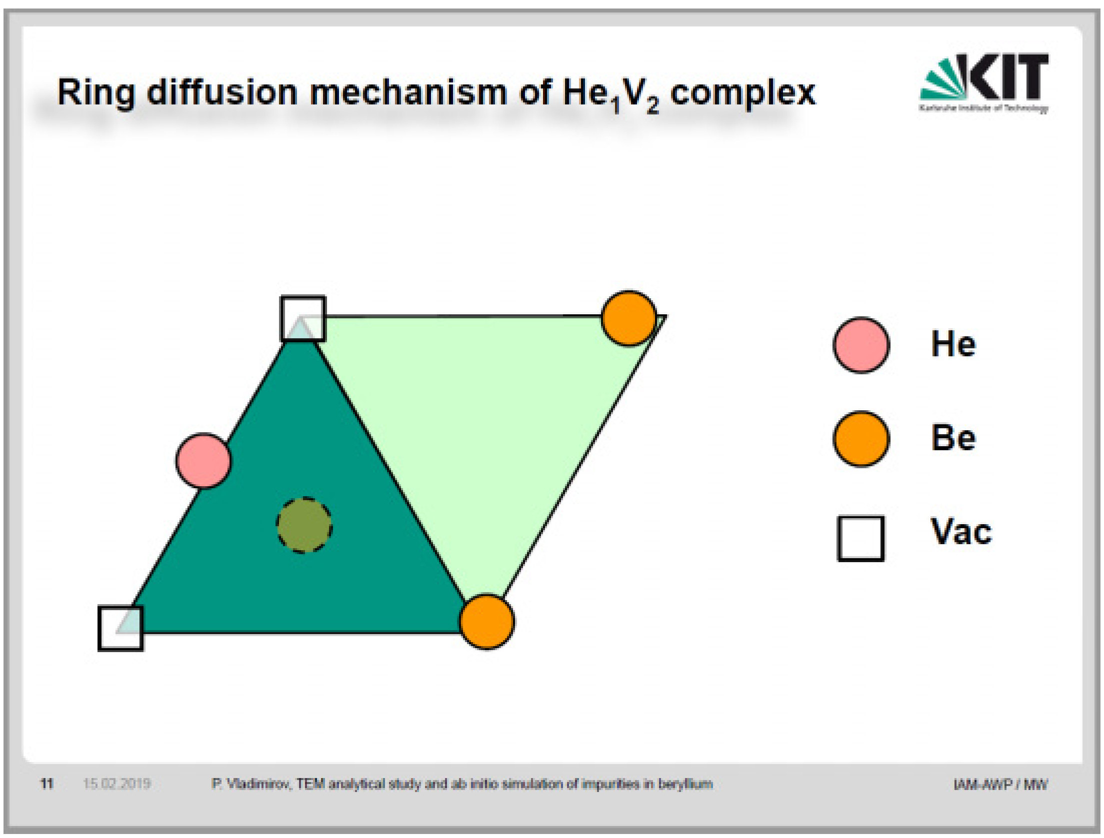
10 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW

Ring diffusion mechanism of He_1V_2 complex



11 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW


Ring diffusion mechanism of He_1V_2 complex
KIT
Karlsruhe Institute of Technology
He
Be
Vac
11
15.02.2019
P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium
IAM-AWP / MW


Ring diffusion mechanism of He_1V_2 complex
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Karlsruhe Institute of Technology
He
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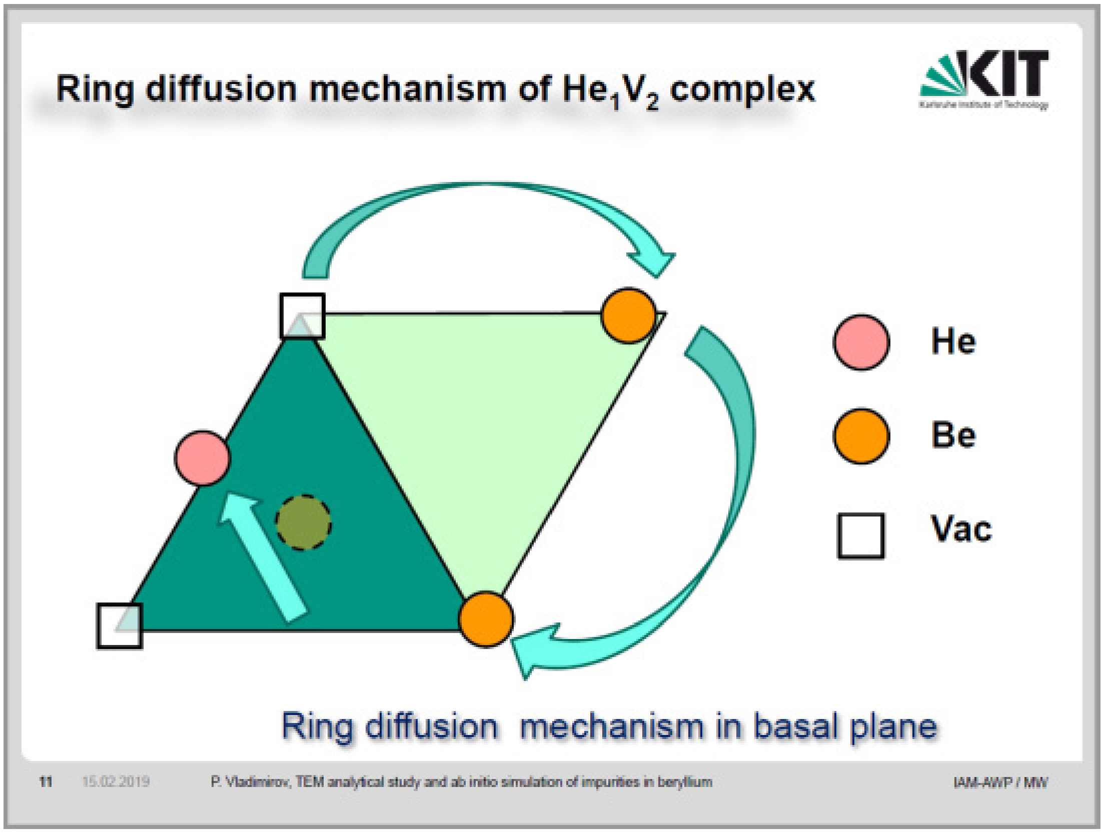
IAM-AWP / MW


Ring diffusion mechanism of He1V2 complex
KIT
Karlsruhe Institute of Technology
He
Be
Vac
Ring diffusion mechanism in basal plane
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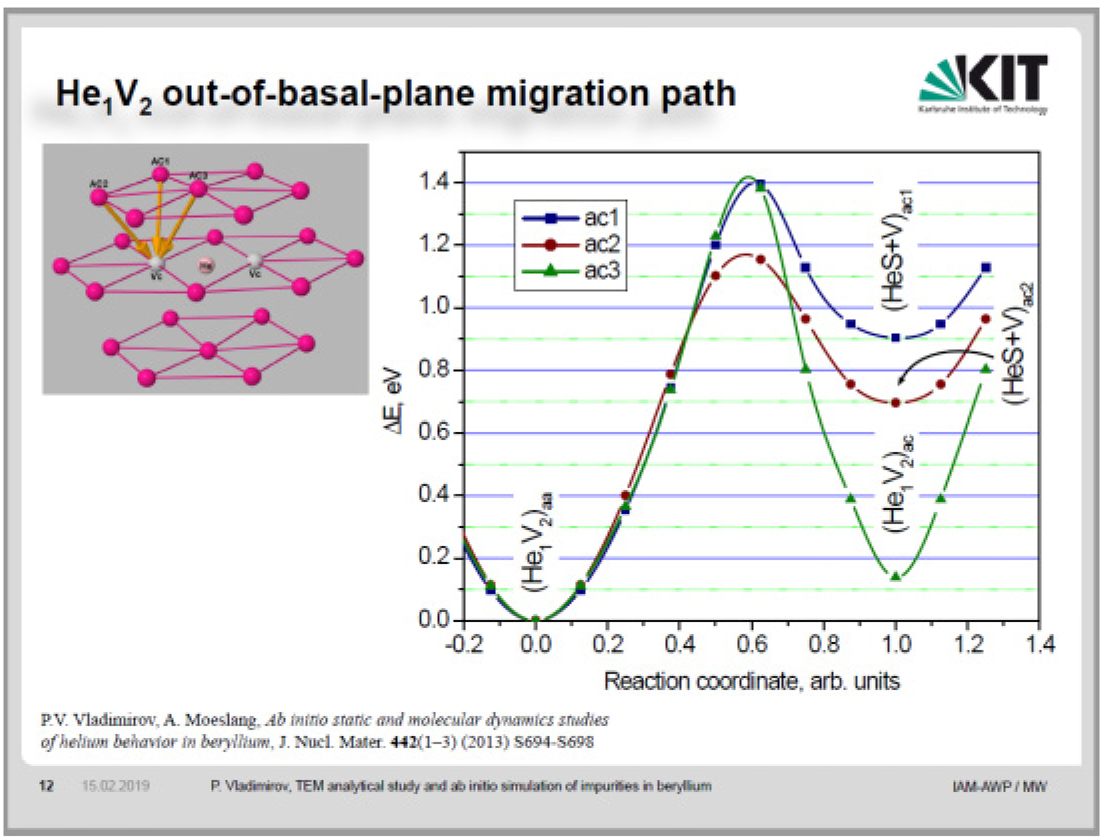
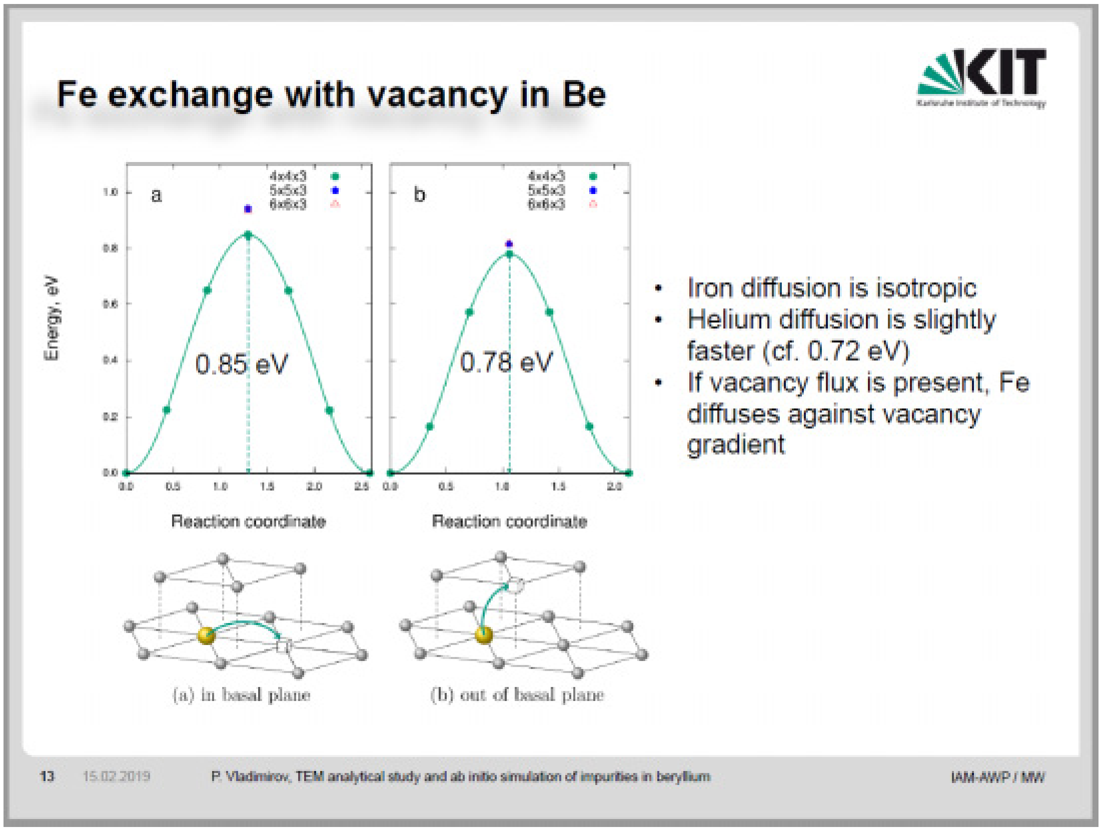
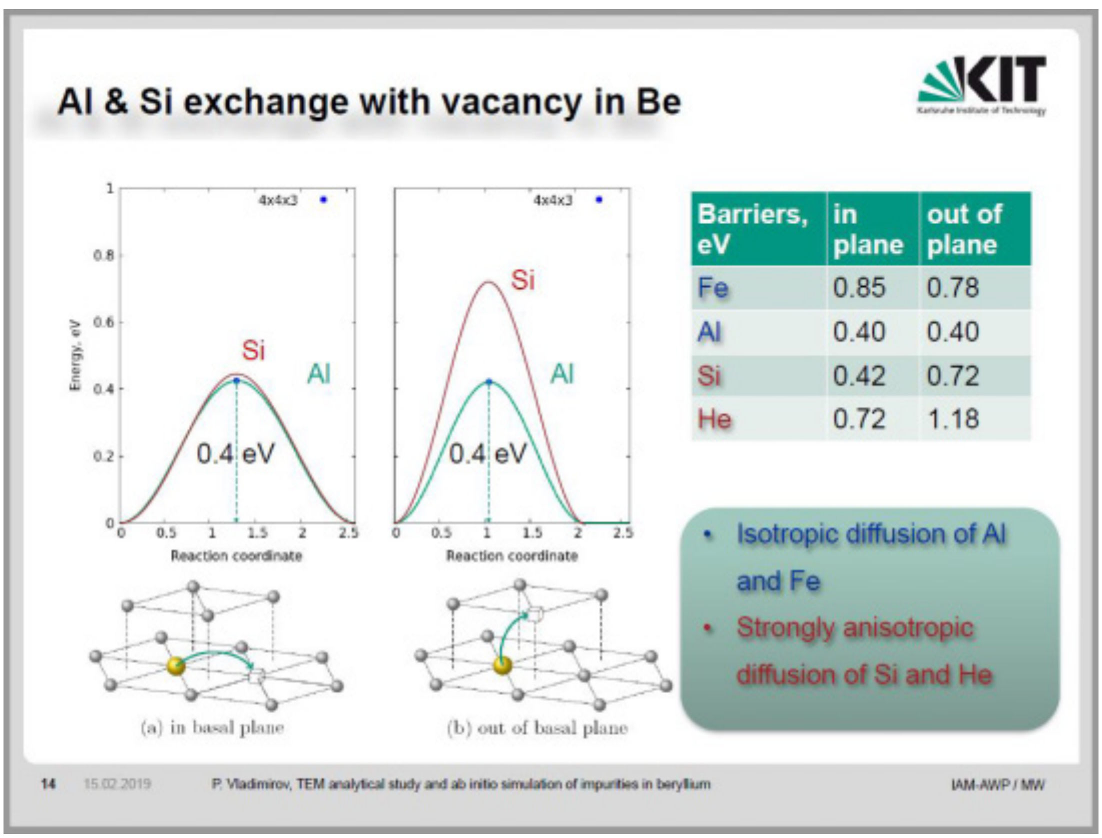
15.02.2019
P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium
IAM-AWP / MW


He1V2 out-of-basal-plane migration path
KIT
Karlsruhe Institute of Technology
ac1
ac2
ac3
(HeS+V)ac1
(HeS+V)ac2
(He1V2)ac
(He1V2)aa
ΔE, eV
Reaction coordinate, arb. units
P.V. Vladimirov, A. Moeslang, Ab initio static and molecular dynamics studies of helium behavior in beryllium, J. Nucl. Mater. 442(1–3) (2013) S694-S698
12
15.02.2019
P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium
IAM-AWP / MW





Barriers, eV	in plane	out of plane
Fe	0.85	0.78
Al	0.40	0.40
Si	0.42	0.72
He	0.72	1.18

Conclusions

- Interaction with vacancies
  - Al and Si attract vacancies, while Fe repel them
  - This coincides with deformation pattern around solute:
    - Substitutional Al and Si result in compression (up to 4%) around them
    - Substitutional Fe fits nicely into Be matrix (very small deformations)
- Diffusion
  - Fe diffusion is isotropic, barriers are like that for vacancy diffusion
  - Silicon is much faster in basal plane
  - Si and He diffusion is strongly anisotropic
- Consequences for diffusion mechanisms
  - Fe migrates against vacancy gradient
  - Si and Al migrate along vacancy gradient
  - Anisotropic diffusion of He, Al and Si should result in dependence of the denuded zone width from grain orientation w.r.t. grain boundary

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16 15.02.2019 P. Vladimirov, TEM analytical study and ab initio simulation of impurities in beryllium IAM-AWP / MW

Beryllium: A Review of Uses, Potential Health Effects and Impacts of Regulatory Activities

Marc Kolanz and Theodore Knudson

Materion Corporation, Mayfield Heights, Ohio, U.S.A.

Beryllium is a critical material with significant applications in the nuclear industry. This presentation will provide information on the significant uses of beryllium, the potential health effects of exposure to beryllium in the workplace and the impacts of regulatory activities in the US and EU.

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Materion Corporation
6070 Parkland Boulevard
Mayfield Heights, Ohio 44124
U.S.A.


MATERION
14th International Workshop on Beryllium Technology
Beryllium: A review of Uses, Potential Health Effects and Impacts of Regulatory Activities
Ted Knudson, MS, CIH
October 25, 2019


Beryllium - Why it is used?
Beryllium Metal
A naturally occurring element
Lightest non-reactive metal (lithium is lightest)
One third lighter than aluminum, stiffer than steel
Transparent to X-ray
Reflectivity (infrared enhancing)
Thermal management
Alloys (<2% Be)
Conductivity
Metal memory (lowest stress relaxation)
Strength (use less metal)
Corrosion resistance
Beryllium Oxide (BeO) Ceramics
Electrically non conductive (insulator)
Thermally conductive
4
Be
9.01218
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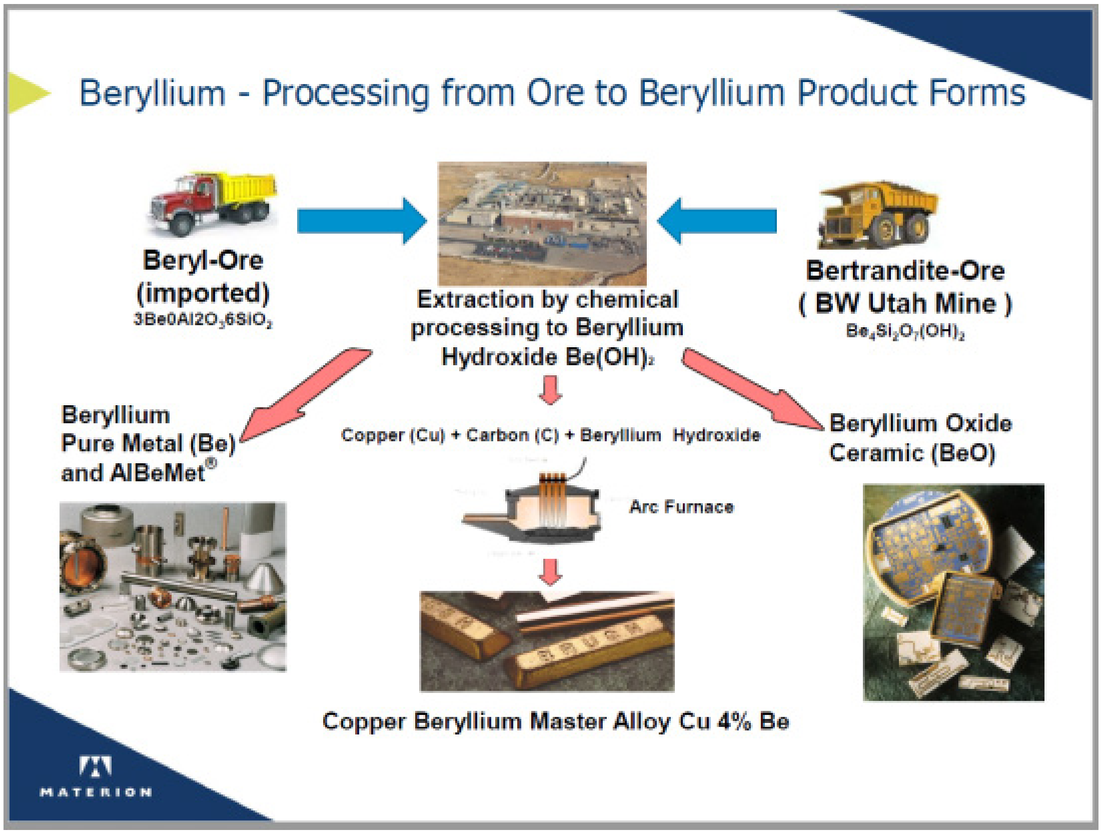
Beryllium - Processing from Ore to Beryllium Product Forms
Beryl-Ore
(imported)
3Be0Al2O3 6SiO2
Extraction by chemical processing to Beryllium Hydroxide Be(OH)2
Bertrandite-Ore
(BW Utah Mine)
Be4Si2O7(OH)2
Beryllium Pure Metal (Be) and AlBeMet®
Copper (Cu) + Carbon (C) + Beryllium Hydroxide
Arc Furnace
Beryllium Oxide Ceramic (BeO)
Copper Beryllium Master Alloy Cu 4% Be
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Beryllium in National Defense
Military fighter jets
Fasteners, equipment supports and structural components such as rudders and wing leading edges
Infrared sensors for fighter jet optical targeting, radar and navigation systems
F-15 Strike Eagle
F-16 Fighting Falcon
F-18 Super Hornet
F-22 Raptor
Unmanned surveillance and reconnaissance flight vehicles
Real time imagery
Global Hawk Unmanned Aerial Vehicle Optical Sensor
Attack and reconnaissance helicopters
Mast-mounted optical systems
Super Cobra, Apache and Kiowa Warrior
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Beryllium in National Defense

- **Guided missile defense system**
  - Infrared and optical sensors
    - Ground-based Interceptor
    - Launch detection satellites

- **Strategic missile defense systems**
  - Precision targeting
    - Trident submarine
    - Minuteman III ICBMs

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Beryllium in National Defense

- **Battle tanks**
  - Sight and fire control beryllium mirrors
    - M60 main battle tank
    - M1A2 Abrams main battle tank
- **Satellites and spacecraft defense support systems**
  - Structures, mechanisms, electronic housings, heat sinks and sensory equipment designed into virtually all military communication satellites

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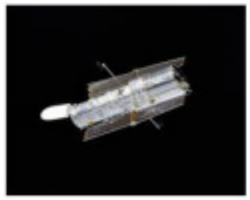
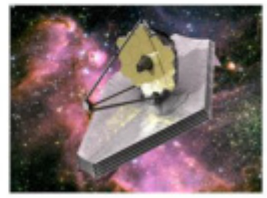
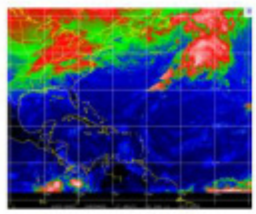
Beryllium in Space Discovery

- **Telescopes**
  - Hubble
  - Webb





- **Spacecraft**
  - Mars Rover
  - Space Shuttle
  - Cassini Orbiter
  - Optics in weather
  - Forecasting satellites



Webb Telescope Time-lapse

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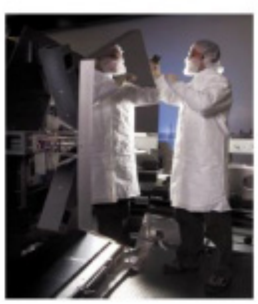
Beryllium Products

- Commercial guidance systems
- X-ray windows
- Optical instruments
- High-end audio



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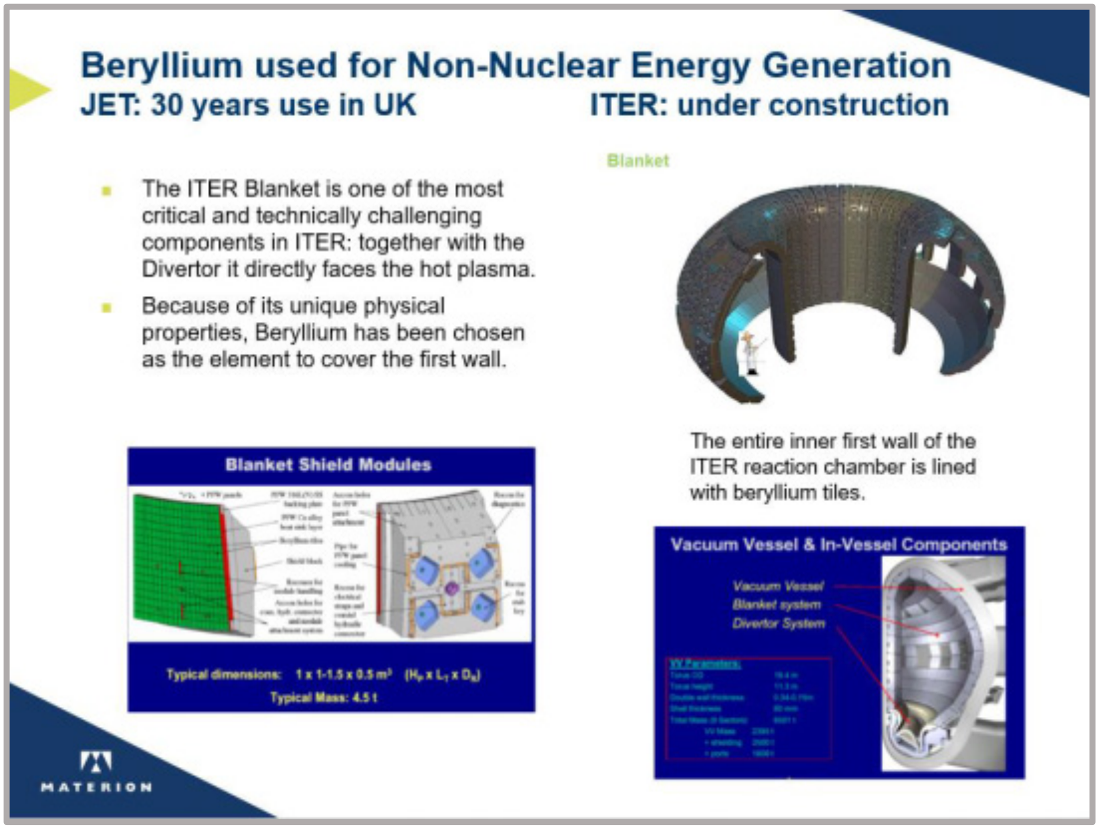
Beryllium used for Non-Nuclear Energy Generation
JET: 30 years use in UK
ITER: under construction
The ITER Blanket is one of the most critical and technically challenging components in ITER: together with the Divertor it directly faces the hot plasma.
Because of its unique physical properties, Beryllium has been chosen as the element to cover the first wall.
Blanket
The entire inner first wall of the ITER reaction chamber is lined with beryllium tiles.
Blanket Shield Modules
Typical Mass: 4.5 t
Vacuum Vessel & In-Vessel Components
Vacuum Vessel
Blanket system
Divertor System
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Beryllium-containing Alloys
Alloy Applications
Current carrying springs
Integrated Circuitry Sockets
Electrical and Electronic Connectors
Pressure Responsive Devices
RF and Coaxial Connectors
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Beryllium-containing Alloys
Alloy Applications
Plastic Injection Molds
Pressure Sensor Bellows
Fire Extinguisher Sprinkler Heads
Undersea Repeater Housings
Oilfield Drill Collars & Friction Bushings
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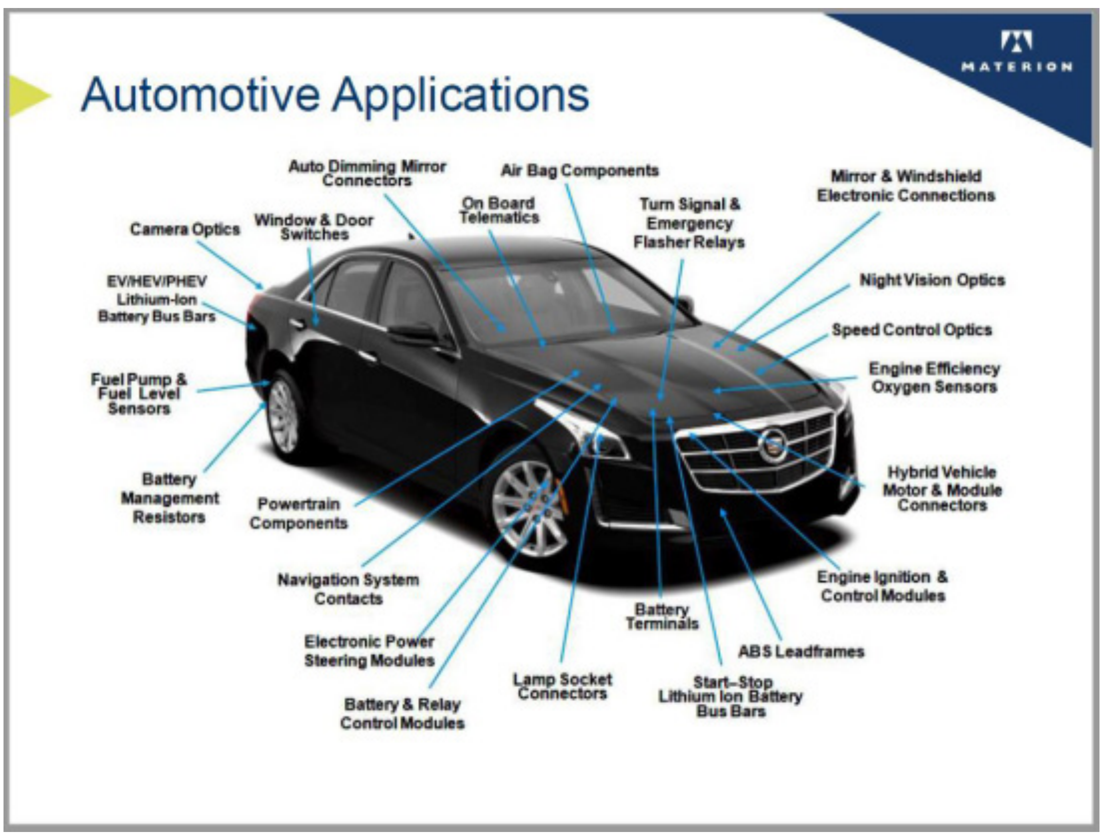
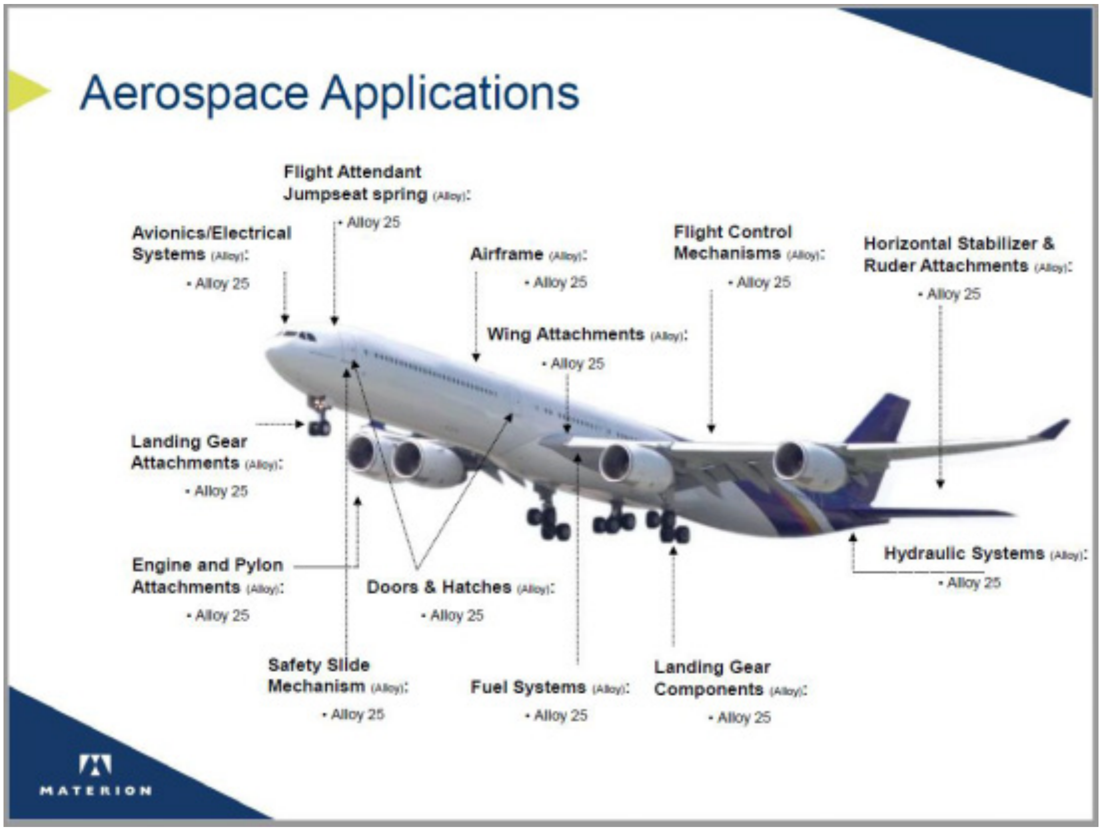
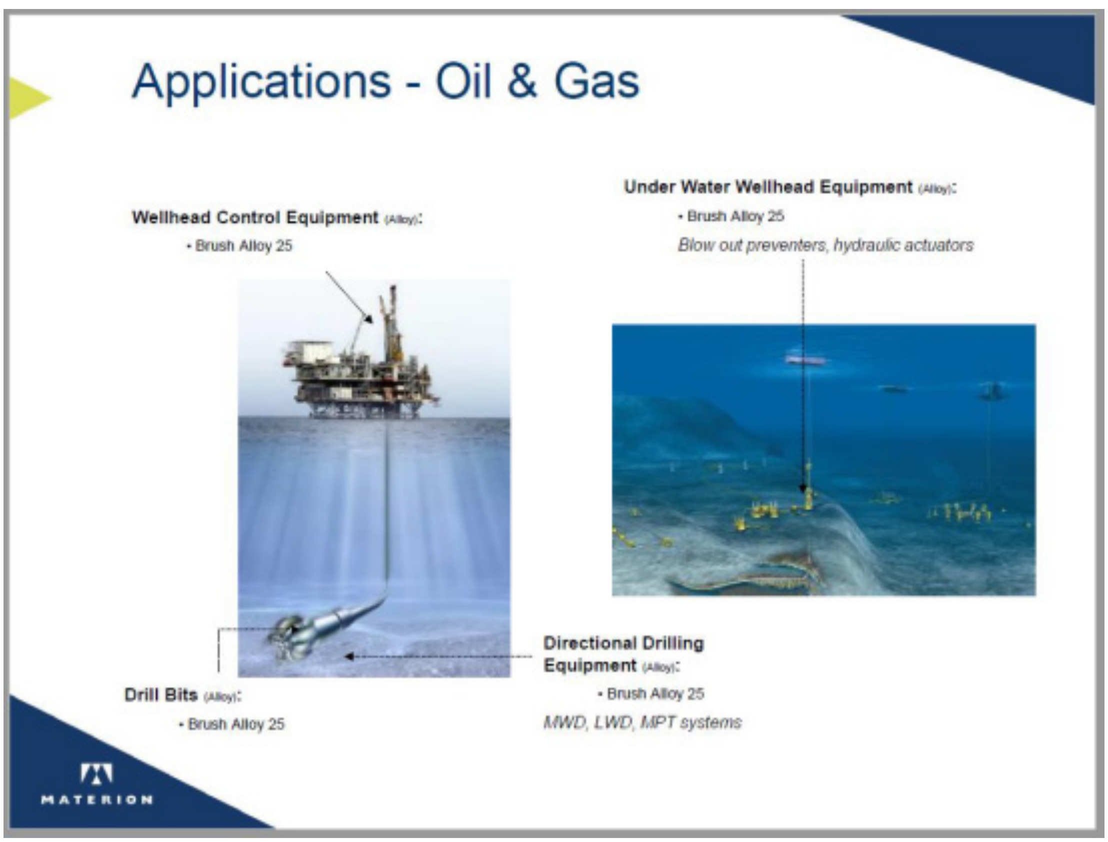

Automotive Applications
MATERION
Auto Dimming Mirror Connectors
Air Bag Components
Mirror & Windshield Electronic Connections
On Board Telematics
Turn Signal & Emergency Flasher Relays
Camera Optics
Window & Door Switches
EV/HEV/PHEV Lithium-Ion Battery Bus Bars
Night Vision Optics
Speed Control Optics
Engine Efficiency Oxygen Sensors
Fuel Pump & Fuel Level Sensors
Battery Management Resistors
Powertrain Components
Hybrid Vehicle Motor & Module Connectors
Navigation System Contacts
Engine Ignition & Control Modules
Battery Terminals
Electronic Power Steering Modules
ABS Leadframes
Lamp Socket Connectors
Start-Stop Lithium Ion Battery Bus Bars
Battery & Relay Control Modules


Aerospace Applications
Flight Attendant Jumpseat spring (Alloy):
• Alloy 25
Avionics/Electrical Systems (Alloy):
• Alloy 25
Airframe (Alloy):
• Alloy 25
Flight Control Mechanisms (Alloy):
• Alloy 25
Horizontal Stabilizer & Ruder Attachments (Alloy):
• Alloy 25
Wing Attachments (Alloy):
• Alloy 25
Landing Gear Attachments (Alloy):
• Alloy 25
Engine and Pylon Attachments (Alloy):
• Alloy 25
Doors & Hatches (Alloy):
• Alloy 25
Hydraulic Systems (Alloy):
• Alloy 25
Safety Slide Mechanism (Alloy):
• Alloy 25
Fuel Systems (Alloy):
• Alloy 25
Landing Gear Components (Alloy):
• Alloy 25
MATERION


Applications - Oil & Gas
Wellhead Control Equipment (Alloy):
• Brush Alloy 25
Under Water Wellhead Equipment (Alloy):
• Brush Alloy 25
Blow out preventers, hydraulic actuators
Directional Drilling Equipment (Alloy):
• Brush Alloy 25
MWD, LWD, MPT systems
Drill Bits (Alloy):
• Brush Alloy 25
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Beryllia Ceramics

- Beryllia Ceramic Applications
  - Laser Bores and Tubes
  - Substrate for High Speed Integrated Circuitry

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Critical / Strategic Material Classification

- US Department of Defense
  - Beryllium is declared the only strategic and critical material
    - Strategic Materials Protection Board, December 12, 2008, Office of the Under Secretary of Defense (Acquisition, Technology & Logistics), Deputy Under Secretary of Defense (Industrial Policy)
- European Union
  - Classified as a Critical Raw Material (CRM)

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Risks of Beryllium-containing Materials

- Beryllium, in **solid form** and as contained in **finished products**, present no special health risks.
- Most end users do not handle beryllium in ways which generate dust, mist or fume.
- However, like many industrial materials, beryllium presents a health risk if handled improperly.
- The inhalation of beryllium-containing dust, mist or fume can cause a serious lung condition in some individuals.

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Potential Health Effects

Chronic Beryllium Disease (CBD)
Lung cancer
Acute Beryllium Disease (ABD)
Skin Effects

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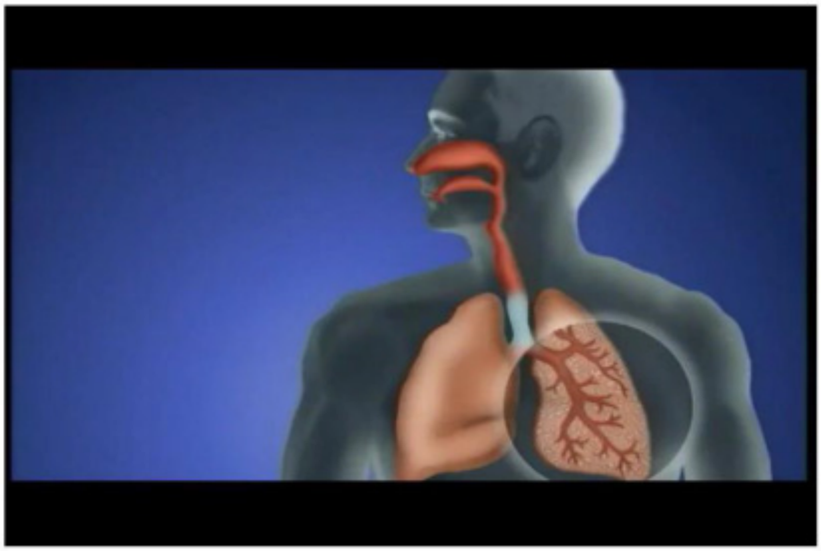
Chronic Beryllium Disease (CBD)



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Chronic Beryllium Disease (CBD)

- Diagnostic criteria prior to late 1980s
  - Individuals exhibit symptoms
  - Changes in chest x-ray
  - Reduced lung function
- Diagnostic criteria after late 1980s
  - Confirmed sensitization to beryllium
  - Presence of pulmonary granulomas
  - Does not require presentation of symptoms
- Surveillance using the new criteria has resulted in increased numbers of persons identified with CBD, most of whom do not have symptoms or any material impairment of health.

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Chronic Beryllium Disease

- Granulomas reduce gas diffusion
- Fibrosis shrinks lungs
- Fibrosis stiffens lungs
- Fibrosis narrows airways

Causing

- Shortness of breath, dry cough, wheeze, fatigue, night sweats
- The extent of these symptoms is variable

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BLPT (Beryllium Lymphocyte Proliferation Test)

- Scientists are continuing to pursue further understanding of the implications of a positive test.
- Test results are not always consistent. Sometimes, positive results are not confirmed by subsequent tests at the same or different laboratories.
- BeS exists in unexposed individuals, up to 1%.
- BeS is not associated with any adverse health effects: it's a marker of exposure in sensitive individuals. BeS is a population risk factor for CBD, but it's predictive value for individual's is uncertain.

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Lung cancer from beryllium

Does research suggest that beryllium could increase the risk of lung cancer (in any chemical form, or at any dose)?

- **Yes,** workers at older plants hired before 1955.
- IARC (International Agency for Research on Cancer) classifies beryllium and beryllium compounds (jointly) as a – Known Human Carcinogen
- The USEPA classifies beryllium and beryllium compounds as a - Probable Human Carcinogen

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Lung cancer from beryllium

In order to evaluate cancer risk, MBI followed the advice of an expert scientific panel and commissioned Dr. Boffetta (world leading cancer risk expert).

His 2014/2015 studies are the largest ever performed, covering 16,115 workers at 15 plants, employed over an 85 year period and followed through 2011.

- Increased risk only for workers exposed at two oldest plants prior to 1955 with very high soluble + insoluble exposures
- No overall increased lung cancer risk 1925-2008
- No increased cancer risk at 8 insoluble only facilities
- No increase in lung cancer for workers exposed to insoluble or soluble Be since 1955

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Dr. Boffetta's findings:

Facility	Lung cancer increased risk?
Elmore	NO
Tucson	NO
Shoemakersville	NO
Distribution Centers	NO
Perkins	NO
St. Clair	NO
Chester	NO
Delta	NO
Luckey	NO
Historical Lorain	YES

Facility	SMR	95% confidence
Elmore	0.75	0.59 – 0.94

SMR is the ratio of actual to expected disease

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Why was there risk prior to 1955?

Photos of Historical Lorain Ohio Plant:

Increased lung cancer occurred in Brush Wellman old Lorain, and Cabot Corp. Reading plant where exposures were >10s µg/m^3, even into thousands

tending the furnace

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Why was there risk prior to 1955?

Photos of Historical Lorain Ohio Plant:

Increased lung cancer occurred in Brush Wellman old Lorain, and Cabot Corp. Reading plant where exposures were >10s μg/m^3, even into thousands

sulfating operation

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Acute beryllium disease

Howard Van Ordstrand, Cleveland Clinic, first reported acute beryllium disease in the United States in 1943

Rapid onset **inflammatory pneumonitis** of the lung from inhaling **high concentrations** of beryllium salts, such as beryllium fluoride, causes severe dyspnea

Potential is only due to high exposure to **soluble salts of beryllium** (>100μg/m^3) **at primary producer** during extraction of beryllium

A historical artifact - 3 cases since 1960, last case > 30 years ago

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Beryllium skin disease

Irritant/allergic skin rash or ulcers from contact with beryllium salts

Skin exposure to beryllium salts **can cause allergic sensitization**

Granulomas may be chronic if particles are imbedded

No effects from solid beryllium or copper-beryllium metal

Beryllium skin nodules

Courtesy of Lee S Newman, MD, MA

UpToDate

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Regulatory Impacts

- Product End-of-Life Management
  - RoHS, WEEE, ELV
- REACH
- Occupational Exposure Limits (OEL)
  - US OSHA Beryllium Standard
  - EU Binding OEL
- Hazardous Waste
- Clean Air Act
- Drinking Water Standard
- Wastewater Regulations

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Beryllium Science and Technology (BeST)

- BeST – Beryllium Industry Association in EU
- Directorate (Ridens Public Affairs) located in Brussels, Belgium
- Members
  - Materion Corporation
  - NGK Berylco France
  - Tropag
- Associate Members
  - Schmeltzmetall AG
  - CBL
  - UK Atomic Energy Authority

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Product End of Life Management

Beryllium and beryllium-containing materials were not included in any end-of-life requirements or restrictions in European Union Directives:

- on the restriction of the use of certain hazardous substances in electrical and electronic equipment (RoHS) (2011/65/EU), 2011/65/EU (RoHS 2) and 2015/863/EU
- on waste electrical and electronic equipment (WEEE) (2002/96/EC); or,
- on End-of-Life Vehicles (ELV) (2000/53/EC and 2002/525/EC).

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Product End of Life Management

Beryllium is one of seven materials under review as part of the European Commission (EC) "Study to support the review of the list of restricted substances and to assess a new exemption request under RoHS 2" that began in January 2018.

During the study consultation conducted by the EC consultant, BeST provided information as to the importance of beryllium in Electrical and Electronic Equipment (EEE), the minimal risk in the use of beryllium in EEE and the consequences for restricting beryllium in EEE.

The EC consultant's assessment of beryllium was completed and published on September 26, 2019 and based on the information provided during the assessment, the EC consultant recommended that beryllium **not be included** on the RoHS restricted substance list.

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REACH

Registration, **E**valuation and **A**uthorization of **Ch**emicals (REACH)

- Beryllium metal and beryllium oxide are registered.
- Massive forms of beryllium metal, beryllium alloys and beryllia ceramic are classified as "articles" and as such do not have to be registered under REACH.
- CoRAP and RMOA conducted for beryllium by German BAuA:
  - Final recommendation was **not** to classify as an Substance of Very High Concern (SVHC), for industry to develop an Voluntary Product Stewardship Program and for EU to identify a binding Occupational Exposure Limit OEL) for beryllium.

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Occupational Exposure Limits

OSHA Beryllium Standard (29 CFR 1910.1024)

- The Action Level (AL) is 0.1 μg/m^3, 8-hour TWA.
- The Permissible Exposure Limit (PEL) is 0.2 μg/m^3,8-hour TWA.
- The Short Term Exposure Limit (STEL) is 2 μg/m^3 determined over a 15 minute period.
- Exposures at or above these levels prompt implementation of other provisions.

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Occupational Exposure Limits

The provisions of the OSHA Beryllium Standard (29 CFR 1910.1024) include:

- Scope
- Permissible Exposure Limits
- Exposure Monitoring
- Beryllium Work and Regulated Areas
- Methods of Compliance
  - Written Exposure Control Plan
  - Engineering and Work Practice Controls
- Respiratory Protection
- Protective Clothing and Equipment
- Hygiene Areas and Practices
- Housekeeping
- Medical Surveillance
- Medical Removal
- Communication of Hazards
- Recordkeeping

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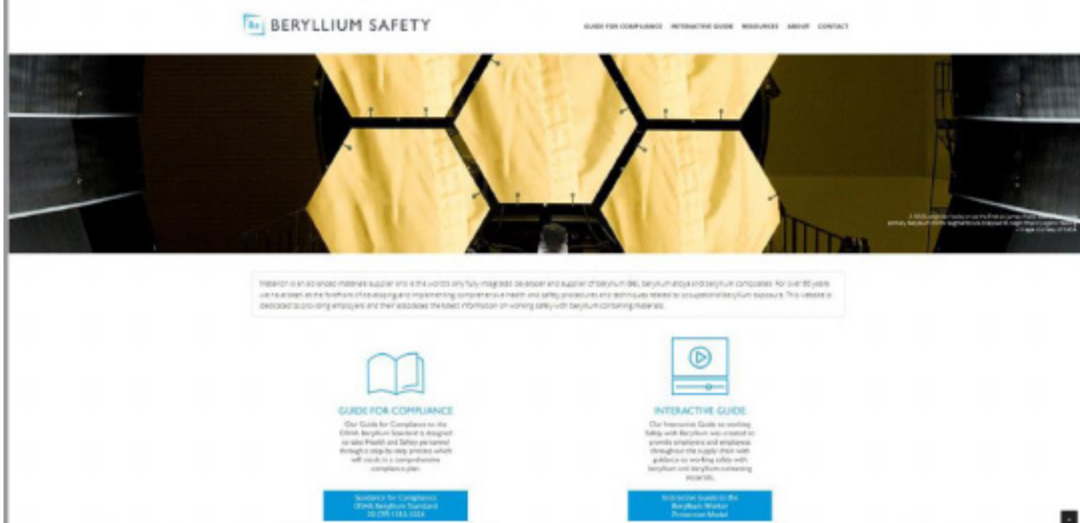
www.berylliumsafety.com Website



www.berylliumsafety.com

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Occupational Exposure Limits

- After several years of study and consideration, the EU recently set a new, binding worker protection standard for beryllium under the Carcinogens and Mutagens Directive (CMD).
- The new Occupational Exposure Limit (OEL) is as follows:
  - 0.6 µg/m^3, Inhalable fraction, 8-hour TWA for a 7-year transitional period (until July 11, 2026)
  - 0.2 µg/m^3, Inhalable fraction, 8-hour TWA after transitional period
- Transitional OEL is equivalent to new OSHA PEL

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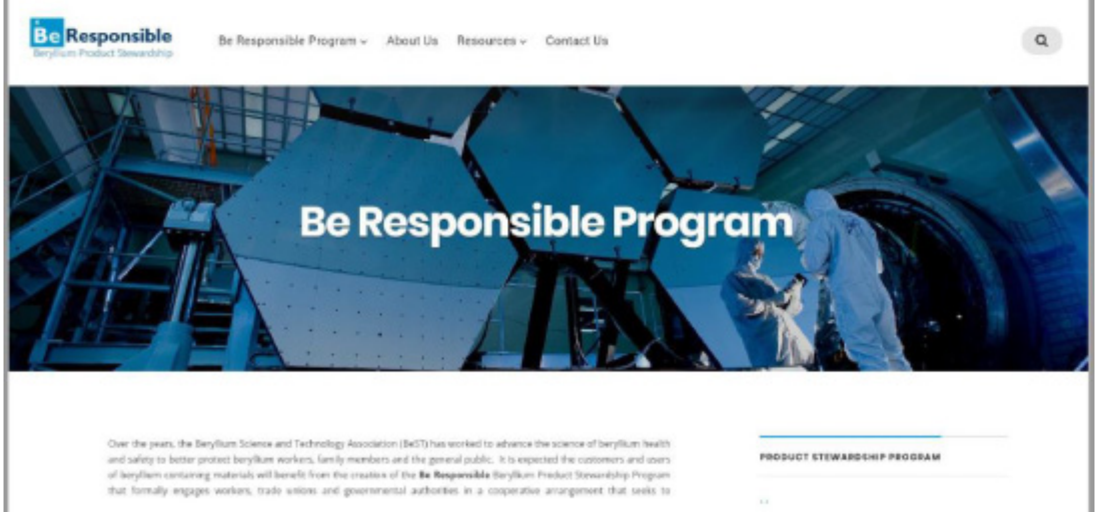
www.berylliumsafety.eu Website



www.berylliumsafety.eu

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Beryllium-Containing Scrap

- Not a hazardous waste under US Federal law.
- An insoluble material with low potential for environmental decomposition.
- A valuable resource recycled by the primary producer.
- Safely disposed of in accordance with state and local laws.

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EPA Hazardous Waste Regulations

Only one form of beryllium is listed as a hazardous waste under Federal law -- commercial beryllium metal powder

Listed as "Beryllium powder - PO15" at 40 CFR 261.33(e)

This definition very narrowly applies only to the discarded commercial metal powder product.

It does not apply to beryllium-containing filtering media, air pollution control dust, wastewater, machining chips or any other manufacturing by-products or other forms of beryllium, powder, or otherwise.

Since May 1990, recycling is the permissible disposal method for beryllium P015 waste. (Materion Brush Inc. recycling capabilities are available to the general industry)

Direct land filling without treatment is prohibited.

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Clean Air Act

Does not apply to facilities handling any material containing 0.1% or less beryllium.

Does not apply to facilities machining alloys containing 5% or less beryllium.

EPA National Emission Standard for Hazardous Air Pollutants (40 CFR 61, subpart C)

- 0.01 micrograms beryllium per cubic meter 30-day average
- As measured utilizing ambient air monitors
- 10 grams per 24-hour total site emission limit

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EPA - Drinking Water Standard

4 µg/l (micrograms per liter)

(Same as 4 parts per billion)

EPA - Drinking Water Standard

Beryllium detected at 277 of 4,346 non-hazardous groundwater sites with an average concentration of 13.6 µg/l (EPA 2000)

Where detected, average concentration in drinking water = 0.19 µg/l (ATSDR 1993)

Range = 0.01 - 1.22 µg/l (ATSDR 1993)

Wastewater Regulations

Vary considerably!

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Comparison of collection efficiencies of Whatman n°1 filter paper and Ghost Wipes for loose beryllium surface contamination

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Abstract

The Joint European Torus (JET) is the world's largest fusion research reactor. Beryllium has been used in JET since 1989. Up to 3 tonnes of beryllium have been installed in the torus as an evaporated deposit and in form of solid components. About 12 tonnes of beryllium will be installed in ITER's First Wall. Although the form of beryllium used at JET is predominantly beryllium metal, some beryllium dust can be expected due to erosion of the first wall surfaces and minor machining that has been carried out. Additionally, exposure can occur in areas where maintenance and decontamination of the components are performed, respiratory protective equipment is cleaned and in beryllium handling areas (e.g. BeHF).

As routes of exposure to beryllium are inhalation and dermal contact, both airborne and surface contamination controls are required. Currently at JET, the level of beryllium surface contamination is monitored by pursuing regular smears (weekly, monthly, quarterly or yearly) using dry sampling methods (Whatman n°1 filter paper).

The purpose of this paper is to compare results of beryllium smear samples analysis obtained by using Ghost Wipes™ and Whatman filter paper. The objective is to determine the most effective method for dust collection and hence improve workplace contamination control and worker protection for purposes of ITER.

Keywords: beryllium, surface sampling, sampling medium, beryllium analysis

1. Introduction

Beryllium is a naturally occurring element that can be found in nature together with other elements. It is lightweight metal with specific stiffness 6 times higher than the specific stiffness of steel [1]. Its excellent physical and nuclear properties make this element very attractive for use in both nuclear and non-nuclear applications [2]. In nuclear fusion, beryllium will be used as plasma-facing material (PFM) and neutron multiplier in Test Blanket Modules (TBM) in ITER [3][4].

In JET, beryllium has been used since 1989 in form of solid components and as an evaporated deposit on the First Wall [5]. Machine operations can cause erosion of the First Wall surfaces causing the generation of dust [5]. Unfortunately, exposure to beryllium dust or fumes through inhalation or dermal exposure can lead to acute and chronic beryllium disease (CBD) [6][7][8]. Symptoms of CBD can appear 10-15 years after the exposure to small amount of beryllium (0.5µg/m^3) [6]. Therefore, it is of great importance to have certain controls of exposure in place in order to protect workers who are at risk of exposure to beryllium. Such measures include:

- Dedicated facilities to each segment of beryllium utilisation with a containment de-sign which will prevent spread of contamination outside of the facility and ventilation which will change the air frequently so that airborne beryllium contamination is minimized.
- Personal and respiratory protective equipment, such as coveralls, gloves, overshoes, pressurized suits, full face masks, hoods and disposable masks. Depending on the task, worker is required to wear suitable personal and respiratory protective equipment.
- Training of the personnel for work with beryllium, medical examination of the workers, guidelines and rules while working with beryllium, designation of the areas in which beryllium can be found, depending on the airborne and surface contamination levels which can be detected (depending on the work).
- Routine airborne and surface beryllium monitoring in all beryllium designated areas and personal exposure assessment.

1.1 Surface Beryllium Contamination Monitoring as a means of Exposure Control

Currently, there is no commercial method for real-time beryllium monitoring. Therefore, any higher levels of beryllium, to which workers might have been exposed, are detected only after the exposure from the air and surface samples taken in the area. This implies that it is not possible to prevent such exposures in time to stop with the work which could potentially harm the personnel.

However, routine sampling and sample analysis can give us beryllium contamination levels which are usual for the area and/or type of work. These levels can help in defining the required personal and/or respiratory protective equipment. Any unusual increase in beryllium contamination noticed in routine samples, would require area to be checked the source of contamination to be identified prior to any exposure to present beryllium contamination levels.

In order to react early when a change in contamination levels appears, it is important to have accurate surface sampling. The accurate surface sampling depends on several things, such as sampling technique, type of surface sampled, and medium used for sampling. There are a few surface sampling methods that can be used [9]. The method used in this work is wipe sampling. Wipe sampling consists of dry or moistened medium applied in a defined motion and direction pattern with constant pressure on the surface. In the case of moistened wipes, wetting agents can be deionized water, alcohol, mild detergents, etc. [9].

In this paper, sampling media are compared regarding their respective collection efficiencies. Chosen media, surface sampled, sampling technique and other factors which can compromise sampling are described in chapter 2. The results obtained are presented and discussed in chapter 3. A conclusion is found in chapter 4.

2. Experimental Overview

2.1 Experiment Objectives and Description

The objective of this work is to compare collection efficiencies of two different media. The media chosen for smearing tests are Whatman n°1 filter paper and Ghost Wipes™. Whatman n°1 filter paper [10] is currently used at JET as a sampling medium for loose beryllium and tritium contamination. The Ghost wipes [11] are considered for use in ITER as a sampling medium for loose beryllium contamination. The basic properties of each sampling medium are presented in the table below:

Table 2.1: Basic properties of chosen sampling media [10][11]

Medium	Surface Area (cm^2)	Wetting Agent
Whatman n°1 Filter Paper	23.75	None (dry filter paper)
Ghost Wipes	225	Yes (de-ionized water)

2.1.1 Surface Sampling Technique

Samples were taken following standard procedure by the Health Physics Group at UK Atomic Energy Authority site in Culham for wipe sampling [12]. Depending on the surface shape and size, surface sampling was performed by wiping the surface in a circular pattern and Z- or S-pattern (see Fig. 2.1).

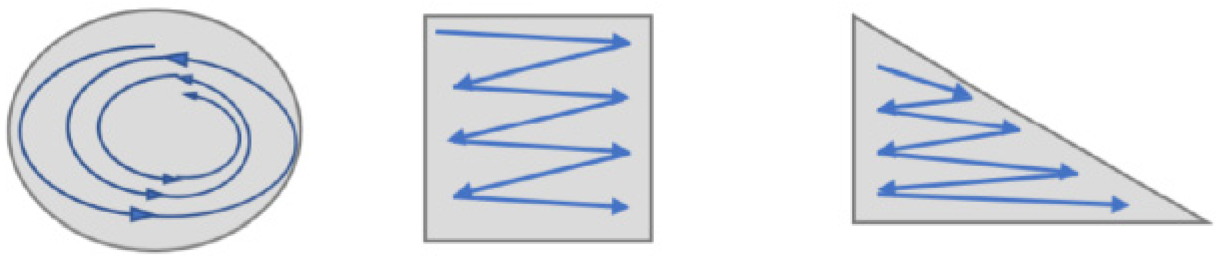

Figure 2.1: Surface wiping patterns - circular (left) or Z-pattern (center and right)

Wiping of the surface consists of applying chosen medium (in this case Whatman n°1 or Ghost Wipe) in a pre-defined method of motion and direction with applied constant pressure onto a surface area of interest. Prior to wiping, technicians would be instructed on previously mentioned technique. It is of great importance that every smear pair (Whatman n°1-Ghost Wipe) was taken applying exactly the same technique, so that results obtained by the samples analysis are comparable. There are many factors in sampling technique which can compromise the sampling and result in producing less accurate results. These factors will be described in Section 2.3.

2.1.2 Sample Analysis Method

Analysis of beryllium samples taken with Whatman n°1 filter paper and Ghost Wipes was performed using UV fluorescence assay, in accordance with NIOSH 9110 procedure [13]. Preparation of beryllium samples was performed according to NIOSH 9110 with minor modifications defined in a work procedure for beryllium samples preparation [14]. Brief representation of preparation and analysis method is shown in Fig. 2.2.

Prior to performing the experiment on collection efficiencies of Whatman n°1 and Ghost Wipes, the preparation and analysis method using UV fluorescence assay was validated for Ghost Wipes. In the validation report [15], it is shown that applying preparation and analysis method used on Whatman filter paper, would recover beryllium collected by Ghost Wipes, even with minor modifications, such as the amount of dissolution solution added during the preparation phase or the vials used during preparation. These minor changes compared to the standard Health Physics procedure are required due to the larger size of Ghost Wipes relative to the Whatman n°1 filter paper, which is a standard wiping medium used on the Culham site.

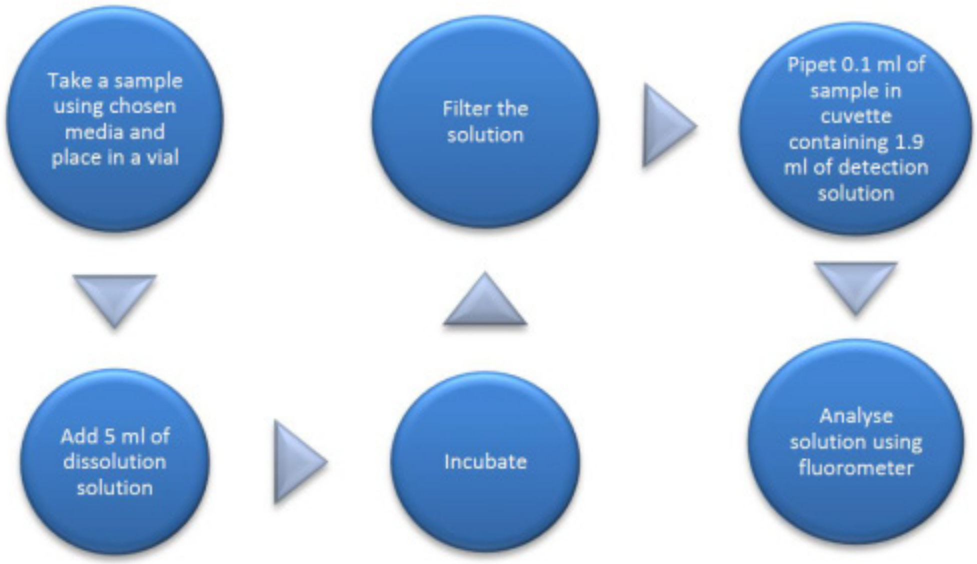

Figure 2.2: Sample preparation and analysis procedure at the UKAEA Culham site

2.2 Stages of the Experiment

The smearing tests were performed in two different environments, hence there are two stages of the experiment. The first stage of the experiment was in the laboratory environment, under ideal conditions. Four glass plates were spiked with a known amount of beryllium and wiped with both media. The results obtained after the analysis of the samples were used to estimate the efficiency in the laboratory conditions. The efficiencies of different media were analysed and compared.

The second stage of the experiment was in the real environment. In this case, the real environment is the Materials Detritiation Facility (MDF) on the Culham site. A surface from a fume cupboard, which is used for intermediate level waste sorting, was used for testing and comparing wiping media. The surface was divided into more smaller areas and each one was wiped with a different medium. As the true levels of beryllium contamination on the surface are unknown, the efficiency was not estimated the same way as it was in the case of laboratory environment. Rather the amount of beryllium from each smear was compared with other smears.

2.2.1 Stage 1 – Lab Environment

The chosen area surface was glass. Glass was chosen because it is not expected that it will absorb any beryllium. Four identical glass plates, shown in the Fig. 2.3, were used in the experiment. Plates were tested for any residue beryllium contamination prior to spiking any beryllium on them.

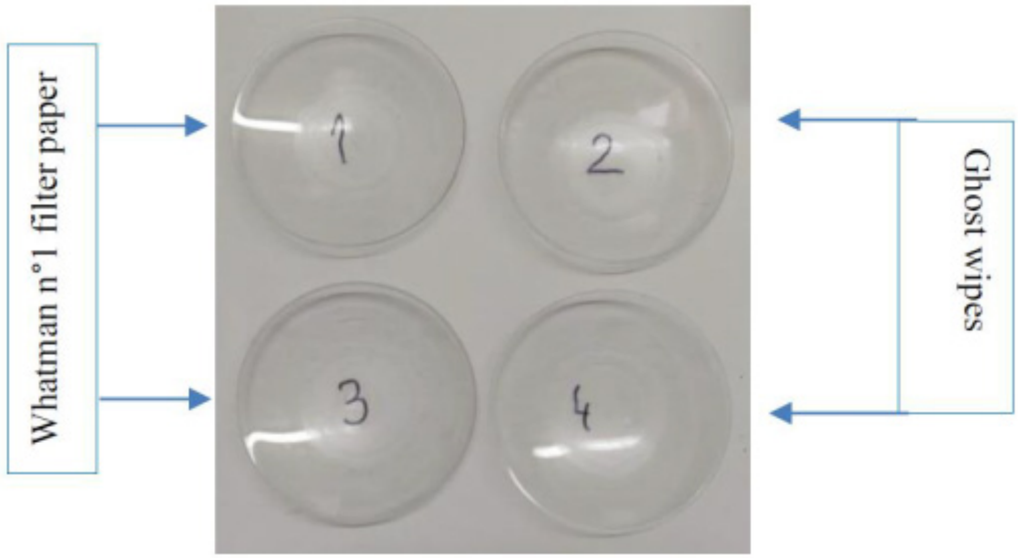

Figure 2.3: Four glass plates used for experiment stage 1 - laboratory environment

Each pair was spiked with a different amount of beryllium liquid standard solution. The beryllium liquid standard solutions used for this experiment are classified as certified reference materials (CRMs). The beryllium standards used are: 0PPB, 40PPB, 200PPB and 800PPB. In Table 2.2, the amount of beryllium spiked on the plates is presented.

Table 2.2: The amounts of spiked beryllium

Beryllium Liquid Standard (PPB)	Plate	Amount of Spiked Beryllium in the Liquid Standard (ml)	Expected Amount of Be in the Sample (g)
0	1	1	0
	2		
	3	2	0
	4		
40	1	1	0.04
	2		
	3	2	0.08
	4		
200	1	1	0.2
	2		
	3	2	0.4
	4		
800	1	1	0.8
	2		
	3	2	1.6
	4		

After spiking, the plates are left to dry in one of the Health Physics fume cupboards. That is done because dried beryllium standard poses a higher inhalation risk than beryllium contained in the solution. Plates 1 and 3 were smeared using Whatman n°1 filter paper and plates 2 and 4 were smeared using Ghost Wipes (see Fig. 2.3). After wiping with the chosen media, the plates were cleaned from any beryllium left on plates using pre-moistened alcohol wipes (AZO brand). After clean-up, the plates were tested for any possible beryllium contamination using standard Health Physics procedure [14]. Once the results confirmed that plates are clean from beryllium, new standard would be spiked onto the plates and left to dry before wiping of the plates would take place.

2.2.2 Stage 2 – Real Environment

The second part of the experiment was in the real environment, which in this case is a surface in a fume cupboard (FC) used for Intermediate Level Waste (ILW) sorting in Materials Detritiation Facility (MDF). The surface was divided into 10 smaller surfaces, as shown in Figure 2.4.

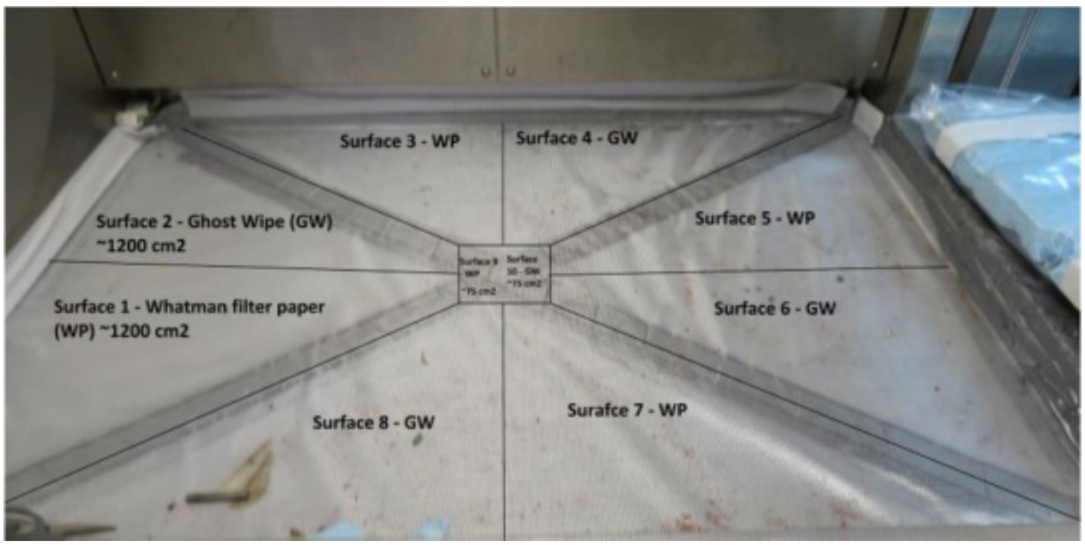

Figure 2.4: Area in FC in MDF chosen as a real environment for testing wiping media

The size of the chosen area is 820mm x 1200mm. As exact levels of beryllium and contamination distribution are unknown, the area was divided into 10 smaller areas and each one was smeared using a different medium. In Figure 2.4, each surface is numbered 1-10 and marked with WP if smeared using Whatman n°1 filter paper or GW if smeared using Ghost Wipes. The size of smaller surfaces is given in Table 2.3.

Table 2.3: division of FC surface into 10 smaller surfaces

<table>
<tr><th>Surface No.</th><th>Size (cm²)</th><th>Wiping Medium</th></tr>
<tr><td>1</td><td rowspan="8">1210</td><td>Whatman n°1 Filter Paper</td></tr>
<tr><td>2</td><td>Ghost Wipes</td></tr>
<tr><td>3</td><td>Whatman n°1 Filter Paper</td></tr>
<tr><td>4</td><td>Ghost Wipes</td></tr>
<tr><td>5</td><td>Whatman n°1 Filter Paper</td></tr>
<tr><td>6</td><td>Ghost Wipes</td></tr>
<tr><td>7</td><td>Whatman n°1 Filter Paper</td></tr>
<tr><td>8</td><td>Ghost Wipes</td></tr>
<tr><td>9</td><td rowspan="2">75</td><td>Whatman n°1 Filter Paper</td></tr>
<tr><td>10</td><td>Ghost Wipes</td></tr>
</table>

Surfaces 1-10 as shown in Figure 2.4 can be grouped into 5 pairs. Each one out of 5 larger surfaces is wiped with both media. The results are then compared within the larger surface, as it is considered that contamination on each part is uniform. For instance, it is more difficult to predict if contamination will be uniformly distributed over Surfaces 1 and 2, or over Surfaces 8 and 1. Therefore, both options are taken into account during analysis of the results. The two options for grouping of Surfaces 1-10 are shown in Figures 2.5 and 2.6.

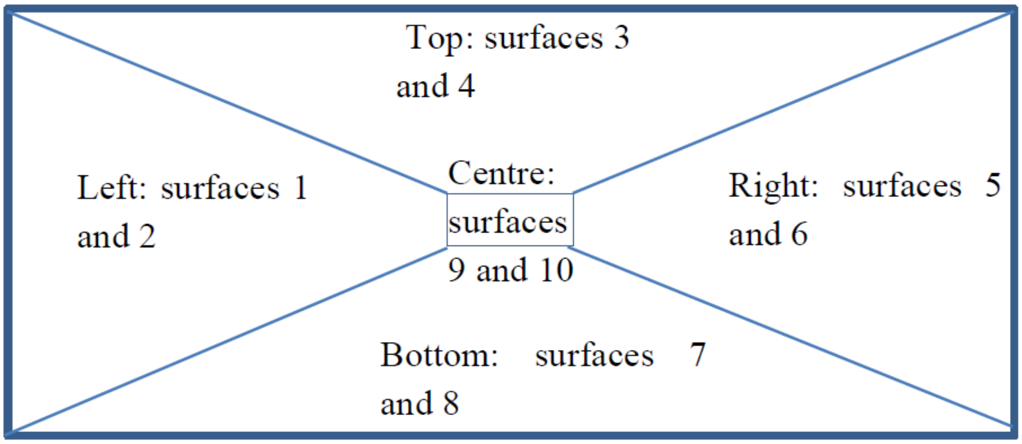

Figure 2.5: FC surfaces selection - 5 pairs, first option

The idea is to see in how many pairs, one of the media will show more beryllium contamination than the other, and how big is the difference in collection efficiency between the chosen media.

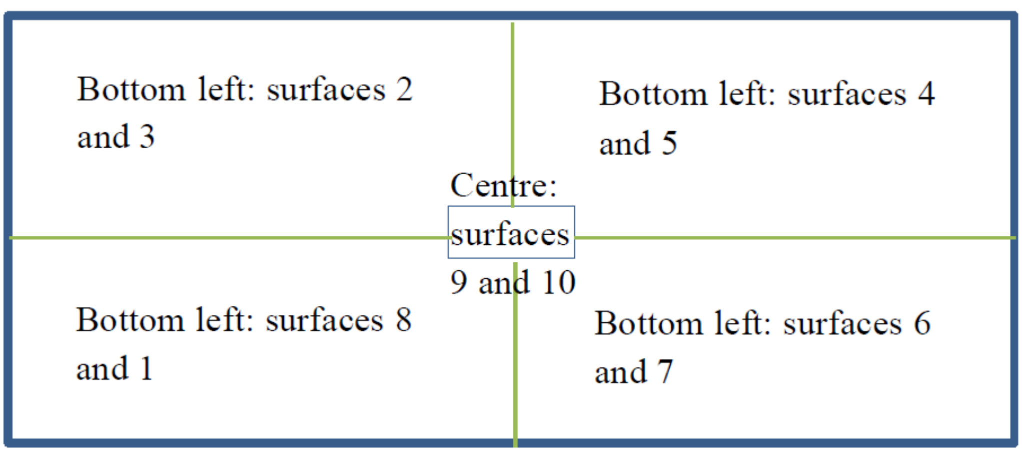

Figure 2.6: FC surfaces selection - 5 pairs, second option

2.3 Surface Sampling – technique factors that compromise sampling and analysis

Throughout Chapter 2, it could be noticed that there are some factors which can positively or negatively affect the final results. It is important that these factors are taken in consideration, as the affected results might not be comparable. The conditions for wiping the surface with Whatman n°1 filter paper and Ghost Wipes must be the same, so that results are a real representation of their collection efficiency and not a representation of the other differences (sampling technique, wiped surface, size of the media etc.). In this section, factors identified as potential to compromise sampling will be analysed. These factors are:

- Media characteristics
- Surface quality
- Sampling technique and sample manipulation

2.3.1 Media Characteristics

The objective of this experiment is to compare the collection efficiencies for two different wiping media under the same conditions. Therefore, it is important to compare the chosen media characteristics to each other and try to adjust some of their properties. For purposes of this experiment, the two media tested are Whatman n°1 filter paper and Ghost Wipes, which are described in the beginning of Chapter 2. From Table 2.1, it can be expected that Ghost Wipes will have an advantage in surface wiping due to their wetting agent and size.

Whatman filter paper has a surface at least 9 times smaller than Ghost Wipes. Moreover, it is a dry medium, hence it is expected to pick up less than a wet medium. As the purpose of this experiment is to compare the quality of the chosen media (performance of the material as it is) and not the quantity (Ghost Wipes are obviously bigger than Whatman n°1 filter paper), no changes were applied regarding the wetting agent.

Figure 2.7: Folded Ghost Wipes (left), Whatman n°1 filter paper (centre), cut Whatman n°1 filter paper (right)

The size was adjusted so that both media had the same size and shape. Initially, the idea was to cut the Ghost Wipes down to match the shape and size of the Whatman n°1 filter paper, but during that process, the Ghost Wipes would lose a significant amount of wetting agent. The amount of wetting agent in the Ghost Wipes was measured during the validation of fluorescence assay on Ghost Wipes [15]. The value obtained in this report is considered during calculations and any loss of the wetting agent would make the dilution factor incorrect. Therefore, it was decided to cut the Whatman n°1 filter paper to match the size of the folded Ghost Wipe, as it is in its packaging (Fig. 2.7). Several Ghost Wipes were taken out of their bags and measured. The size of the surface of the folded Ghost Wipe is 27mm x 42mm.

2.3.2 Surface Quality

The quality of the surface which will be wiped with chosen media should be the same in case of both media with, if possible, same amount of beryllium contamination and same kind of beryllium contamination, e.g. BeO, acetate, etc. In this experiment there are two stages.

The first stage is in the lab environment, meaning that the sampling conditions are created. The surface chosen was glass. The purpose in this stage of the experiment was not to compare the amount of beryllium collected from Ghost Wipes and Whatman n°1 filter paper directly, but rather to compare the recovered beryllium from the wipe with amount of beryllium spiked onto the surface. For this reason, surface chosen for wiping had to be made of a material which would not absorb any beryllium. Therefore, plates made of glass, were chosen. The shape of the glass plates is concave. The spiked amount of beryllium is presented in Table 2.2. In this way the amount and quality of beryllium contamination as well as the quality and size of the surface were the same.

In the second stage of the experiment, surface chosen to be wiped is the surface from the FC in MDF. The FC is used for sorting of ILW. In this case, it is not possible to have equal amounts of beryllium contamination on the surfaces, but quality of the surfaces (vinyl cover on the surface) is the same. The distribution of the contamination is not known in advance. Therefore, it was assumed that the contamination is uniform and justification for that is the surface division.

For instance, if the contamination is localized in one corner of the surface, then smearing one side of the surface with one medium and other with another medium will not represent the real situation regarding the collection efficiencies of the media. Therefore, more segments were divided. If contamination is localized in the corner between Surfaces 1 and 8, part of that contamination will be collected with one medium and part with another medium. If the contamination was localized on the right side, between Surfaces 5 and 6, it would still be detected with both media.

The possibility of having higher contamination on a smaller surface far from the centre is low because of the nature of work performed on the chosen surface. The contamination is, therefore, expected around the centre of the FC surface. With the division of the surface as described earlier, the difference in recovered beryllium from both media can be understood as the difference due to efficiency and not due to beryllium contamination distribution.

2.3.3 Sampling Technique

In Section 2.1.1, the surface-sampling technique applied in this experiment was described. In Stage 1 of the experiment, the glass plates were wiped using a circular pattern, covering the whole contaminated surface. In Stage 2 of the experiment, all 10 surfaces were wiped using a Z-pattern. The Stage 1 wiping was performed by a different technician each time, whereas the Stage 2 wiping was all performed by the same technician.

Each group of samples was collected by a different technician. Although the same instructions were given and every technician followed them, many differences were still noticed in the applied techniques. Many of them are not easy to quantify. The differences recognized are:

- Pressure and number of strokes: Each technician had a different approach in handling the glass plates. Some would take a plate with one hand and wipe with other. As the plates are concave, holding the plate with one hand would provide stability, hence higher pressure was applied on the surface during wiping. One technician made very few strokes, still enough to wipe all the surface, while another would use a greater number of strokes, but apply less pressure, also holding the plate. Others would not take the plate in the hand but try to hold it in place with one hand on the FC surface and wipe with other hand. As the plate was less stable using this last technique, the pressure applied in that case would be even less. The number of strokes would be more than 10.

- Pick-up of the contamination and folding of the smear: Some technicians tried to pick up visible contamination while having the wipe on the surface and fold it immediately without lifting the medium from the surface, while the others would wipe, lift the unfolded medium, and then fold it. In this case, it seems like less contamination would be picked up, particularly when wiping with a dry medium. All smears were folded with the contamination on the inside.

This is not considered as a big factor to the results reported in this paper, because each technician who sampled the glass surfaces was sampling the whole group of the surfaces (4 glass plates) in the same way. For instance, the glass plates spiked with 40PPB beryllium liquid standard solution (1ml for Plates 1-2 and 2ml for Plates 3-4), were wiped by the same technician in the same way and at the same time. Therefore, if the sampling technique was less efficient than technique of someone else, it was less efficient in sampling using both the Whatman n°1 filter paper and Ghost Wipes. Therefore, the results will be comparable to each other. This can have an effect in calculating mean recovery, but still the mean recovery of one medium is obtained under same conditions as the mean recovery of the another medium. Hence, the results are still comparable.

3. Results and Discussion

3.1 Lab Results

In Stage 1, there were 8 pair-samples, therefore a total of 16 samples analysed. In case of 100% recovery, the expected amount of beryllium, given in μg/sample, is presented in Table 2.2. The results were obtained in PPB for each sample. The results were then converted into μg/sample by applying the dilution factor, which depends on the amount of dilution solution and detection solution added. In the case of Ghost Wipes, the amount of de-ionised water from the wipe was included in the dilution factor. The results are then compared against the spiked amount of beryllium on the plate in μg. The beryllium recovery for each smear was calculated using the following formula:

$$Be\ recovery\ [\%] = \frac{Recovered\ beryllium\ [\mu g]}{Spiked\ beryllium\ [\mu g]}\ x\ 100$$

The mean recovery for Whatman n°1 filter paper and Ghost Wipes is shown in Table 3.1.

Table 3.1: Mean recovery and RSD [%] for Whatman n°1 filter paper and Ghost wipes

Media	Mean Recovery (%)	Standard Deviation	Relative Standard Deviation (%)
Whatman n°1 Filter Paper	28.67	0.22	77.33
Ghost Wipes	87.66	0.10	10.95

The difference between the collection efficiencies of Whatman n°1 filter paper and Ghost wipes was visible even with the naked eye. From Figure 3.1, it can be seen that Plate 3 smeared using Whatman n°1 filter paper still contains some dried beryllium standard, while Plate 4 wiped with Ghost Wipes looks completely clean.

From Figure 3.2, it can be seen that the mean recovery of Ghost Wipes is around 3 times higher than that of Whatman n°1 filter paper. The mean recovery based on results from this experiment supports the mean recovery of Ghost Wipes based on results from the paper of Sarah K. Dufay et al. [16], where the reported mean recovery for Ghost Wipes was reported as 85.9%.

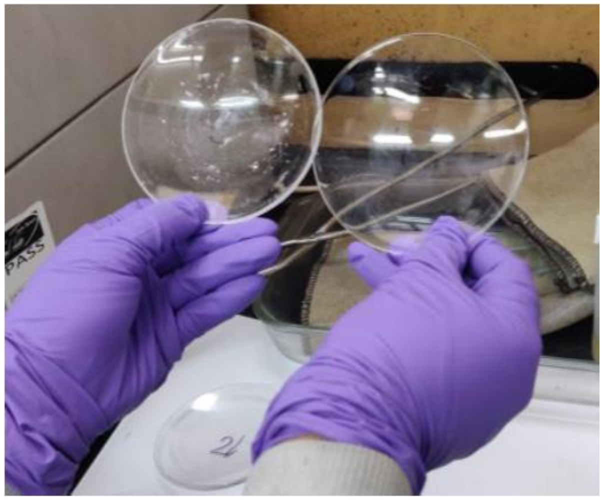

Figure 3.1: Plates 3 (on the left) and 4 (on the right) wiped with Whatman n°1 filter paper and Ghost Wipes, respectively

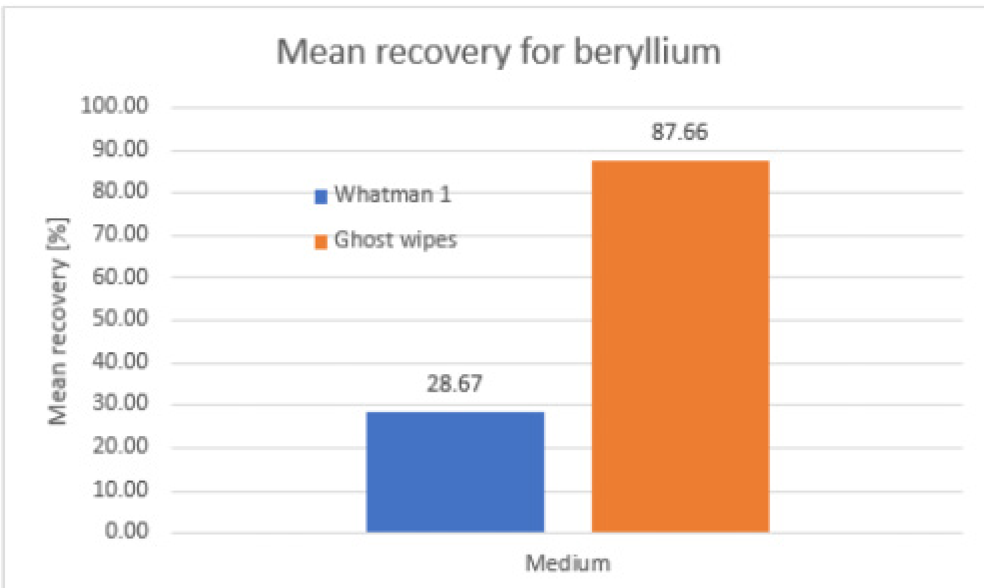

Figure 3.2: Comparison of mean recovery for Whatman n°1 filter paper and Ghost Wipes.

According to the results from this stage of the experiment, beryllium recovery depends on the amount of beryllium contamination. It can be seen from Table 3.2 that the efficiency of the Whatman n°1 filter paper is significantly higher when tested on surfaces with higher levels of contamination, while the efficiency of Ghost Wipes is greater than 70% in case of any amount of beryllium contamination.

Therefore, the relative standard deviation (RSD) reported in Table 3.1 is significantly higher for Whatman filter paper than for Ghost Wipes. This might be due to the fact that the Whatman n°1 filter paper was used in a dry condition.

Table 3.2: Comparison of mean recovery for Whatman n°1 filter paper and Ghost wipes with change of Be contamination.

Beryllium Standard (PPB)	Recovery using Whatman n°1 Filter Paper (%)	Recovery using Ghost Wipes (%)	Comments on Differences in Efficiencies
40	0-15	85-100	GW are at least 7 times more efficient
200	20-40	75-90	GW are 1 to 6 times more efficient
800	55-65	90-100	GW are less than 2 times as efficient

3.2 Real Environment Results

In Stage 2 of the experiment, there were 5 pair-samples, in total 10 samples taken from the surface in FC used for ILW sorting in the MDF. The division of the FC surface into 10 smaller surfaces allowed for more accurate comparison of the efficiencies, as the contamination on smaller area is more likely to be uniform and therefore the same for both media. From Figure 3.3, it can be seen that beryllium surface contamination results from Ghost Wipes (purple) are significantly higher comparing to the results from Whatman n°1 filter paper (red).

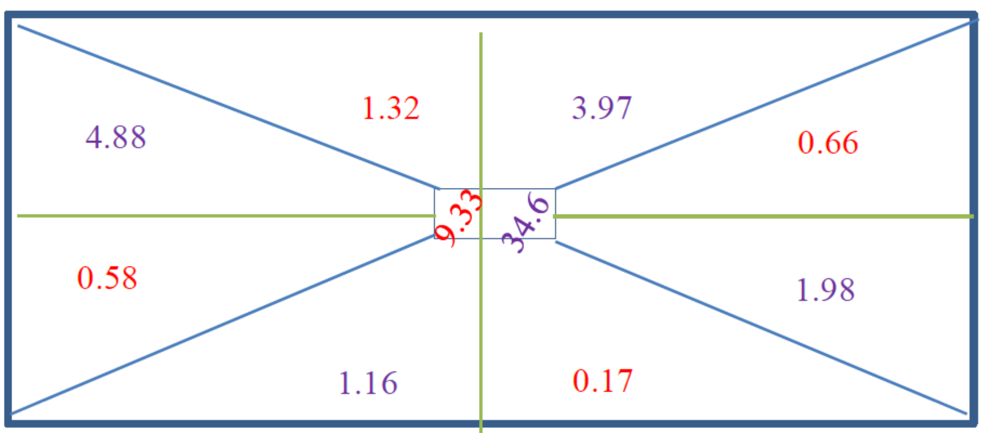

Figure 3.3: Beryllium recovered from different parts of FC using Whatman n°1 filter paper (red) and Ghost wipes (purple). Results are expressed as μg/m^2.

In Figures 3.4 and 3.5, the results are compared using the groupings from Figures 2.5 and 2.6, respectively. It can be seen that in both cases, Ghost Wipes were more efficient than dry Whatman n°1 filter paper. Having the pairs grouped either way (from Fig. 2.5 or Fig. 2.6), comparison showed that Ghost Wipes collected between 5 and 5.5 times more beryllium from the surface than dry Whatman n°1 filter paper.

For the comparison shown in Figure 2.5, the biggest gap in beryllium recovery was for the left side (Surfaces 1 and 2), where Ghost Wipes collected 8.43 times more beryllium than dry Whatman n°1 filter paper. The smallest gap was for top and right side (Surfaces 3-6), where Ghost Wipes collected 3 times more beryllium than the dry Whatman n°1 filter paper, which is in agreement with the Stage 1 experiment results.

For the comparison according to grouping shown in Figure 2.6, the biggest gap was for the bottom right segment (Surfaces 6 and 7), where Ghost Wipes collected as high as 12 times more beryllium than the dry

Whatman n°1 filter paper. The smallest gap was for the bottom left (Surfaces 1 and 8), where Ghost Wipes collected only two times more beryllium than the dry Whatman filter paper. In Section 3.1, it was mentioned that the gap between beryllium recovery for chosen media was bigger in case of lesser amount of beryllium contamination on the surface. From the results presented in Figures 3.4 and 3.5, the same conclusion can be reached. The bigger gaps between the results were where beryllium contamination was not too high (e.g. bottom right from Fig. 3.5).

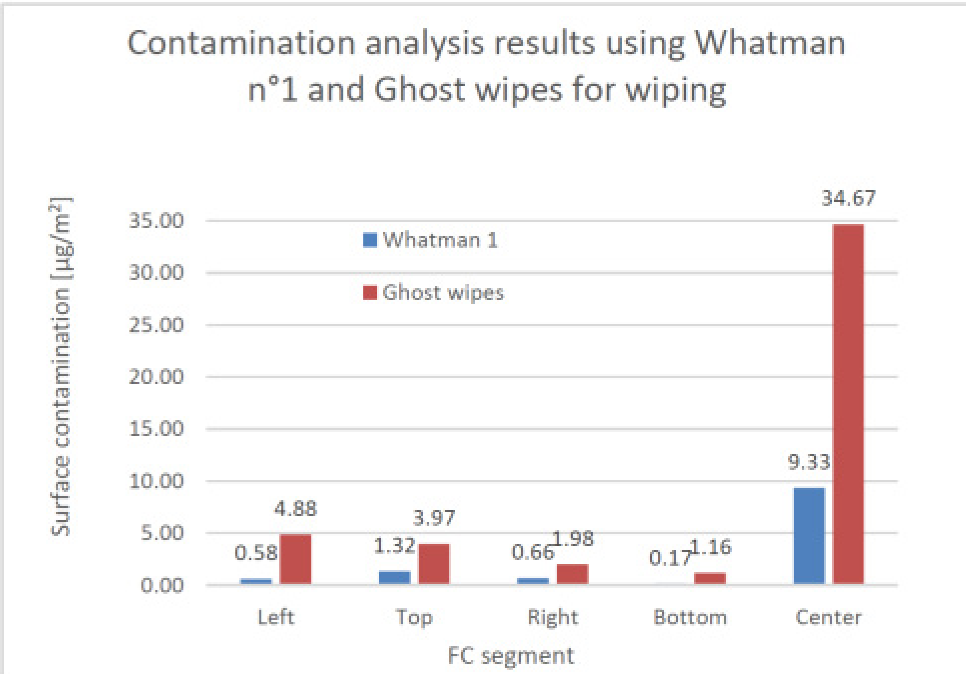

Figure 3.4: Results comparison for smear pairs grouped as in Fig. 2.5.

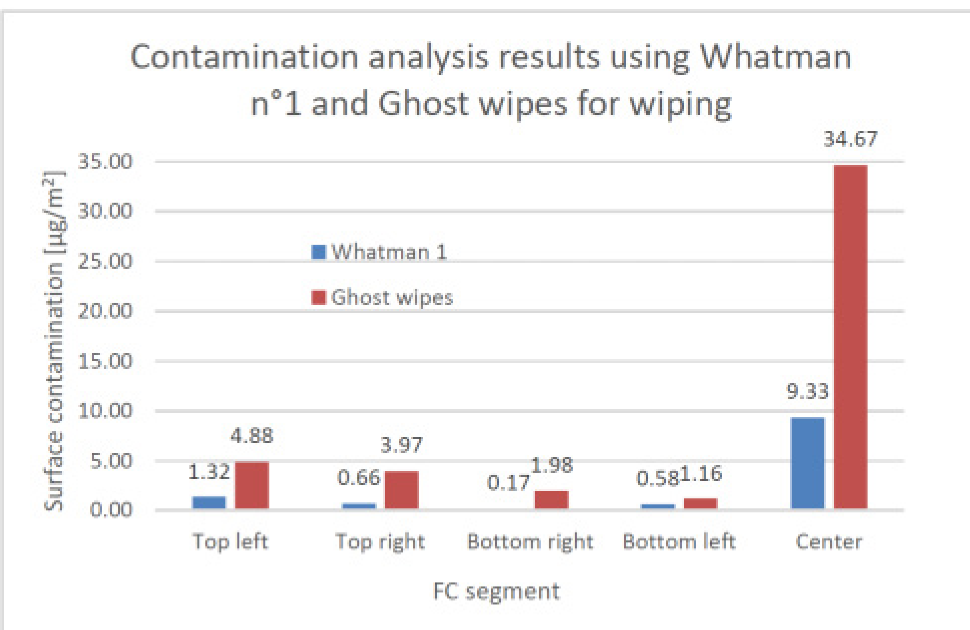

Figure 3.5: Results comparison for smear pairs grouped as in Fig 2.6.

4. Conclusion

The results indicate a significant difference between collection efficiencies of dry Whatman n°1 filter paper and Ghost Wipes. Under laboratory conditions, the Ghost Wipes showed a mean recovery of 87.66%, with a standard deviation of 0.1 and a relative standard deviation (RSD) of 10.95%. The dry Whatman n°1 filter paper showed a mean recovery of 28.67%, with a standard deviation of 0.22 and RSD of 77.33%. In the real environment, Ghost Wipes recovered more beryllium in all smear pairs. The amount of beryllium collected using Ghost Wipes was from 2 to 12 times higher than amount of beryllium collected using dry Whatman n°1 filter paper (see Figs. 2.5 and 2.6).

Based on efficiency results, it seems that using Ghost wipes will provide better recovery for loose beryllium surface contamination with ~87.66% recovery (see Fig. 3.2). In both stages of the experiment, it appears that Whatman n°1 filter paper shows lower efficiency when wiping the surface with low beryllium contamination. Its efficiency increases with the level of beryllium contamination (see Table 3.2 and Figs. 3.4 and 3.5).

4.1 Future Work

As previously mentioned in the Conclusion above, Ghost Wipes showed better collection efficiency for loose beryllium surface contamination than dry Whatman n°1 filter paper. These results were obtained based on analysis of 16 samples in a laboratory environment and 10 samples in real operational environment, for a total of 26 samples. The difference between beryllium recoveries for chosen media varies from 2 to 12 times in favour of Ghost Wipes.

It was also noticed that collection efficiency depends on many factors, such as sampling technique, the quality of the medium used, and the quality of the surface wiped. In order to have a narrower range of efficiency differences, more samples would be needed for analysis, especially for the case of low beryllium contamination. Also, it would be necessary to test the wipes on different surfaces and components in the real operational environment.

The surfaces used in this experiment were glass and a surface covered with vinyl. Due to the relative smoothness of these materials, the wiping of the surfaces is relatively feasible. Some surfaces can be more challenging in the sense of being rougher. This might have an impact on the quality of the sampling technique. The rough surface can damage the sampling medium and therefore result in a change in the sampling technique (e.g. more tapping instead of actual wiping) for such surface.

Acknowledgments

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 and 2019-2020 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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EUROfusion

Comparison of collection efficiencies of Whatman n°1 filter paper and Ghost wipes for loose beryllium surface contamination

Mirjana Damjanovic

cea CCFE CULHAM CENTRE FOR FUSION ENERGY

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 and 2019-2020 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

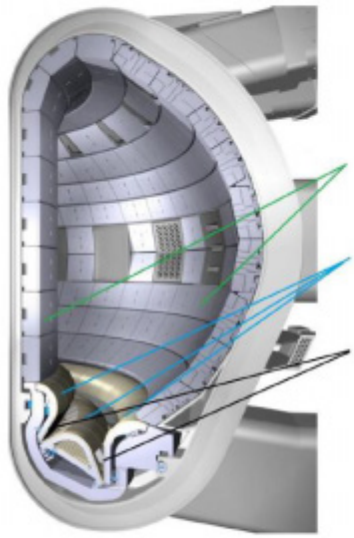
Content

Introduction

- Beryllium and its application in fusion

https://periodictable.com/Samples/004.1/s13.JPG

Properties	
Atomic number	4
Atomic weight [amu]	9.012
Density [g/cm^3]	1.85
Electronic structure	$1s^2 2s^2$



Beryllium first wall
- Good oxygen gettering ability
- Low radiative power losses due to low Z
- Low tritium inventory
- ➢ But high melting, physical sputtering yield and n-induced brittleness, toxic

Tungsten
- Low physical sputtering yield
- Low tritium inventory
- ➢ But high radiative losses, melting and activation

Carbon-fibre-composites (CFCs)
- Good thermal shock and fatigue resistance
- Low radiative power losses due to low Z
- Does not melt
- ➢ But high erosion, requires conditioning and change of thermal conductivity due to n-irradiation, high tritium retention

https://www.iter.org/fr/album/media/7%20-%20technical

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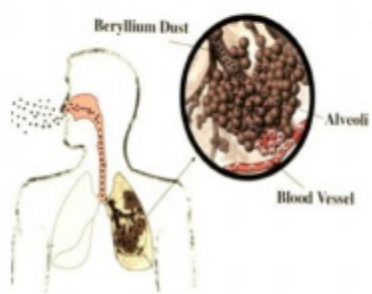
Beryllium effects on health

- Exposure to fumes and dust
  - Acute beryllium disease
  - Sensitization
  - Chronic beryllium disease (CBD)



https://whiteleyresearch199.weebly.com/beryllium-diseases.html

- Beryllium exposure control
  - Dedicated facilities for beryllium handling
  - Personal and respiratory protective equipment
  - Beryllium contamination monitoring (airborne and surface)
  - Occupational exposure limits & 'no protection' limits (airborne and surface contamination limits)
  - Local instructions, training and medical examination

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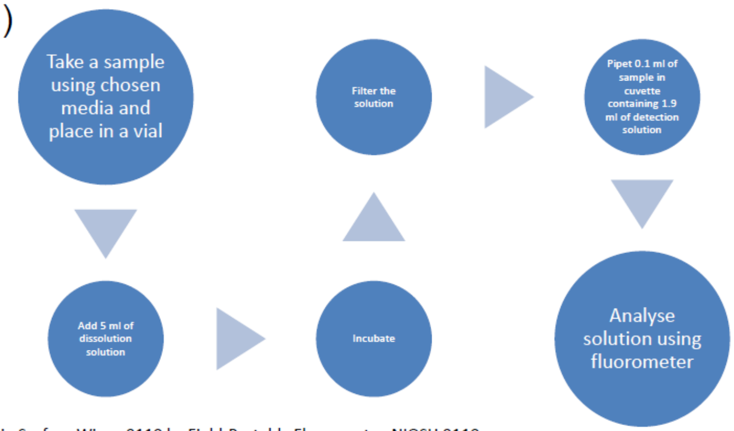
Sampling and analysis method at CCFE

- Smearing medium – Whatman filter paper n°1
- Dissolution solution – Ammonium bifluoride
- Detection solution - Hydroxybenzoquinoline sulfonate (HBQS)



BERYLLIUM in Surface Wipes 9110 by Field-Portable Fluorometry, NIOSH 9110 - https://www.cdc.gov/niosh/docs/2003-154/pdfs/9110.pdf

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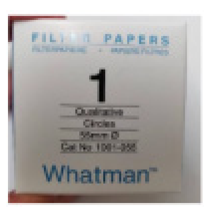
Experiment overview

- Beryllium smear samples analysis method
  - Following NIOSH 9110 procedure for beryllium smear samples analysis
  - Following validation report from Health Physics Group (HPG), UKAEA on Ghost wipes smear samples analysis for beryllium using fluorometry (HPG/WI 24-21)
- The objective of the experiment is to show that collection efficiency of Ghost wipes will be higher than the efficiency of the Whatman n°1 filter paper currently used by HPG in CCFE.



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Experiment overview

- Two parts of the experiment:
  - Laboratory environment: smearing surfaces with known amount of beryllium using different medias and compare recovery results
  - 'Real environment' (unknown levels): smearing surfaces/components with high potential of beryllium contamination on site using different media and compare results

- Media characteristics
  - Whatman n°1 filter paper (dry):
    - D = 55 mm
  - Ghost wipes (wet):
    - Surface: 15 x 15 cm
    - moistened with deionised water

– Adapt the surfaces of smearing media to match

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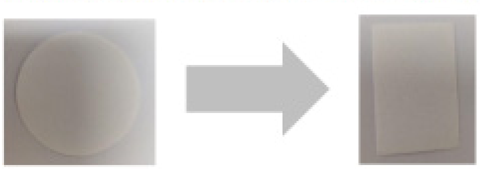
Experiment overview

- Factors with an impact on beryllium recovery from the surface
  - Wet/dry medium – advantage for the Ghost wipes
  - Size of the medium – adapted to be the same
  - Smeared surface material – glass plates, surface covered with vinyl
  - Manipulation of the medium (pressure, folding the smear, picking up the dust etc.) – dependent on the technician, different for each group* of samples
- Size adaptation
  - Ghost wipes folded in a package: 2.7 x 4.2 cm
  - Whatman filter paper 1 is cut to exact same size



* 4 glass plates / fume cupboard surface smeared by same person

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Experiment overview

- **Laboratory experiment details**
  - 4 identical glass plates: D = 10 cm
  - Beryllium liquid standards (0, 40 PPB, 200 PPB, 800 PPB)
    - Plates 1&2 spiked with 1 ml of standards
    - Plates 3&4 spiked with 2 ml of standards
    - Left to dry
  - Using 2 different media to take smears
    - Plates 1&3: Whatman n°1 filter paper
    - Plates 2&4: Ghost wipes

Whatman Ghost wipe

1 ml

2 ml

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Experiment overview

- **'Real environment' details**
  - Fume cupboard surface in Materials Detritiation Facility (MDF) in Culham used for ILW sorting
  - Divided in 10 areas
    - 8 areas with surface ~1200 cm^2
    - 2 areas with surface ~75 cm^2
  - Assumption: uniform contamination justified by area division

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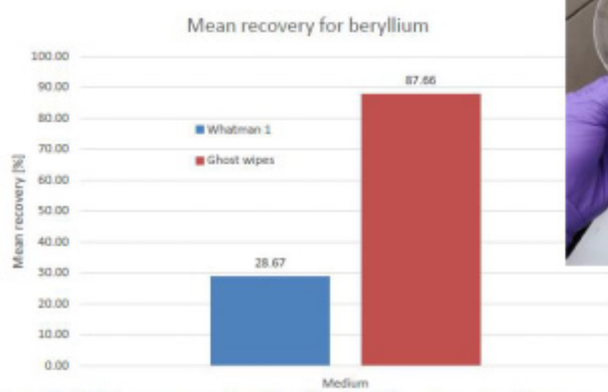
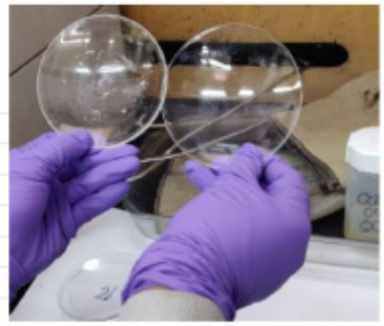
Results

- **Lab results**



Comparison of the mean recovery of beryllium from glass plates using Whatman n°1 filter paper and Ghost wipes



Glass plates 3 (on the left) and 4 (on the right) smeared with Whatman n°1 filter paper and Ghost wipes, respectively

*Supports the results for Ghost wipes mean recovery reported in: Sarah K. Dufay and Melecita Archuleta, "Comparison of collection efficiencies of sampling methods for removable beryllium surface contamination", 2006, Journal of environmental monitoring, DO - 10.1039/b601526n

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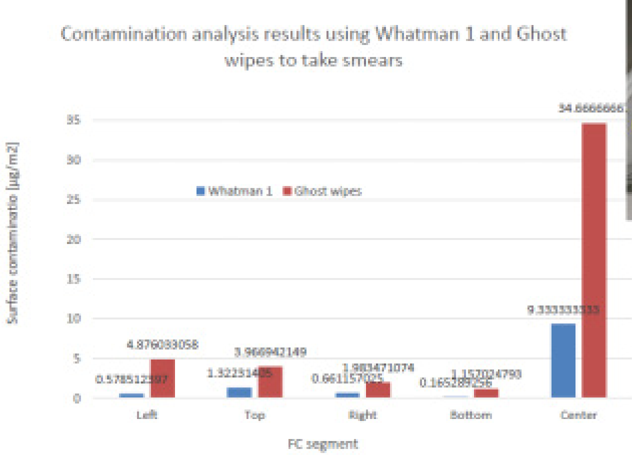
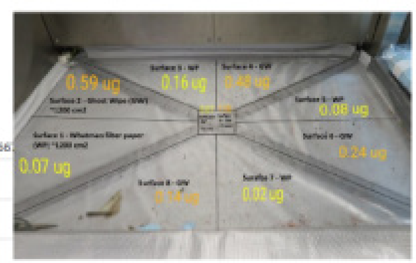
Results

- **MDF Fume cupboard results**



Comparison of the beryllium surface contamination collected with Whantman n°1 filter paper and Ghost wipes



Quantities of beryllium (micrograms) detcted on each side of FC surface using Whatman n°1 filter papers (in yellow) and Ghost wipes (in orange)

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Conclusion

- Ghost wipes are ~ 3 times more efficient than Whatman n°1 filter paper for glass surfaces
- The collection efficiency is dependent on the manipulation of the smear (difficult to quantify)
- In unknown-contamination environment, Ghost wipes recovered more beryllium in each smear pair

Future work

- Take more samples from 'real environment' (other contaminated components and surfaces)
  - Surface in other FC in MDF
  - CFC tiles used as PFM in JET
- Test different surfaces (performance on rough surfaces and different materials)
  - Emery cloth / sandpaper in lab conditions

Thank you for your attention !

Beryllium Fire, Combustibility & Explosivity Risks in Fusion Energy

Marc Kolanz and Theodore Knudson

Materion Corporation, Mayfield Heights, Ohio, U.S.A.

Beryllium metal powder does have the potential to be combustible and/or react under certain conditions. Beryllium metal powder is classified as a "Flammable Solid" for transportation purposes and exhibits mild explosive potential by several test methods. This presentation will review the information on the potential combustibility and explosivity of beryllium and explore these potential risks for the use of beryllium in fusion energy production.

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ITER is evaluating the available evidence regarding three potential risk scenarios

- Fires of combustible materials in the presence of components containing solid beryllium
- Ignition of finely divided beryllium particulate
- Generation of loose matter (beryllium/tungsten) during plasma/wall interactions

The US Department of Energy sponsored a report by Mishima in 2006* which contains information responsive to the first two questions.

*Mishima, J.; Foppe, T. L.; Laul, J. C.; McEahern, P. M.; Pinkston, D. M.; Restrepo, L. F. Proposed Beryllium Metal Bounding Airborne Release Fractions (ARFs)/Rates (ARRs) and Respirable Fractions (RFs) for DOE Facility Accident Analysis, LA-UR-05-1096, Los Alamos National Laboratory, NM, April 2005; Rev.1, September 2006.

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Fires of combustible materials in the presence of solid beryllium components

Mishima's review of decades of published reports found no reports, studies or documented events indicating the ignition of large coherent pieces of metal.

Blumenthal and Santy 1964 heated beryllium metal rods (0.5 inch x 3/16 inch) to 1500 degrees centigrade (above the melting point of beryllium) found "no indication of ignition or rapidly accelerating reaction;"

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Fires of combustible materials in the presence of solid beryllium components

Practical examples:

- 1995 Hanger fire burned a Kiowa Scout Helicopter. 1650° C
- Beryllium metal shapes during reentry from space. >1500° C
- Decade of beryllium use in Joint European Torus Reactor (JET)
  - Fusion requires a temperature of around 150,000,000° C (10X our Sun)

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Combustion of accumulations of beryllium particulate

The Mishima report confirms that a layer of beryllium powder can be ignited and can intensely burn at high temperature.

NOTE: Water should never be used to extinguish a beryllium fire due to the potential for a steam explosion or the ignition of hydrogen gas caused by the disassociation of hydrogen from water at high temperatures.

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Combustion of accumulations of beryllium particulate

Practical examples:

- Materion Brush ductwork fires
  - Welding
  - Grinding sparks

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Ignition of airborne beryllium particulate

Table IV

Rating of explosion severity according to the Kst value[23]

Dust explosion class	K_{st} (bar.m/s)	Characteristic
St 0	0	No explosion
St 1	>0 and <= 200	Weak explosion
St 2	>200 and <= 300	Strong explosion
St 3	> 300	Very strong explosion

KST: The size-normalized maximum rate of pressure rise under standard testing conditions.

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Ignition of airborne beryllium particulate

The Mishima report review of explosibility used older test methods and classified beryllium metal powder as having a category S1 (weakly explosive) potential.
Today's test method utilizes a standardized 20L testing chamber method.

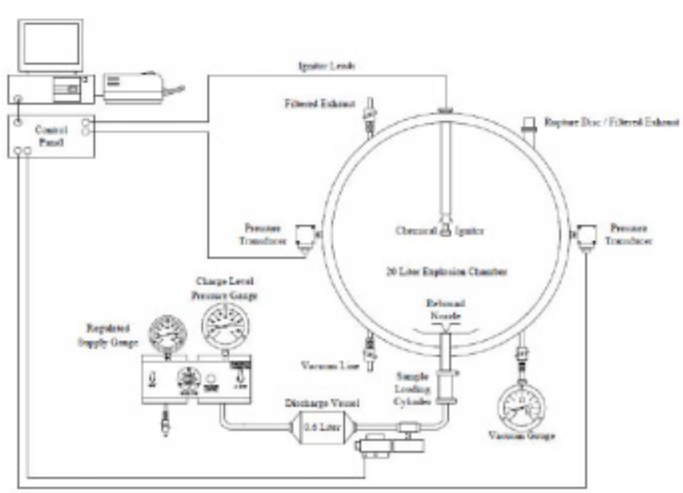

Figure 2: Apparatus for measuring dust explosion parameters (EXP)

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Two studies identified evaluating beryllium using the 20L chamber

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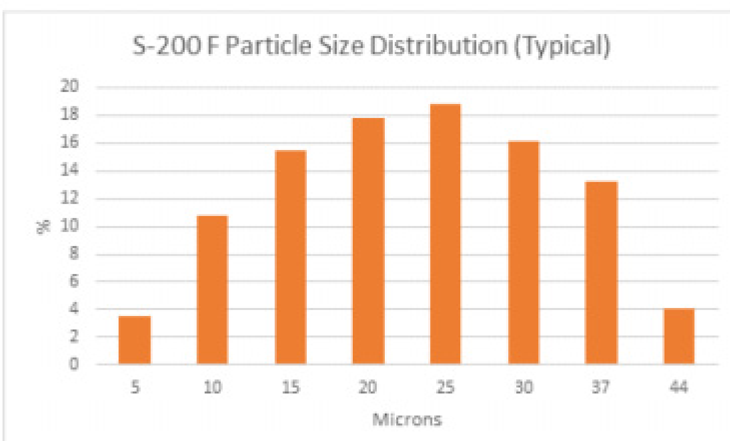
Ignition of airborne beryllium particulate

2009 Fike test of Materion Brush Inc. S-200 beryllium powder product

- Powder is 44 µm or less sieve measurement
- 1500 g/m^3
- Maximum Kst = 39 (St 1- weak explosion = 0-200)



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Ignition of airborne beryllium particulate

Idaho National Laboratory ITER Explosion Indices of Be dust (baghouse dust)

- **Powder is 0.1µm < d < 20µm**
- **250 g/m³**
- **Maximum Kst = 222 (St 2 – strong explosion "lower range" of 200-300)**

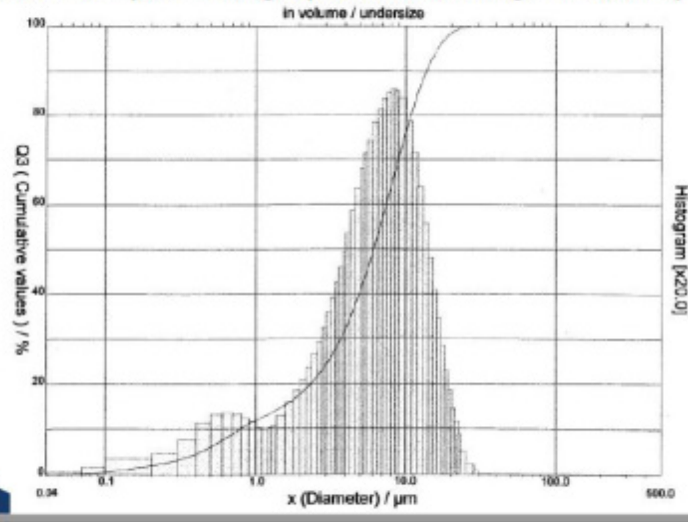

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Table 4. Beryllium dust explosion test summary.

Test ID	Concentration (g/m³)	Dust mass (g)	P_{max} (bar)	dP/dt_{max} (bar/s)
2	0	0	0	0
4	60	1.2	0†	0†
6	125	2.5	8.2	333*
20	125	2.5	8.6	619
22	125	2.5	8.8	606
8	250	5	10.0	979
12	250	5	9.4	803
18	250	5	9.4	672
10	500	10	9.1	785
14	500	10	8.1	660
24	500	10	8.8	636
16	750	15	7.5	622

* In this case, the KSEP software did not properly identify the steepest slope. The value in this table was determined manually by a linear fit to seven data points (span: 2.4 ms) in the appropriate region.

† No dust explosion occurred in this case; the pressure trace (Figure 45) was similar to shots with igniters only and no dust (Figure 30, Figure 44). This may have resulted from much of the 1.2 g remaining in the dust container.

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Ignition of airborne beryllium particulate

Idaho National Laboratory ITER Explosion Indices of Be dust (baghouse dust)

- Test #6 at 60g/m3 showed no ignition.
  - Study speculates on the result but failed to repeat the negative test to confirm a LEL
- Use of beryllium metal manufacturing baghouse dust could contain other non-beryllium combustibles which may account for the significantly higher Kst versus the Fike test of pure beryllium powder product using the same method.
  - The INL test dust size was smaller

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Material Explosibility Examples

Kst Examples and ST class test results table

Material	Kst	ST Class
	bar.m/sec	
Be (S-200)	38	ST I
Flour	65	ST I
Grain dust	89	ST I
Sugar	138	ST I
Starch (rice)	220	ST2
Be (highest)	222	ST2
Wood dust	224	ST2
Aluminum dust	515	ST3

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JET ITER-like walls dust generation (JET-ILW)

Issue:

- *"Concerns regarding reactor safety and diagnostic performance in a reactor-class device are the main driving forces for dust studies in current machines."* (Fortuna-Zalena)
- *"Generation of dust particles in a reactor-class thermonuclear reactor is considered as a potential hazard which would create serious issues regarding safety and economy of operation. In the worst case of a massive water leak onto hot dust a pressure build-up and an explosion of the released hydrogen could occur. Air ingress may lead to ignition."* (Rubel)

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JET ITER-like walls dust generation (JET-ILW)

Reviewed two papers evaluating beryllium dust generation and splash deposits at the JET-ILW reactor.

2017 E. Fortuna-Zalena et al., Fine metal dust particles on the wall probes from JET-ILW, Phys. Scr., T170 (2017) 014038 (9pp)

2018 M. Rubel et al., Dust generation in tokamaks: Overview of beryllium and tungsten dust characterization in JET with the ITER-like wall. Fusion Engineering and Design 136 (2018) 579-586

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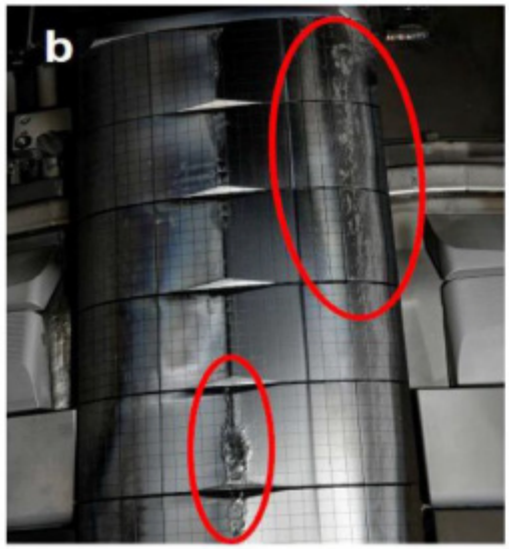
JET ITER-like walls dust generation (JET-ILW)

Comprehensive collection of loose dust by vacuum cleaning resulted in the collection of about 1 gram of loose dust (all species) in each of 3 campaigns (2011-2012), (2013-2014) and (2015-2016). The second campaign had 19.5 hours of plasma operation. The amount of loose dust remained around 1 gram despite the energy levels increasing steadily from 150 GJ to 201 GJ to 245 GJ.



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JET ITER-like walls dust generation (JET-ILW)

<u>Adhered deposits</u> of particles, flakes, tile fragments and splashes were collected using adhesive carbon pads (120 locations), dust collection monitors (test mirrors and silicon plates) and erosion deposition probes. The main aim of the work was to characterize the metal particle portion, focusing on beryllium.

Phys. Scr. T170 (2017) 014008 E Fortuna-Zaleśna et al

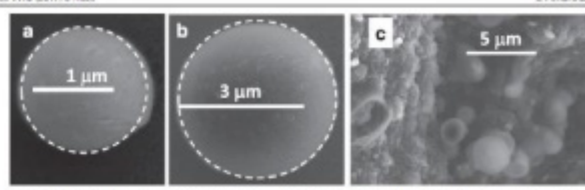

Figure 6. (a) and (b) Spherical beryllium droplets on the dust monitor; (c) first-ever recorded beryllium bubbles in the Be-rich deposit on the divertor tile.

Phys. Scr. T170 (2017) 014008 E Fortuna-Zaleśna et al

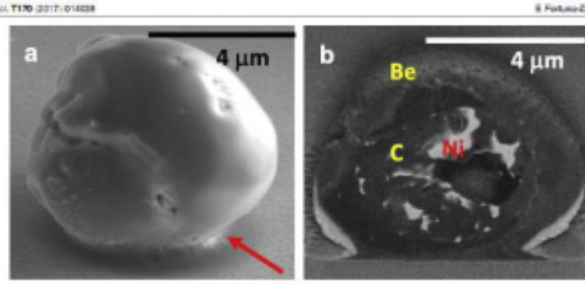

Figure 8. Agglomerated dust particles on the outer wall monitor: (a) whole particle and (b) a cross-section obtained by FIB. The object is coated with a tight Be mantle masking smaller dust particles of C and Ni.

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JET ITER-like walls dust generation (JET-ILW)

"visual inspection of the monitors indicated the presence of thin deposited layers and only very few particles which could be considered as dust"

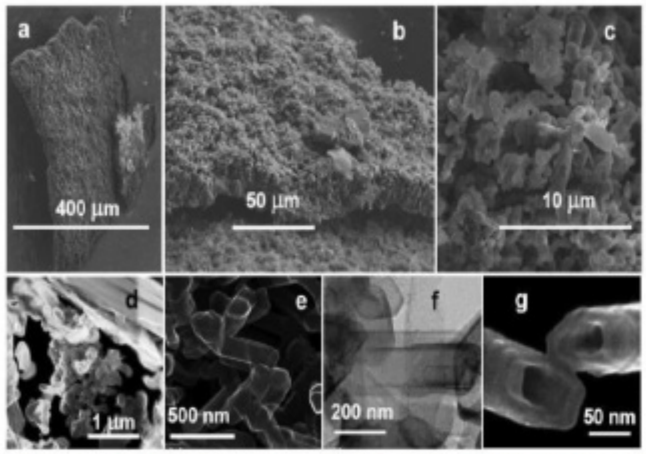

Fig. 7. Thick beryllium deposits retrieved from the divertor carriers: (a)-(c) surface topography recorded by SEM and (d)-(g) internal structure recorded by STEM from the FIB-produced lamellae. Images (e) and (g) recorded in secondary electron mode, while (d) is high angle annular dark field image and (f) is a bright field image.

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JET ITER-like walls dust generation (JET-ILW)

"Particles distributed uniformly on the entire surface of the monitor from the inner wall are mainly beryllium splashes." "…despite internal stresses the splashes adhere well to the substrate.".

Phys. Scr. T170 (2017) 014035 E Fortuna-Zalesna et al

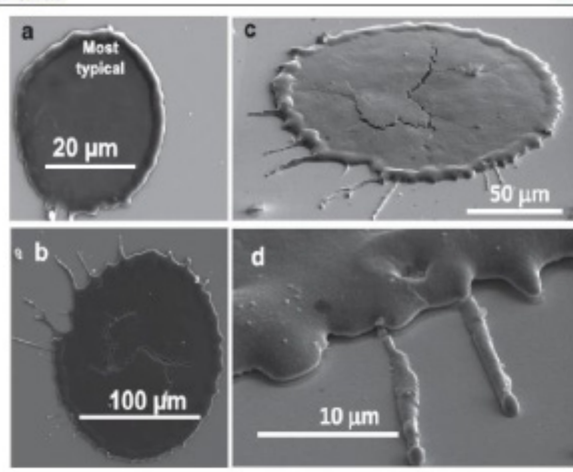

Figure 3. Micrographs of beryllium splashes on the inner wall monitor. Images (a) and (b) taken at normal angle while (c) and (d) at 58°.

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JET ITER-like walls dust generation (JET-ILW)

The paper concluded that the splashes did not occur throughout the campaign, but occurred during the last operation day when there was a *"generation of run-away electrons."*

An estimated total loss of beryllium as "splashes/droplets" from the 12.5m^2 inner divertor was 0.52 gram (0.3 cm^3).

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JET ITER-like walls dust generation (JET-ILW)

In an ITER-like reactor:

- Fortuna-Zalena concluded ***"The results of this work, based on dust quantification, may allow some optimism, as they indicate only minor material losses"***
- JET-ILW has operated for about a decade with no apparent reports of beryllium particulate posing a combustion/explosion or spontaneous ignition event.
- In 3 successive campaigns, only 1 gram of loose dust is released per campaign (all constituents)
- About 0.5 gram of beryllium is eroded and redeposits securely on surfaces (mostly during a "run-away electron event" at the end of a campaign)

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ITER differences vs. JET

- The first wall in both ITER and JET is beryllium
- ITER will have a more powerful plasma
- ITER run time is 100+ seconds versus JET at 20 seconds
- ITER volume is 830 m^3 vs JET at 100 m^3
- ITER beryllium surface area is 613 m^2 vs expected "plasma wetted" Be area expected to be about 10% of this area.
- 40% of wall erosion at ITER is estimated to be like JET
  - JET believed to **only** have wall erosion
- 60% of loss is estimated from edge-localized modes (ELMs).
  - ITER plans to control ELMs using Resonant Magnetic Perturbations
- Will off-normal events at ITER mimic JET off-normal events?

ELM = What happens when the plasma pressure at the edge goes above a certain threshold, the plasma then expels a burst of particles/energy to relax and decrease the pressure.

MATERION

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ITER differences vs. JET

- Using the estimated 0.52 g of beryllium loss from a 12.5m^2 beryllium divertor converts to about a 25 gram loss in ITER.
- HOWEVER…Fortuna-Zalena and Rubel state that the available JET evidence does not mean it can be directly scaled (to ITER)
- ITER is performing work seeking to better define the range of potential beryllium losses in the ITER reactor.

MATERION

Relationship between surface contamination and airborne particulate of hazardous materials; including the perception of associated risk and how this has a wider impact on Perception of Health and Safety

Beth Walker

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With substances that are an airborne hazard such as asbestos, silica, and beryllium, there is often a perception of risk. This does not always correlate with the actual risk. With airborne contamination, the primary risk is the substance entering the lungs. However, it has been observed that workers and public often relate this to the physical object itself rather that the particulate surrounding the object.

For example, you will often hear people say 'don't touch that, it's asbestos' but people may walk through a heavily contaminated area with no controls in place. Asbestos and beryllium are relatively harmless in their solid form, as they are not readily airborne. However, an asbestos or beryllium-contaminated area will have fine particulate that is easily airborne, and thus, high risk. This demonstrates a disparity between the perceived risk and actual risks.

What would the control limit for a swab of the physical item be? This question was asked because the current control limit (in Europe) is 0.002mg/m^3 of air. As swabs will give results in mg/cm^2 of surface material, there is no way to translate this easily, especially when you consider the Van der Waals force. There is little research that is related to this issue. In the asbestos industry, swab testing has been used for many years, but has never been used as a definitive marker, and is increasing falling out of favour, as it cannot be used on items containing asbestos, only following a removal of asbestos, where the dust has settled, and then only to give an indication of the level of cleanliness, not contamination. The Health and Safety Laboratory in the UK has done some research. However, this only refers to fibres that have been disturbed and left to settle, not a physical object. This means that there is very little data surrounding whether the airborne risk has a linear relationship with a physical object.

How cautious should we be of physical objects that produce an airborne hazard? Due to the lack of concrete evidence people are left to make their own mind up, and this relies on the subjective nature of risk awareness and risk culture. As this differs from workplace to workplace, there is currently no consistent standard to be drawn upon. The aim of this research paper is to begin the process of making a relationship, if it exists and opening the way for further research into the subject.

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Research proposal

THE QUEEN MARY
BeWS-14
2019
14th International Workshop
On Beryllium Technology
Long Beach, California, USA
24-25 October 2019

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Relationship between surface contamination and airborne particulate of hazardous materials;

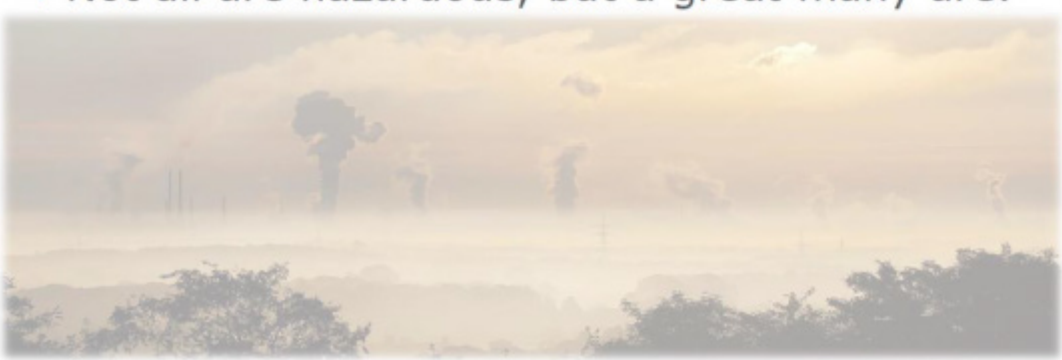
including the perception of associated risk and how this has a wider impact on Perception of Health and safety

Beth Walker CMIOSH NEBOSH Dip

25th October 2019

Introduction

- Airborne Contaminants take on many forms, from bacterial and biological to human skin cells.
- Not all are hazardous, but a great many are.



2

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Introduction

- The relationship with this has always been a little inconsistent in people's minds.
- For example, during outbreaks of illnesses that can be transmitted through breathing, there is often a prevalence of people who decide to wear face masks. (health care workers' risk perception(….) 2014)
- They then take these off once the risk of illness has lessened. The risk has only diminished slightly and there are many other airborne illnesses that can still be picked up.

3

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Introduction

- People are also phobic of things like germs, but unperturbed by much more dangerous airborne risks such as chemical exposure (for example, people often expose themselves to serious risk by mixing cleaning products in pursuit of eradicating the lesser risk of germs and bacteria).
- This risk extends to airborne contaminants that are a by-product of a hazardous material. Asbestos, Silica and Beryllium are the best examples of these.

4

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Current thinking

- One of the current tests for Beryllium is to swab test surfaces of Beryllium to ascertain the risk, since this is not yet airborne and there is no proven relationship to airborne risk
- Swab testing is carried out in the Asbestos industry, but predominantly to ascertain the settled dust particulate in the area, which has a proven relationship with airborne dust. (HSE, 2006, 15)

5

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Hypothesis

- Based on the research problem the hypothesise that will be put forward for testing are:
  - That people have a disproportionate view of risk when it comes to assessing the hazards of air borne particulate.

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Hypothesis

- Based on the research problem the hypothesise that will be put forward for testing are:
  - People are often more wary of the physical object than the dust it produces.

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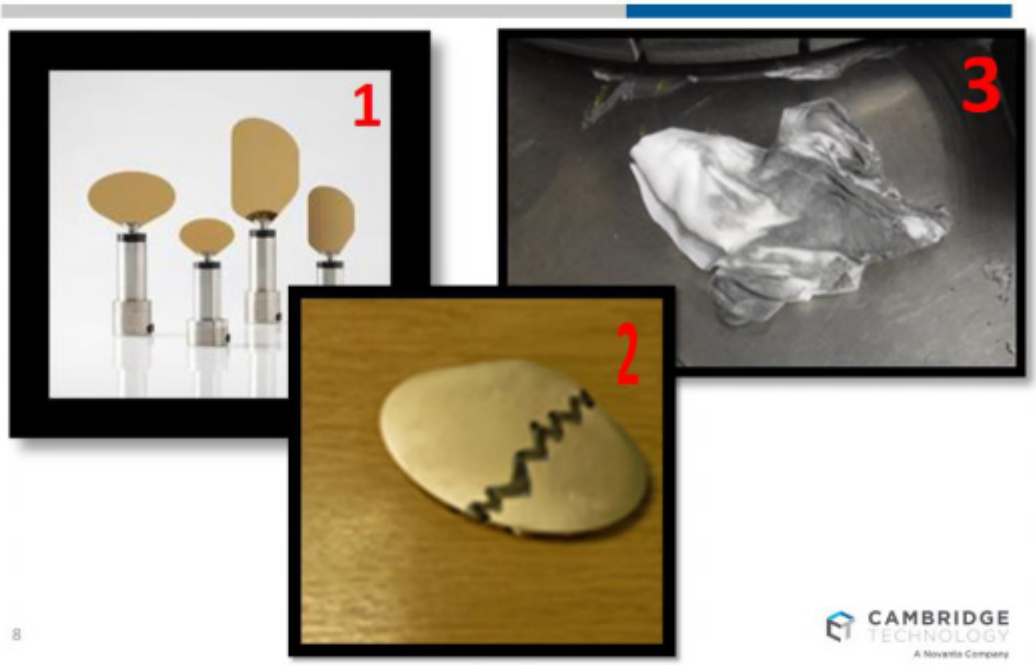
Conditions offered to respondents



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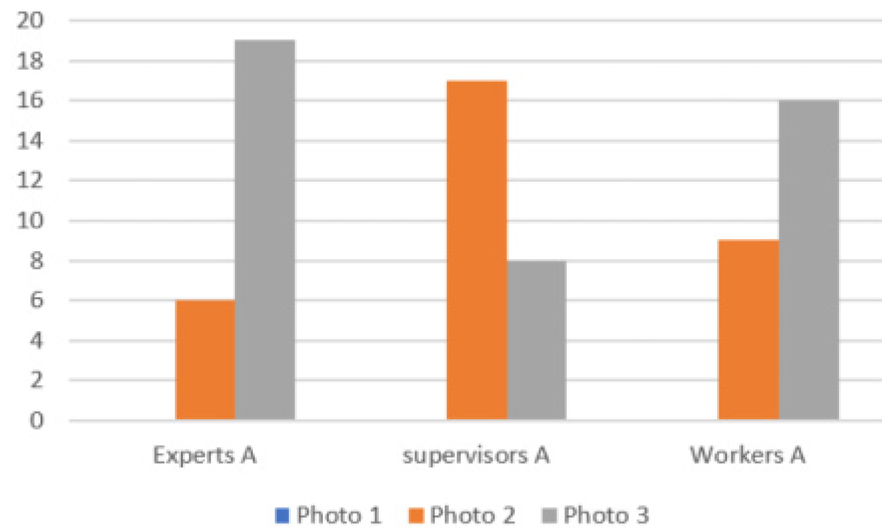
Hazards as rated by various groups



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Hypothesis

- Based on the research problem the hypothesise that will be put forward for testing are:
  - There is a demonstrable, positive and linier relationship between the physical object and the airborne hazard that can offer people a definitive way to ascertain the hazard produced by the physical object.

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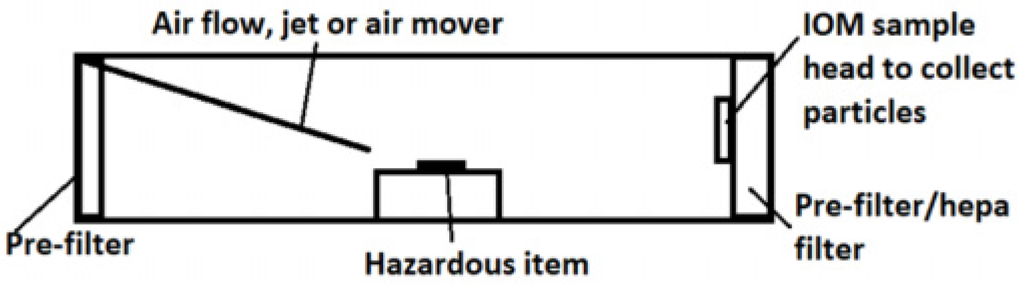
How do we measure this relationship



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How do we measure this relationship

- Swab sample of item
- Place item in chamber
- Turn air movement on
- Measure airborne particulate with IOM sample head
- Repeat for different conditions and at different air speeds.

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How do we measure this relationship

- To ensure that the study is as accurate as possible the NIOSH Beryllium Sampling technique will be adopted to take a sample of each Beryllium sample (NIOSH, 2008) as this will give consistent results.

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How do we measure this relationship

- The chamber can have a humidity of around 60-70% to reduce the effect of the Van der Waals forces.
- The Air flow can be at a 45-degree angle as used by Fletcher et al (2008) to ensure maximum disturbance.

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Identifying Beryllium Exposure Risks and the Diagnosis of Chronic Beryllium Disease

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Abstract

The successful identification of workplace beryllium risks and disease requires specially trained occupational health professionals and physicians using special techniques and tests. Without these key elements, risk priorities can be misidentified, and disease can easily be misdiagnosed.

While all occupational exposure limits rely on weight-based methods, the more accurate risk indicator is particle number. Studies are presented that highlight these now apparent differences. Further, Chronic Beryllium Disease (CBD) has been misdiagnosed as sarcoidosis in progressive cases and as onset of old age for the less progressive or early symptomatic conditions.

Experience within the US Department of Energy (DOE) contractor facilities gives a clear example of how CBD can go unrecognized. After several CBD cases were diagnosed at the DOE Rocky Flats Plant, worker studies were initiated that included the Beryllium Lymphocyte Proliferation Test (BeLPT). Historical identification of CBD and other illnesses from the US and select countries is presented. Early diagnosis is critical to enable treatment to inhibit the progression of the disease, to give feedback for occupational health and safety practices, and to allow for medical removal from beryllium exposure. Recommended methods for identifying CBD with current diagnostic tests along with available guidance on the application and interpretation of those tests are discussed.

Keywords: Beryllium Exposure, Chronic Beryllium Disease, Beryllium Risk, Risk Assessment, Beryllium Sensitization, Nickel Sensitization, Copper Beryllium, Ultrafine Particulate Exposure, T-Cell Mediate Immune Response

1 Introduction

Beryllium is a light-weight element that comes from ores that are processed into beryllium metal, beryllium oxide, beryllium-containing alloys and beryllium salts. Beryllium metal is primarily used in the defense, nuclear, and aerospace industries. Beryllium-containing alloys of aluminum, nickel, magnesium, and iron alloys are increasingly used in various industries (Müller-Quernheim, 2006). The primary very widespread use of beryllium is not the pure metal but is copper-beryllium alloy.

Beryllium exposure is known to cause the occupational diseases of Acute Beryllium Disease (ABD), conjunctivitis, dermatitis, lung cancer, and Chronic Beryllium Disease (CBD). ABD, conjunctivitis, dermatitis, and lung cancer are mostly diseases of the past when exposure levels were less controlled and much higher than current workplace exposure limits (Hardy, 1965). This article discusses primarily CBD, which is a risk even under current occupational exposure limits used for beryllium.

In order to provide a clear understanding of the exposure risk, the systems involved in CBD which are the skin, the immune system, and the lungs are discussed. Some basic information on how genetics plays a role is given since only a subset of the population is susceptible or genetically predisposed to acquiring CBD.

Further, the steps to progression to CBD are described. This leads into the current understanding of the risks of occupational exposure to beryllium. A historical perspective of lessons learned from the US Department of Energy is given to allow the reader to understand that there is a current risk today at manufacturing and research facilities that process beryllium-containing components. Diagnosis of CBD is discussed to include an understanding of the necessary medical tests, given that CBD can and currently is easily misdiagnosed.

2 Human Body Systems Involved in CBD

As mentioned earlier, three body systems play a role in the development of CBD from exposure to beryllium. Also described is a very basic understanding of genetics so that the terms used here can be more easily understood.

2.1 The Skin

The skin is the largest organ in the body and the first line of defense against foreign invaders. The skin has three primary layers, the epidermis, the dermis, and the hypodermis as shown in Figure 2.1. While the skin provides a passive physical barrier, the skin also contains elements of the innate and adaptive immune system that actively defend against foreign invaders. Foreign invaders are also called antigens. The skin contains immune cells and lymphatic vessels that allow communication of the skin with the immune system to activate the body’s defenses against antigens.

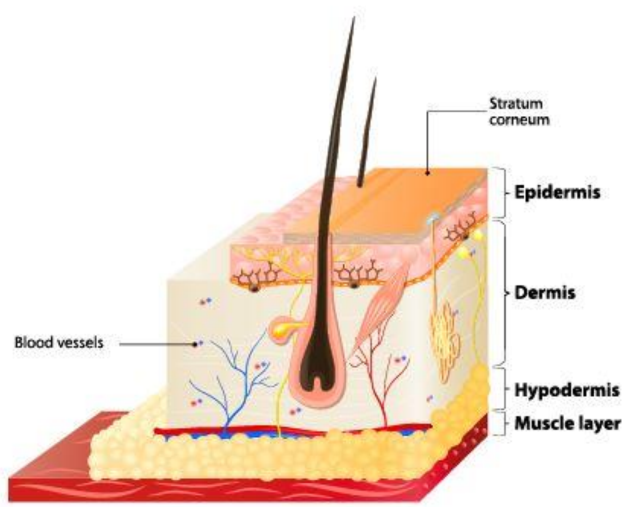

Figure 2.1: Diagram of the layers of the skin.

2.2 The Immune System

The immune system as shown in Fig. 2.2 consists of the tonsils, thymus gland, bone marrow, spleen, and the lymphatics and lymph nodes. Further, this system has immune cells that are produced initially in the bone marrow, mature in various organs, and circulate throughout the body through the bloodstream and the lymphatic system where some of them reside in other system's organs such as the skin and lungs.

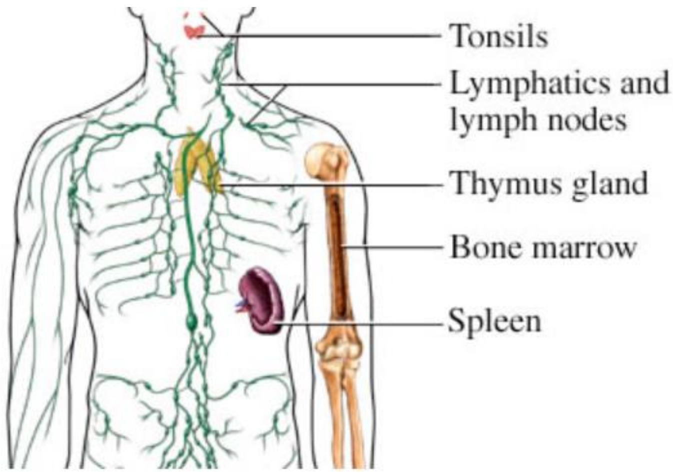

Fig. 2.2: Pictorial of the Human Immune System.

There are many types of immune cells, and in this article, the description primarily includes cells that are part of the adaptive immune system and are involved in the human's response in developing CBD.

The skin's epidermis layer contains Langerhans cells (a form of dendritic cell) which capture, process, and present antigens to T-Cells (also called T Lymphocytes). The name T-Cell is derived from the fact that the cells mature in the thymus. Langerhans cells are a subset of macrophages that reside in the skin. Macrophages are cells that engulf antigens in a process called phagocytosis. A well-known type of macrophage is a white blood cell. Figures 2.3 and 2.4 are examples of a dendritic cell and T-Cell showing the cell extensions where the receptor sites are located.

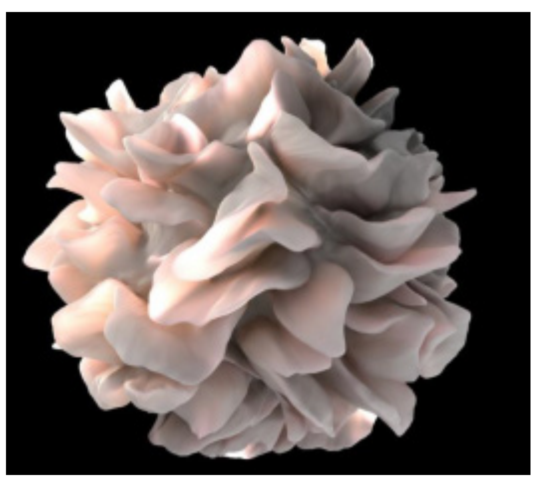

Figure 2.3: Artistic rendering of the surface of a human dendritic cell illustrating fin-shaped dendrites or cell extensions that fold back onto the membrane surface. Wiki image.

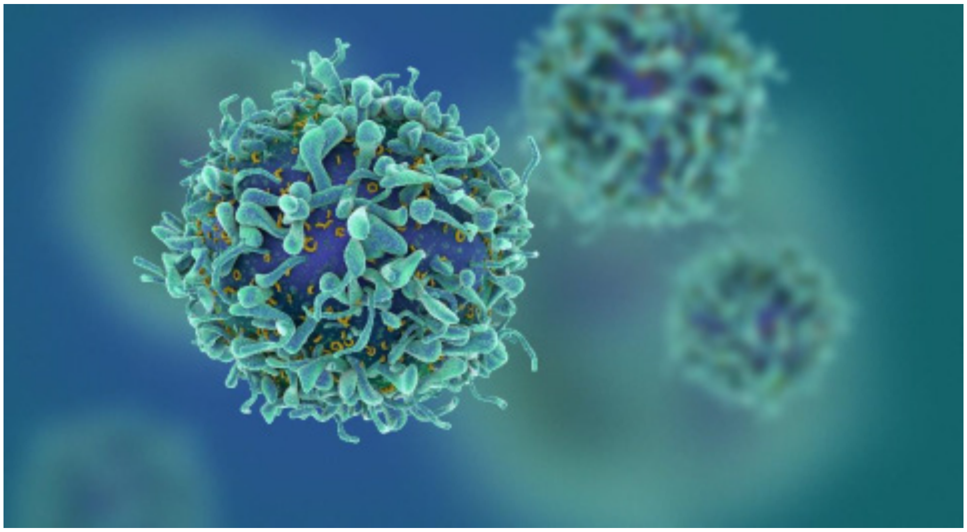

Figure 2.4: T-cells showing the receptor cites outside the cell.

The basic function of the immune system is to locate and destroy then remove foreign invaders. To provide an understanding of the immune system in general, all categorical system reactions are described, including protective responses such as fighting infections, and the harmful responses of hypersensitivity to a perceived foreign invader. There are two types of immune system reactions, the innate and the adaptive. The innate reactions happen immediately and are non-specific. A type of adaptive reaction is one where the cells remember or know the foreign invader from prior exposure. The adaptive reaction takes days to be initiated. An adaptive reaction that is well known is our body's defence against bacteria. B-Cells produce antibodies and T-Cells kill and remove the infected cells.

However, some individuals have immune cells that overreact; their adaptive immune system responses are harmful to the body. For the purpose of this article, this is called a hypersensitivity reaction as shown in the diagram in Fig. 2.5.

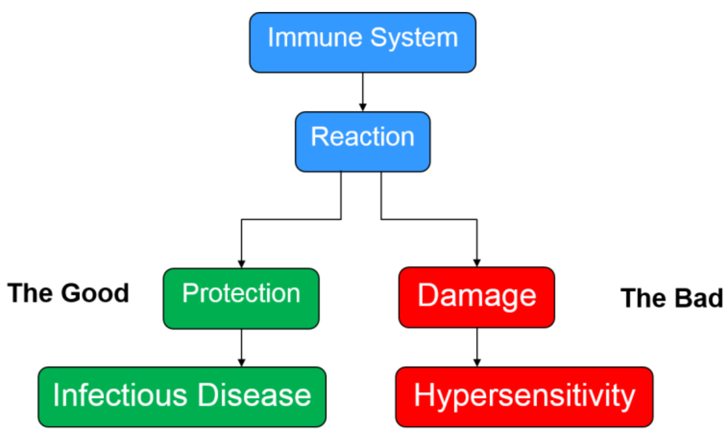

Figure 2.5: Simplistic diagram of the good and harmful responses of the immune system.

Common allergies to grasses, pollen, and certain foods are a type of hypersensitivity reaction. There are four types of hypersensitivity reactions, Type I through IV as shown in Fig. 2.6. CBD is a Type IV T-Cell mediated immune system reaction. The term "T-Cell mediated" means that T-Cells convey the antigen's presence to other immune cells. Type IV immune system reactions do not involve antibodies, but rather the immunity involves the activation of macrophages, antigen-specific T-Cells, and release of these cells' cytokines (signalling proteins) in response to the antigen.

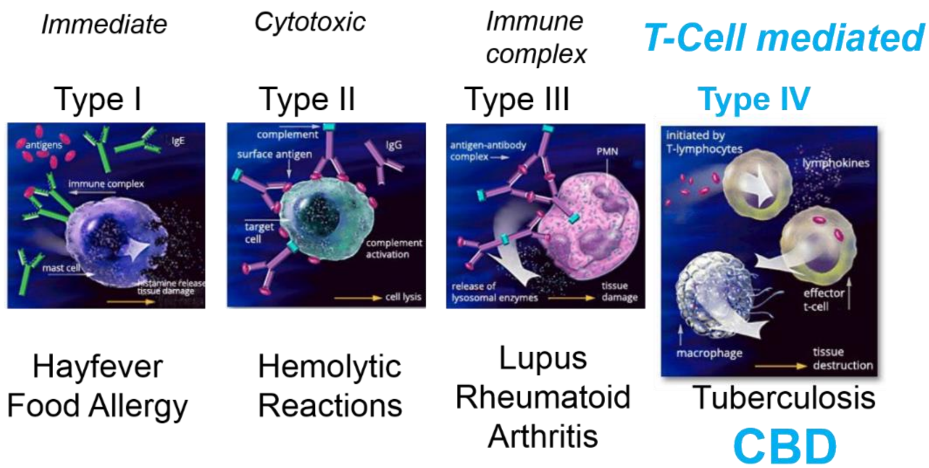

Fig. 2.6: Four types of hypersensitivity reactions with a few of their associated diseases. Images courtesy of Dental Care.

T-Cells carry receptor molecules that have the shape and charge designed to recognize specific targets on foreign invaders. These molecules are called Major Histocompatibility Complex (MHC) molecules. One can think of these molecules as an extension outside the cell that have a cup-like or fin-shaped receptor. The cups or fins are varying shapes and have different electrical charges to enable binding to or holding onto a specific antigen. Our bodies make the MHC molecules. There physical size and shape of the cups or folds (i.e., receptor locations) are specific and differ depending on the coding of our genes.

A short discussion of how genetics plays a role in immune system response is in order. Humans have 23 pairs of chromosomes which exist inside all cells in the nucleus. Chromosomes are comprised of strands of deoxyribonucleic acid (DNA) and genes are a subset of the DNA. DNA is made of chemical building blocks called nucleotides. These building blocks are made of three parts: a phosphate group, a sugar group and one of four types of nitrogen bases.

To form a strand of DNA, nucleotides are linked into chains, with the phosphate and sugar groups alternating. The four types of nitrogen bases found in nucleotides are: adenine (A), thymine (T), guanine (G) and cytosine (C). The order, or sequence, of these bases determines what biological instructions are contained in a strand of DNA. For example, the sequence ATCGTT might instruct for blue eyes, while ATCGCT might instruct for brown. Chromosomes have two pairs; one from each parent. Individuals that have one copy of a gene are called heterozygotes and those that have two identical genes are called homozygotes. See Figs. 2.7, 2.8, and 2.9.

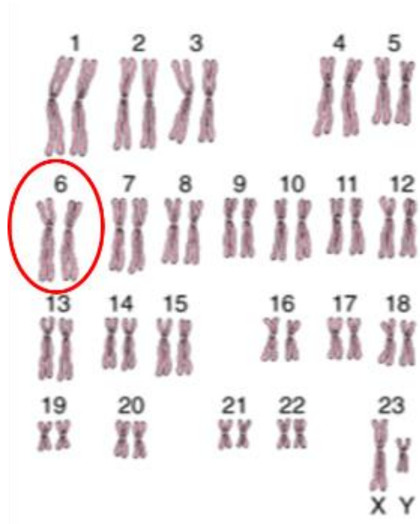

Figure 2.7: Twenty-three base pairs of chromosomes. Chromosome six is highlighted because the markers for CBD are located on this chromosome. Wiki image.

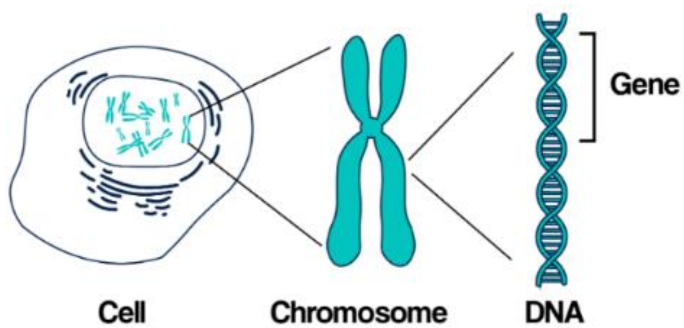

Figure 2.8: Pictographs of cells with 23 chromosome pairs, a chromosome, DNA and genes. Wiki image.

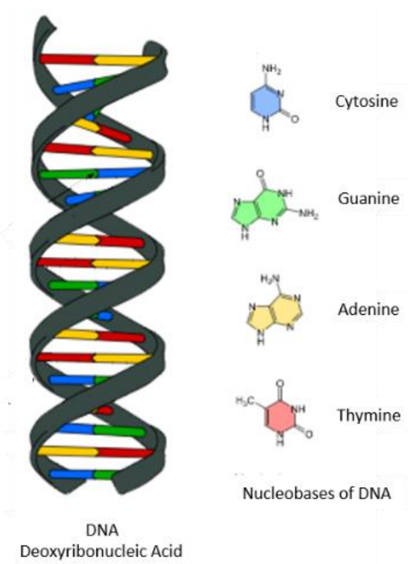

Figure 2.9: DNA double helix showing colour coded bases of cytosine, guanine, adenine, and thymine. Wiki image.

2.3 The Respiratory System

The respiratory system is divided into two parts: the upper respiratory tract and the lower respiratory tract. The upper respiratory tract includes the nose, mouth, and the upper trachea. The lower respiratory tract includes the lower trachea and the lungs, which are further divided into the bronchi, bronchioles and the alveoli.

The gas exchange surface area of the lungs ranges from 50 to 75 square meters, or approximately the size of a tennis court. The alveoli are small air sacs that have a single-layer membrane with blood capillaries in direct contact with this membrane. This is where the oxygen and carbon dioxide are exchanged in the lungs. Figures 2.10 and 2.11 show the lower respiratory tract and the alveoli.

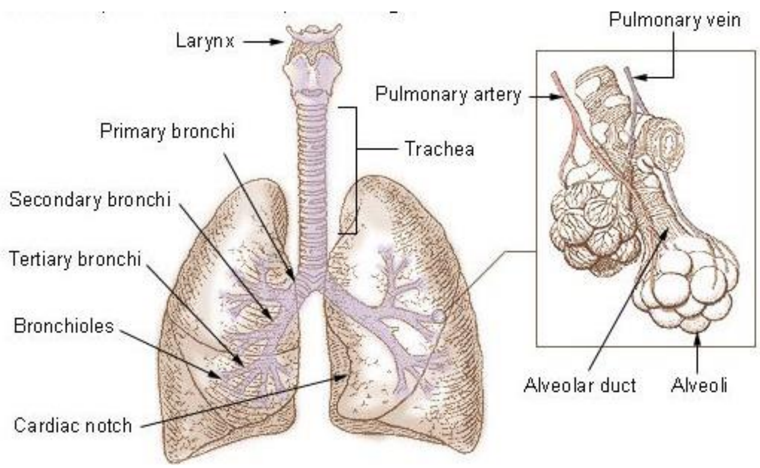

Figure 2.10: Drawing of the bronchi, bronchial tree and the lungs in relation to the size of the alveoli. Wiki image.

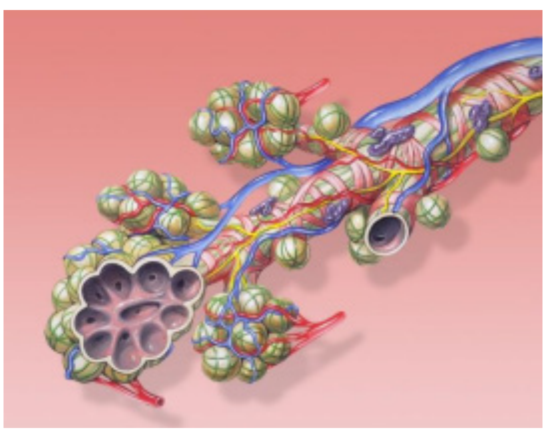

Figure 2.11. Expanded view of the alveoli showing the open air sacs and the capillaries in direct contact with the outside of the alveoli. Wiki image.

Inhaled airborne beryllium particulate can deposit in the lungs through the physical processes of interception, impaction, sedimentation, and diffusion. The location in the lung where the particles deposit and the percent captured depends on the size of the particles. The lung deposition curve (Fig. 2.12) shows that large particles are deposited mostly in the upper airways and the tracheobronchial regions, whereas the small particles deposit over all surfaces. It is a common misconception that small particles, here referred to as ultrafine particles (i.e., less than 100nm), are not deposited in the lungs. This is not the case and clearly 10nm particles are almost all (~90%) deposited in the lungs.

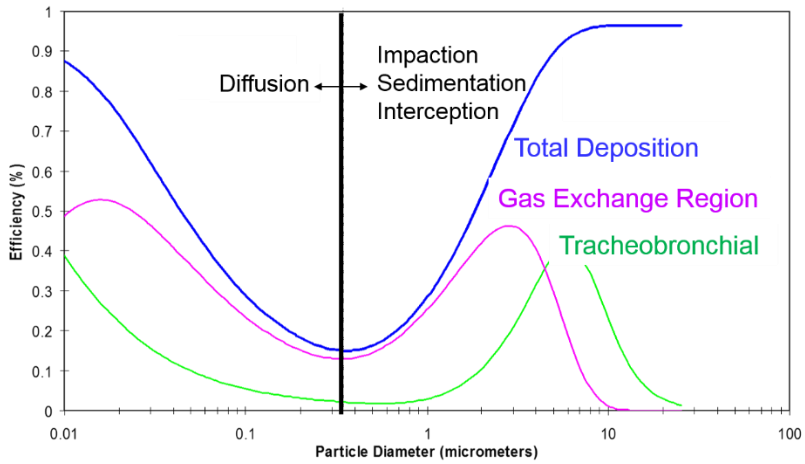

Figure 2.12: Lung Deposition Curve showing the regions in the lung that particles deposit versus particle size. Image courtesy of Michael McCawley.

3 Steps to Progression of CBD

The major events in the acquisition of CBD are initial sensitization through skin exposure combined with inhalation exposure to airborne beryllium particulate. An individual must be susceptible in order to acquire CBD. If the individual is not susceptible or does not have CBD specific genetic markers, they can receive higher exposures and still not acquire CBD. CBD genetic markers are specific chromosome coding for the body to make CBD specific cup-like or fin-shaped immune cell extensions.

This paragraph describes a more complete understanding of the specific known genetic expression, but the reader may choose to skip this. Susceptible individuals are known to have a variation at the human leukocyte antigen in the DP (HLA-DP) sub region that presents this gene coding for the Major Histocompatibility Complex (MHC) Class II antigen-bearing cells of T-Cells. This HLA complex is a series of genes located on chromosome 6. There is a group of HLA-DP variations called alleles where glutamic acid residue at position 69 is found in individuals with CBD. The symbol used for this is HLA-DPβ1^{E69}, but it is more commonly referred to as Glu69 (McCanlies, 2003, Falta, 2010). This specific code translates to a more electronegative receptor cup that can "bind" to or hold onto beryllium (Snyder, 2003).

However, while this marker is associated with individuals who have CBD, over 40% of the population have this marker. We know that roughly up to only 20% of the exposed workers acquire CBD, so there is more research needed before genetic markers can be relied upon as an indicator of susceptibility. Yet, if an exposed individual is a homozygote and has rare alleles, they can have as high as 70% probability of acquiring CBD. The allelic frequencies were higher for Chinese and Hispanics as compared to Caucasians and African Americans (Kreiss, 2016, Weston, 2002).

It is important to note the difference between the two terms susceptibility and sensitivity as these are a common cause for confusion and misunderstanding. Susceptibility is the ability for an individual to react to an antigen based on their genetic makeup, whereas sensitivity or sensitization is a reaction to an antigen in a susceptible individual. Simply stated, when the term sensitization is used, the individual has been exposed to an antigen and is reacting to it.

3.1 Skin Exposure

Langerhans cells, and T-Cells are the immune system cells that are involved in establishing the sensitization reaction to beryllium in the skin. Of course, beryllium particulate can go into the epidermis and dermis when there are wounds and intact skin. There have been studies to support the theory that beryllium particulate can penetrate the skin through common flexing of the skin (Tinkle, 2003).

However, a more logical means by which beryllium can enter the skin was presented by research that proposed that sweat can dissolve metal into ions which can then enter the skin (Stephaniak, 2010). This theory is strongly supported by a common skin sensitization reaction to nickel. There is ample evidence that nickel ions from solid nickel metal enter the body through skin contact (Satio, 2016). Sweat may not be necessary for the migration to occur.

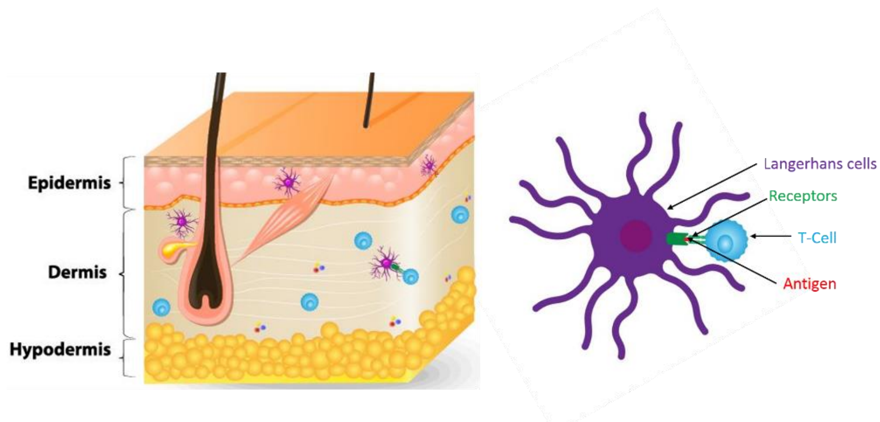

Figure 3.1. Schematic of the skin with the immune cells locations on left and names and shapes of immune cells described further in Section 3.

3.2 Immune System Recognition & Lung Exposure to Beryllium Particulate

Through skin contact with a solid beryllium-containing particle, the beryllium ions move into the epidermis. Langerhans cells in the skin's epidermis find the beryllium and become activated. The beryllium is bound to the Langerhans cells external antigen receptor. The Langerhans cell moves into the dermis layer and presents the beryllium to the naive T-Cell.

The naive T-Cell learns from the Langerhans cell that beryllium is a foreign invader or antigen and becomes a helper T-Cell, also called CD4+ T-Cell. The helper T-cell travels to the lymph nodes where the helper T-Cells activate the system to make an abundance of helper T-Cells (i.e., the cells proliferate). These helper T-Cells then circulate throughout the lymphatic system and the bloodstream and along with other locations, they can reside in the lungs. See Fig. 3.2.

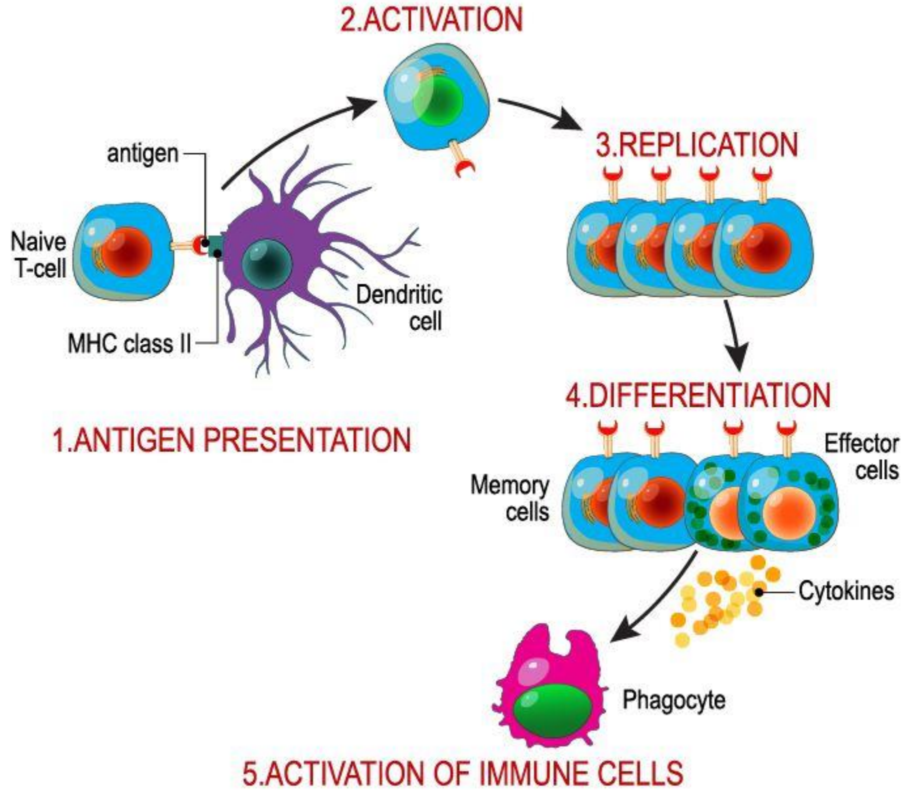

Figure. 3.2: Diagrame of the full process of immune cell reactions. Steps 1 and 2 occur in the skin, step 3 occurs in the lymphatic system and the lung, and steps 4 and 5 occur in the lungs.

Once the sensitized individual has beryllium particulate deposited in the lungs, the immune cells find the particles or ions. A helper T-Cell will then multiply and at the same time excrete cytokines, which are signals for other cells such as macrophages to come investigate.

Macrophages blindly move in the lungs and will encounter and engulf beryllium particles. With CBD, multi-nucleated giant cells develop when the macrophage engulfs the beryllium particle. See Fig. 3.2. Giant cells are seen in sarcoidosis patients as well as CBD and hence the reason that CBD is easily misdiagnosed as sarcoidosis. (Müller-Quernheim, 2006) The alveoli sacs are where the most damage occurs, where giant cells become surrounded by additional immune cells to form what is termed a non-caseating granuloma. This group of cells can be described as 3-dimensional sphere-like accumulation where the cells form onion-like layers. The alveoli become clogged with a mass of immune cells that die in the process of trying to remove the beryllium. Fibrosis results in the most progressive cases which is similar to asbestosis and silicosis.

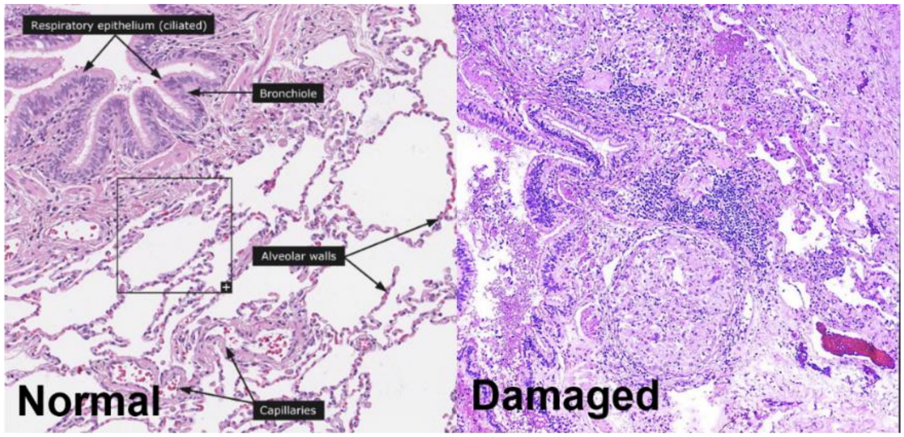

Fig. 3.3: Normal cells and damaged cells. A giant cells with multiple nuclei is noted. Wiki images.

4 Understanding Beryllium Exposure Risk

There are two primary exposure risks for beryllium, contact with the skin and inhalation of particulate. Skin contact with particulate is a risk especially if it stays on the skin for long periods of time. Higher incidence of sensitization has been shown when the skin is wet, such as in operations like plating, pickling, and ultrasonic bath parts cleaning. Further, skin exposure to soluble beryllium salts such as beryllium nitrate can also be a risk for acquiring beryllium sensitization.

There are differences in CBD risk for beryllium oxide (ceramic), beryllium metal, and beryllium salts. The more soluble the chemical is, the less risk there is since soluble particles are more likely to dissolve and therefore cannot be engulfed by a macrophage. Beryllium oxide is the most insoluble and therefore the highest risk. It is important to note that the naturally occurring form of beryllium, which is a form of beryllium aluminium silicate of beryl, beryllide, bertrandite, etc. is not a known risk.

All beryllium regulatory limits for beryllium are based on mass or weight. However, CBD is an immune system response with damage beginning with individual particles deposited in the lung. Given that immune cells move blindly, the probability of the cells finding a particle is increased if the number of particles increases. It is not likely that a large particle has more risk than a small one for this type of disease.

One million 100nm-sized particles have the same weight as one 1µm particle. It has been proven that the mass-based exposure limits are not predictive of CBD (McCawley, 2001). Further, it has been proven that average airborne beryllium exposure levels as low as 0.02µg/m^3 can cause CBD (McCawley, 2001, Schuler, 2005). A better indicator of risk is that of airborne beryllium particle number and in relation to the deposition rate for the specific particle size. It is unfortunate that standard exposure monitoring methods do not measure beryllium particulate number and the author speculates that this is the primary reason that regulatory limits in the US are not particle number based.

DOE's proposed action level is set at 0.05µg/m^3 (DOE, 2016) and in private discussions with the authors (Creek, 2017) they reported that this level was established in order to prevent any airborne beryllium. This is supported by Dr. David Michaels' paper entitled "Beryllium's Public Relations Problem: Protecting Workers When There Is No Safe Exposure Level" (Michaels, 2008). Thus, DOE's proposed action level takes into consideration the analytical detection limit. Therefore, this limit was set to allow the measurement to be technically feasible. DOE is not under the same requirement as is OSHA to evaluate economic feasibility of proposed standards. DOE is both the regulator and the funding source for work performed by DOE contractors. In contrast, OSHA is required to prove economic feasibility and has set their action level at a higher level of 0.1μg/m^3 (OSHA, 2016). While this standard will reduce the prevalence of CBD, it is almost a certainty that it will not eliminate it. Further, companies following rote regulatory compliance will focus on operations that have the higher weight-based exposures and may ignore other operations that could potentially be a higher risk.

To fully understand the risk of an operation's airborne exposure, an evaluation of airborne particle number as well as size should be conducted. While a method is available to determine total deposited particle number, some of the equipment used for this method is expensive and not standard to most occupational health practitioners (McCawley, 2009). Yet even without these tools, a subjective evaluation can be made. Operations that involve high speed and high heat as well as those wet operations that generate bubbles (plating, ultrasonic bath) will generate large numbers of ultrafine particulate.

4.1 Beryllium-Containing Alloys

It is a common misconception that copper-beryllium operations do not result in enough exposure to cause CBD, given that copper-beryllium has a low percentage of beryllium (<2%). It has been well documented in multiple studies and with health outcomes noted in copper-beryllium industry disease rates that exposure to copper-beryllium alloys can cause CBD (Schuler, 2005, Maier, 2019, Thomas, 2009).

Another misconception is that skin exposure to copper beryllium plays no role in initiating the disease. The beryllium in copper beryllium is no different than beryllium metal. Copper beryllium is an alloy, not a chemical compound where two elements are bound to each other. Fig. 3.5 shows an SEM image of copper-beryllium where the beryllium metal is a gold color. Therefore, exposure to beryllium particulate whether it comes from the beryllium metal or the beryllium metal alloy is still a risk. The only difference in the risk is that the contamination level of a solid alloy would be less, but it is still high enough to be a concern.

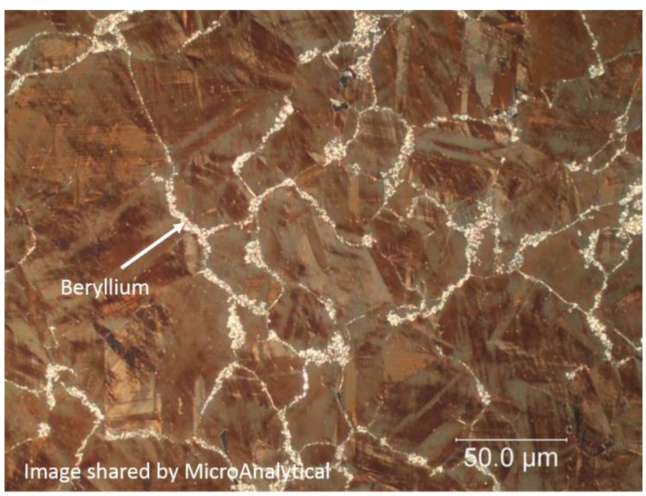

Fig. 3.5: Scanning electron microscope image of copper beryllium alloy.

While it is believed that wet operations are a control method for airborne particulate, if the operation involves high speed processes such as machining, small mist/beryllium particles can be easily generated. Exposure can result from inhalation of the mist that contains beryllium particles. Further, the mist can evaporate resulting in only beryllium particles that can also be inhaled (Donaldson, 2019). This is especially true for lathing operations of large-diameter parts. Cutting fluid is recycled, can easily become contaminated, and the filters are not designed to remove the ultrafine particulate. Operations that involve heat are also an exposure risk. As quoted from Donaldson, 2019, electron discharge machining (EDM) generates a very fine smoke and fume that can be seen rising out of the pool of dielectric oil. Small particles can linger in the air. As an example, it takes over 13 days for a 100nm particle to fall one meter.

Dust contaminated with man-made beryllium can become resuspended and can be a primary risk for locations where operations have ceased but the contaminated equipment and room surfaces remain. DOE establishes a surface contamination housekeeping limit of 3μg/100cm^2, requiring work surfaces to be below this level at the end of a shift (DOE, 1999).

One final exposure risk not discussed earlier is the risk to mucous membranes. While it is not well documented, it is possible that beryllium exposure to mucous membranes such as the eye mucosa are a risk to developing sensitization. This is somewhat supported in that conjunctivitis was prevalent during the time period from 1930-1960. The fact that conjunctivitis develops from exposure to beryllium shows that the mucous membranes have some ability to react to beryllium (Creek, 2001). Therefore, the eyes should be protected from contaminated hand to eye contact and for exposures above the exposure limit to the eye conjunctiva by using respirators that protect the eyes.

5 History of CBD in the DOE Complex

While there are many excellent articles on various industrial exposures and the risk of disease, (Michaels, 2008, McCawley, 2001 to name a few) the best lessons learned come from experience with the DOE and its contractor sites. This historical perspective is given to allow the reader to understand what it means to have unchecked exposure risks and not provide sufficient medical surveillance or exposure monitoring for workers. Prior to 1983, the hazard of beryllium within the DOE Complex was thought to be adequately controlled. Sites had administrative or policy documents that included control of beryllium exposures. Worker exposure monitoring was conducted on a routine basis at most of these DOE sites. Medical surveillance testing had not uncovered any disease using standard testing such as chest X-rays and pulmonary function tests.

Concerns started to arise when Rocky Flats Environmental Test Site had their first case of CBD diagnosed in 1984. This was thought to be an anomaly from some high exposure event. The second case in the early 1990s, however, was the triggering event to DOE's initiating beryllium worker studies, which included testing the workers with the Beryllium Lymphocyte Proliferation Test or BeLPT.

This second case has many interesting aspects in that the worker was a semi-professional soccer player and had trained in the Rocky Mountains. He had at one time been in excellent health where his high-altitude training resulted in a very high lung capacity (>140%). The worker describes how medical doctors would marvel at his chest x-ray, commenting that his lungs were so large that a non-standard size x-ray film had to be used. Yet as a Rocky Flats worker in his 40's, he started to experience shortness of breath during his sports activities. He consulted his private physician as well as Rocky Flat's physicians. Company doctors did not believe the individual's condition was work related and his private doctor was not knowledgeable about CBD. The worker went to a local facility that specializes in pulmonary diseases, National Jewish Health, where he was diagnosed with CBD.

During this timeframe, the DOE believed that beryllium exposures at Rocky Flats and other DOE facilities were well controlled. Many of the sites had conducted beryllium machining operations since the 1960s. However, the common sampling method used fixed-area air samplers based on radiation protection methodology. The samples were collected well outside the breathing zone. Although the results were below the Occupational Safety and Health Administration Permissible Exposure Limit (OSHA PEL) of 2μg/m^3 8-hour Time-Weighted Average (TWA) used at the time, the workers could have had substantially higher exposures. The method of sampling underestimated the exposure levels.

There is a picture of the Rocky Flat's beryllium machinists inside the beryllium machine shop having a holiday party and holding a long submarine sandwich. Over half of the workers had beards showing respiratory protection was probably not worn. This photo emphasizes that the workplace controls were insufficient. Nonetheless, Rocky Flats second case contributed to the DOE's decision to perform the Beryllium Worker Studies and provide the BeLPT to those who had worked in or accessed the beryllium areas.

When the results revealed beryllium sensitization and CBD at all the sites in the initial studies, the DOE issued the first expanded regulatory standard in the world, with an action limit set at 0.2μg/m^3 (DOE, 1999). The standard included the requirement to provide the BeLPT to the exposed population. Today, it is known that nearly 75% of the current sites tested (20 out of 27 locations or companies), have workers that are sensitized and 50% of the sites have cases of CBD (ORISE, 2017). Further, the numbers in the ORISE reports do not reflect the total number of cases since they don't report former workers or current workers that are bypassing their employer's system by using the Energy Employees Occupational Illness Compensation Program (DOL, 2000).

Some of the reasons for limited recognition of beryllium hazards during the early 1990s at DOE sites are discussed. All sites had medical surveillance programs, with most having board certified occupational physicians and certified industrial hygienists. These professionals were of the opinion that their program should have identified or uncovered a problem if one existed. As mentioned, they believed that the exposure limit was protective. It was believed that since exposure levels were below the regulatory limit the workers would not acquire the disease. There was no or little consideration of low percentage beryllium alloys as being a potential risk. There was loss of personal testimony due to retirement or employees relocating. They did not anticipate significant contamination or risk for locations where only finished components were handled. There were complications due to classified work.

To further elaborate, the excuses included statements like "we don't have beryllium on-site", "we only use copper beryllium", "we only handle the finished parts", and "we monitor for radiation and it should pick up the problem if we have one". However, the reality was that sites that stated they didn't have beryllium, used it in rocket fuel research and as a nuclear moderator for nuclear reactors. The site that used radiation monitoring as their control method, conducted open air explosive testing of assemblies containing beryllium parts. This location is highly beryllium contaminated and resulted in at least one very severe case of CBD.

Some of these sites turned out to be in the top 5 of the highest number of CBD cases within the DOE complex. In fact, when one facility started investigating, the industrial hygienists set up the equipment calibration area (a location that is supposed to be contaminated free) in one of the most contaminated areas within the facility. The individuals that performed the evaluations were Certified Industrial Hygienists (CIH), yet in general, surface sampling in the IH field is rarely done. Further it was discovered that the facility that only handled beryllium parts had highly contaminated parts containers. Handling of the contaminated containers generated enough exposure to cause disease as can be seen in the ORISE report (ORISE, 2017).

The point to be made here is that for the majority of today's beryllium processing facilities, there is probably a risk of workers getting CBD. Beryllium facilities across the globe can apply this DOE experience to their processes. While DOE has some unique work, the levels of contamination would be similar in private industry if not higher as shown by the France worker studies (Vincent, 2009, Rousset, 2019). Responsible employers should be obligated to provide the BeLPT to all workers that go into the processing area. The BeLPT is discussed in the next section.

6 Diagnosis of CBD

Diagnosis of CBD for involves two primary steps, first the determination if the individual is beryllium sensitized and then a medical follow-up for those diagnosed with beryllium sensitization. It is well established by the experience of the DOE that the only definitive way to diagnose sensitization and CBD is by using the Beryllium Lymphocyte Proliferation Test or BeLPT. "Absence of evidence is not evidence of absence" (Michaels, 2008). Without the use of the BeLPT, CBD can and is being misdiagnosed as sarcoidosis, Chronic Obstructive Pulmonary Disease, and Hypersensitivity Pneumonitis (Müller-Quernheim, 2006, Maier, 2019). Sarcoidosis is a disease of unknown cause. One author suggests that sarcoidosis is CBD and recent comparisons of the two continues to show how the two diseases have a strong overlap (Rossman, 2003, Culver, 2016).

6.1 Definition of Beryllium Sensitized or BeS

Once again, terminology is important, and the definition of beryllium sensitized or BeS can vary depending on the organization that is using it. However, the golden definition by the Energy Facilities Contractor Operating Group (EFCOG, 2008) and further simplified by (Maier, 2019) for beryllium sensitization are test results as follows:

- 2 Abnormal BeLPTs
- 1 Abnormal and 1 Borderline
- 3 Borderline BeLPTs
- Abnormal Bronchoalveolar Lavage (BAL) BeLPT

The EFCOG's BeLPT Interpretation Guide is a useful resource to help determine the next step for a borderline or abnormal test result. The BeLPT is a blood test that uses live cells. The blood cells are exposed to a beryllium salt. If a person is sensitized, their cells will multiply (or proliferate) and this is called a BeLPT+ or an abnormal result.

6.2 Routine Exam

The medical tests and procedures conducted that are standard for a beryllium medical surveillance routine exam are as follows:

- Medical and work history with emphasis on past, present, and anticipated future exposure to beryllium
- Respiratory symptoms questionnaire
- Physical examination with special emphasis on the respiratory system, skin, and eyes
- Low dose computed tomography scan when recommended
- Pulmonary function test (spirometry) for forced vital capacity and forced expiratory volume (FEV1)
- Beryllium lymphocyte proliferation test (BeLPT, i.e. the blood test)

- Other tests deemed appropriate by the examining physician for evaluating beryllium-related health effects
- Follow up testing for BeLPT+, borderline, or uninterpretable cases
- Medical follow up for BeLPT+ cases

6.3 Medical Follow-Up for BeS Cases

The medical follow up for those diagnosed as Beryllium Sensitized (BeS) involves invasive tests and should be performed by a medical facility that has experience diagnosing pulmonary diseases preferably one that has experience diagnosing CBD. The following test and procedures are conducted:

- Chest radiograph (B reading) or High-Resolution Computed Tomography
- Pulmonary function testing
- Diffusing Capacity of the Lung for Carbon Dioxide (DLCO)
- Exercise Physiology
  - Workload, Maximum Oxygen Update (VO2 max)
  - Gas exchange (rest, exercise)
- Bronchoalveolar Lavage (BAL)
  - Percent lymphocytes
  - BAL LPT
- Lung biopsy with subsequent histopathology tests (Fig. 6.1 shows normal and abnormal alveoli cells)

The BAL is a test where a bifurcated tube (two branches) is placed in the lungs, one side is used for the patient breathing, the other side is filled with sterile fluid to collect (lavage) the cells for further testing.

The BeLPT has been repeatedly and incorrectly referred to a test that is plagued with inaccurate results. However today it is known that this test is as accurate or better than some of our standard screening tests such as those for breast, colon, and pancreatic cancers (Mroz, 2019). Further, some governmental organizations refuse to use this test because their position is that a blood test is too invasive. A blood test is a common medical procedure and even children are at times given blood tests to diagnose the more harmful diseases. Testing the workers is the only prudent thing to do.

These excuses fall short from a good justification especially when there is a valid risk of disease. No excuse is good enough to walk away from protecting workers from the risk of CBD. A more likely reason is that some organizations do not want to use the BeLPT because they fear what happened to DOE will happen to them. If an organization processes beryllium, they are probably right, it could happen to them. Initiating the BeLPT on an exposed population is not an easy or pleasant task but it is a necessary one.

Regarding frequency of testing, CBD has a long latency period. Therefore, the medical surveillance should be repeated in a set frequency (i.e., yearly) and ongoing to continue after employment but less frequent after exposures cease.

6.4 Tests Under Consideration

A final discussion on additional tests under consideration. While there is some use of beryllium urinalysis, this has not yet been demonstrated to be accurate at predicting exposure intake or dose. Many food and other products contain naturally occurring beryllium, which can be an interferant. Studies conducted by INRS in

France have used the exhaled breath condensate and tested for the immune cytokines of TNF-α (Rousset (2019). This test shows promise at being an effective screening test since a positive result indicates the worker is sensitized and reacting to beryllium in the lung.

7 Summary

Available evidence indicates that beryllium sensitization and disease occurs in over 75% and 50% respectively of all workplaces where beryllium and beryllium alloys are present. With increasing evidence that weight-based exposure limits are not effective at preventing beryllium sensitization and CBD, it is incumbent on employers to take additional measures to accurately identify processes and activities that present the greatest risks to workers.

Characterization of processes by particle number generated rather than total mass is the best method to accomplish this. Implementation of a medical monitoring program utilizing the BeLPT is a critical and necessary step to further identify worker groups at risk and allow for medical removal of sensitized workers, hopefully reducing the number of cases that progress to CBD.

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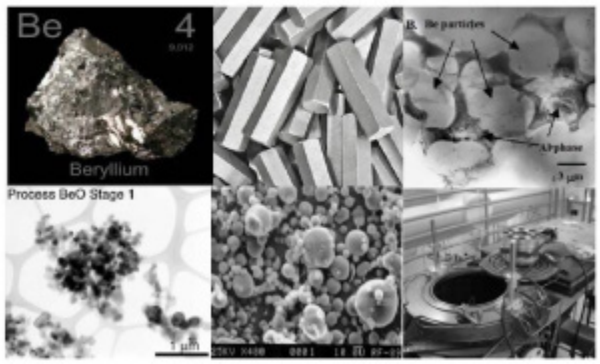
Identifying Beryllium Exposure Risks & Diagnosis of CBD

Kathryn Creek, CIH, MS



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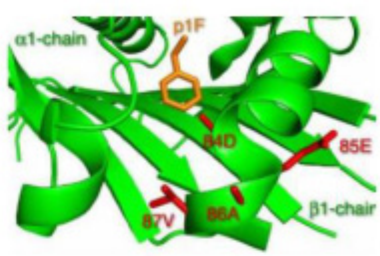
Outline

- Human Body Systems Involved in CBD
- Types of Immune System Reactions
- Steps of Progression to Chronic Beryllium Disease
- Understanding Beryllium Exposure Risks
- Diagnosing CBD



HLA-DPB1 antigen pocket

L. Silveira, CBC, HLA-DPB1, and the DP Peptide Binding Groove, Journal of Immunology, 2012

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Page 2

From the clear to the blurry then back again

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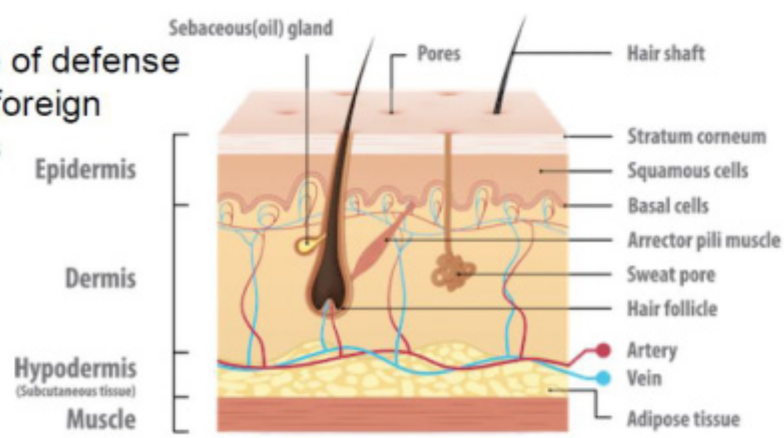
The Skin

- Largest organ in the body
- First line of defense against foreign invaders



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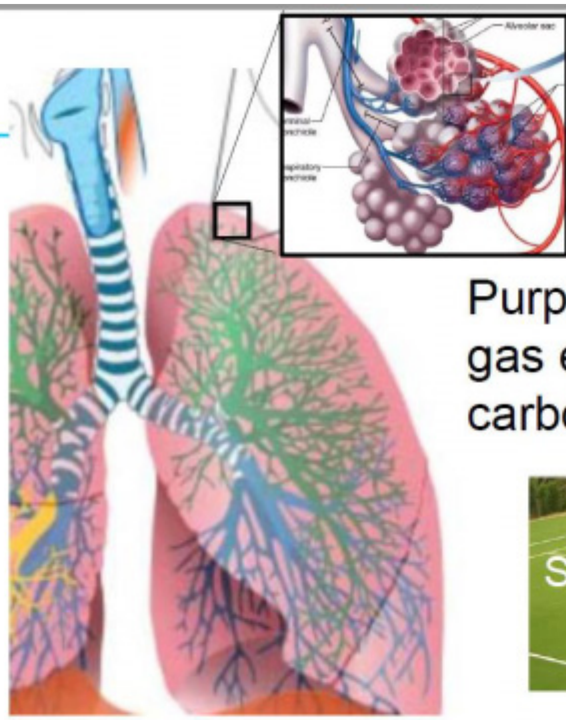
The Lungs



Purpose of the lungs is gas exchange (oxygen in, carbon dioxide out)

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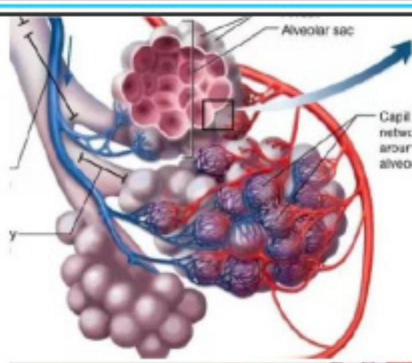
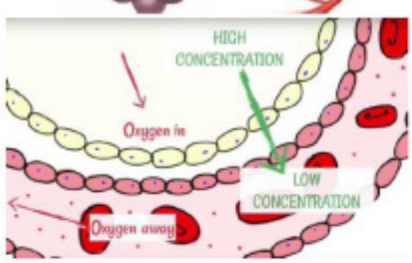
Gas Exchange Region





- Alveoli are air sacs with surrounding capillaries
- Particles, (especially ultra-fines particles deposit there
- There are a few cells in depth where oxygen is transferred to blood cells

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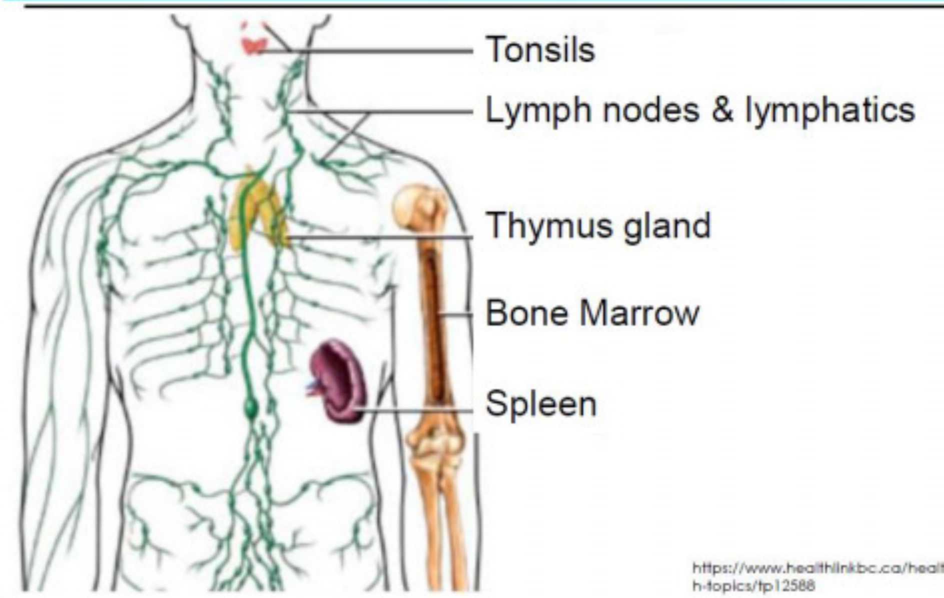
Immune System



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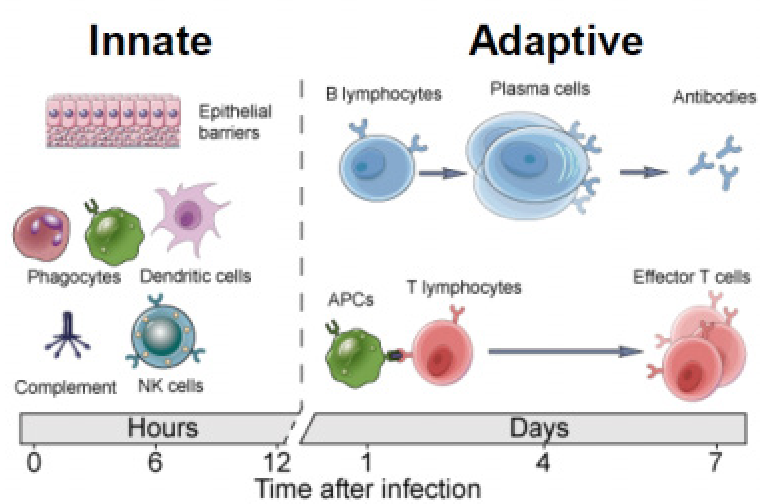
Protective Immune System Reactions



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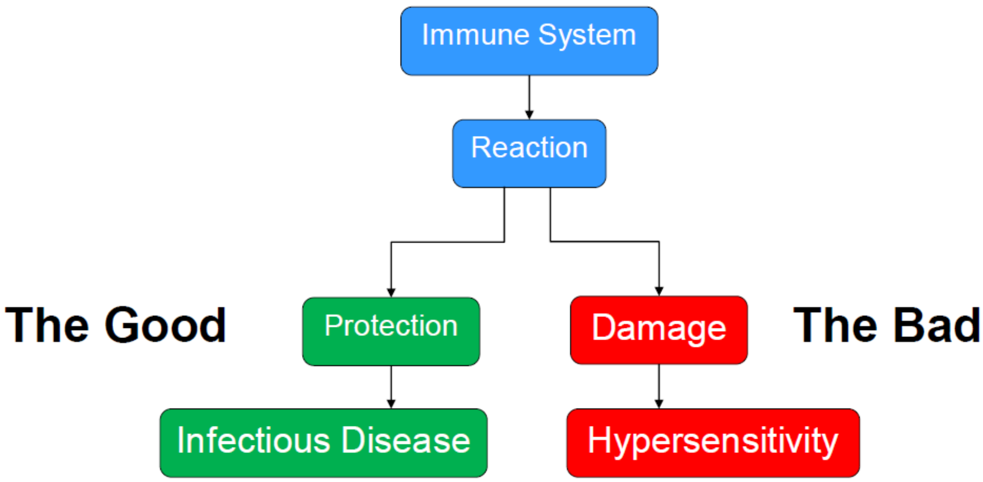
Reactions of the Immune System



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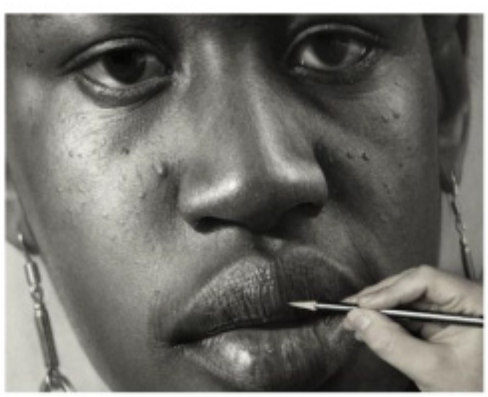
Hyperrealism Art



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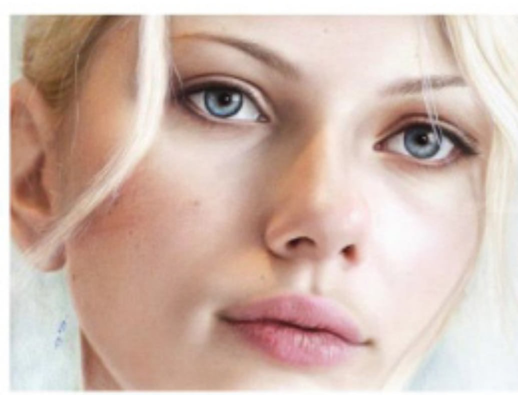

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Important Terms

T-Cell = T-lymphocyte

antigen = foreign invader

Susceptibility ≠ Sensitivity

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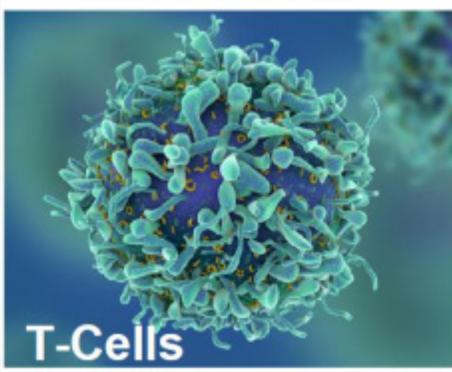
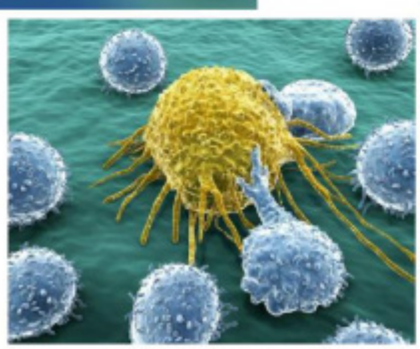
Immune Cells and Proteins



Wikipedia

Creative Biolabs



Macrophages, phagocytic cells

Phoenix Clinical Research

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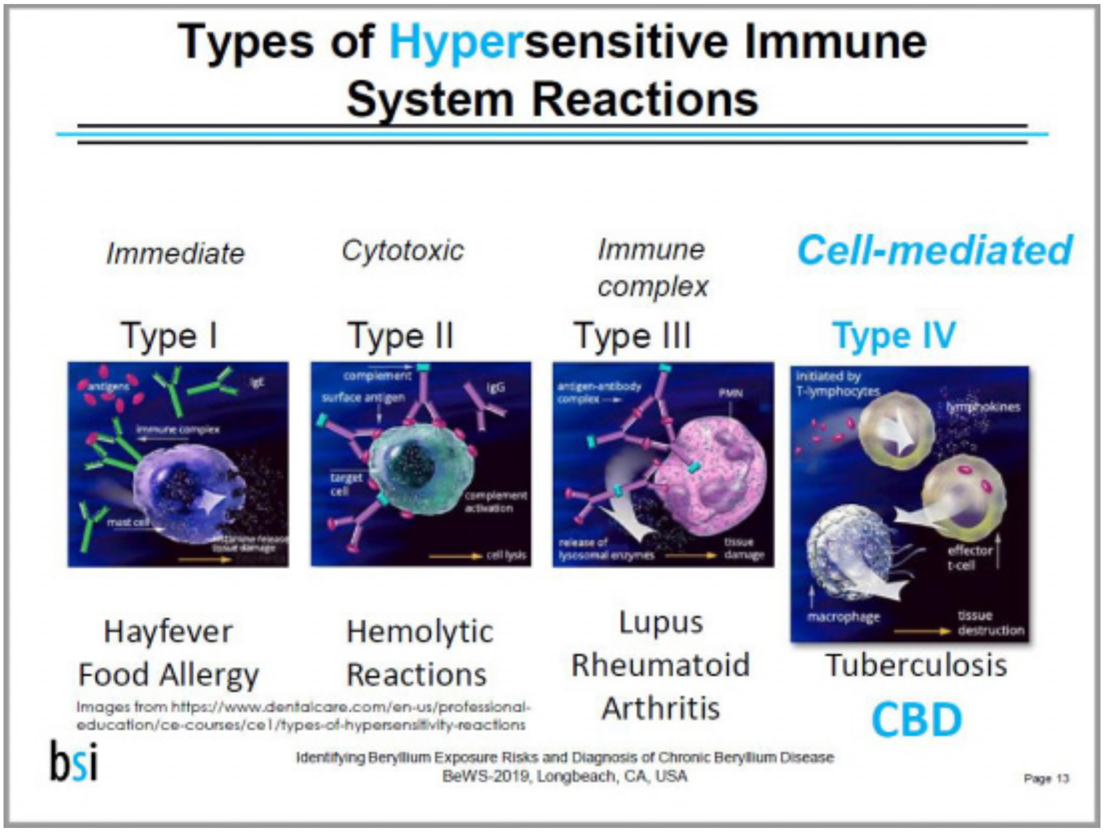
Types of Hypersensitive Immune System Reactions
Immediate
Type I
Hayfever
Food Allergy
Cytotoxic
Type II
Hemolytic
Reactions
Immune complex
Type III
Lupus
Rheumatoid
Arthritis
Cell-mediated
Type IV
Tuberculosis
CBD
Images from https://www.dentalcare.com/en-us/professional-education/ce-courses/ce1/types-of-hypersensitivity-reactions
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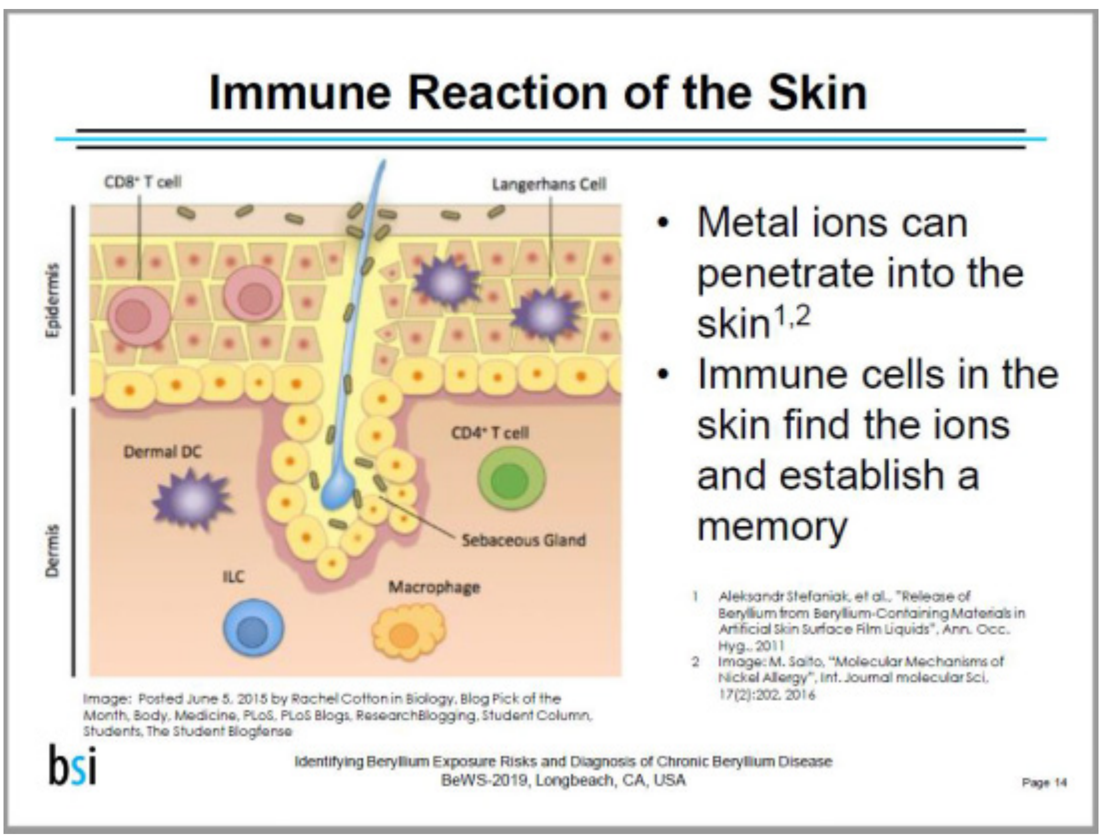
Immune Reaction of the Skin
CD8+ T cell
Langerhans Cell
Epidermis
Dermis
Dermal DC
CD4+ T cell
Sebaceous Gland
ILC
Macrophage
• Metal ions can penetrate into the skin1,2
• Immune cells in the skin find the ions and establish a memory
1 Aleksandr Stefaniak, et al., "Release of Beryllium from Beryllium-Containing Materials in Artificial Skin Surface Film Liquids", Ann. Occ. Hyg., 2011
2 Image: M. Saito, "Molecular Mechanisms of Nickel Allergy", Int. Journal molecular Sci. 17(2):202, 2016
Image: Posted June 5, 2015 by Rachel Cotton in Biology, Blog Pick of the Month, Body, Medicine, PLoS, PLoS Blogs, ResearchBlogging, Student Column, Students, The Student Blogfense
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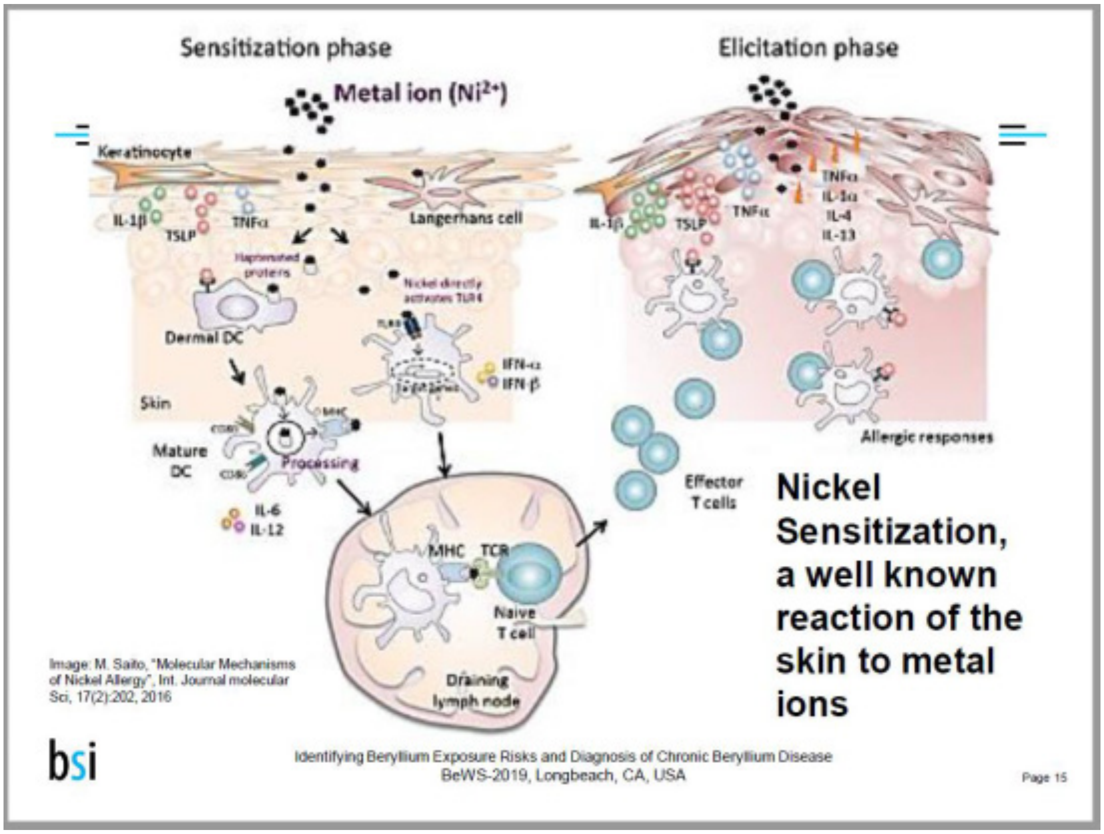
Sensitization phase
Elicitation phase
Metal ion (Ni2+)
Keratinocyte
IL-1β
TSLP
TNFα
Langerhans cell
Dermal DC
Skin
IFN-α
IFN-β
Mature DC
Processing
IL-6
IL-12
MHC
TCR
Naive T cell
Draining lymph node
Effector T cells
IL-1α
IL-4
IL-13
Allergic responses
Nickel Sensitization, a well known reaction of the skin to metal ions
Image: M. Saito, "Molecular Mechanisms of Nickel Allergy", Int. Journal molecular Sci, 17(2):202, 2016
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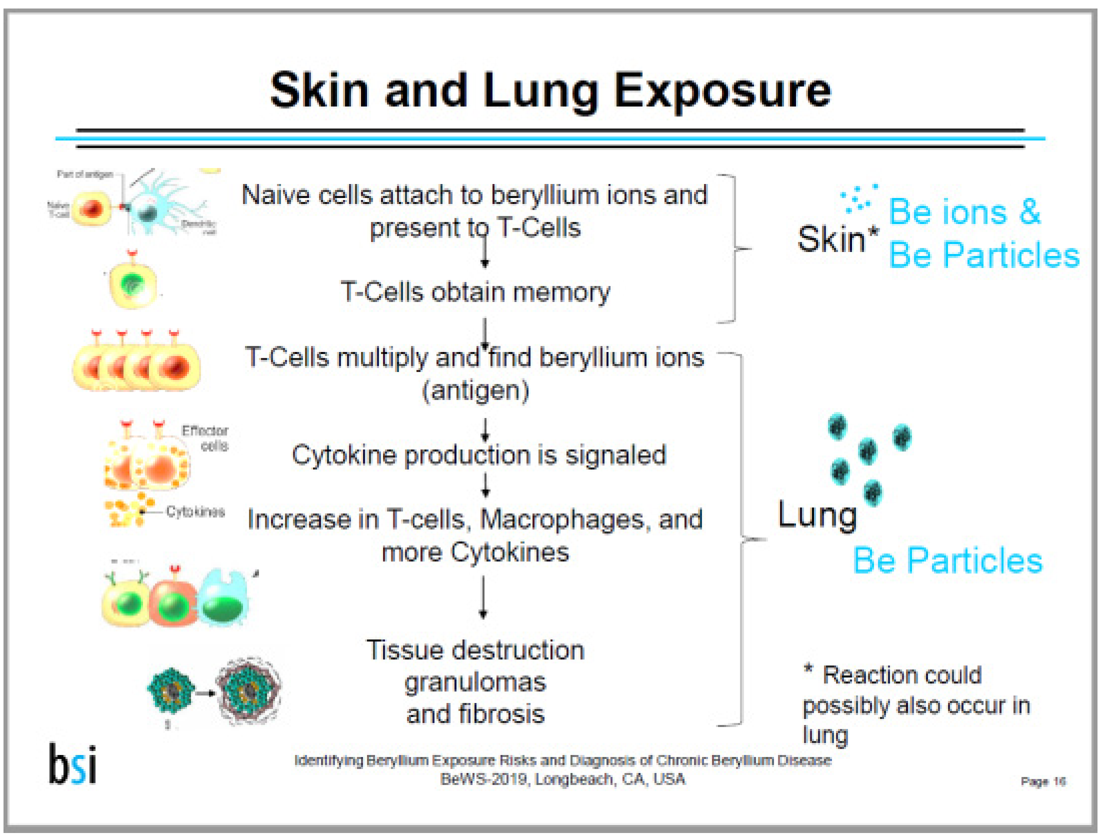
Skin and Lung Exposure
Naive cells attach to beryllium ions and present to T-Cells
T-Cells obtain memory
T-Cells multiply and find beryllium ions (antigen)
Cytokine production is signaled
Increase in T-cells, Macrophages, and more Cytokines
Tissue destruction granulomas and fibrosis
Skin*
Be ions & Be Particles
Lung
Be Particles
* Reaction could possibly also occur in lung
Effector cells
Cytokines
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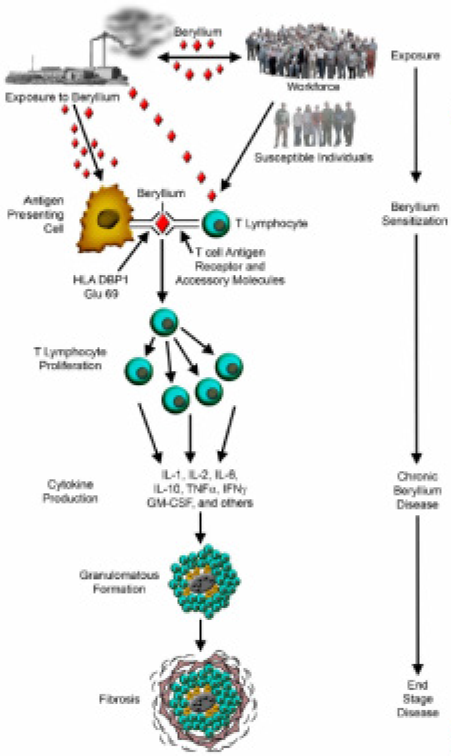
Progression to CBD



Not everyone reacts to beryllium

L. Maier, "Beryllium Disease, Chapter 65, Occupational and Environmental Medicine, 2006

isks and Diagnosis of Chronic Beryllium Disease
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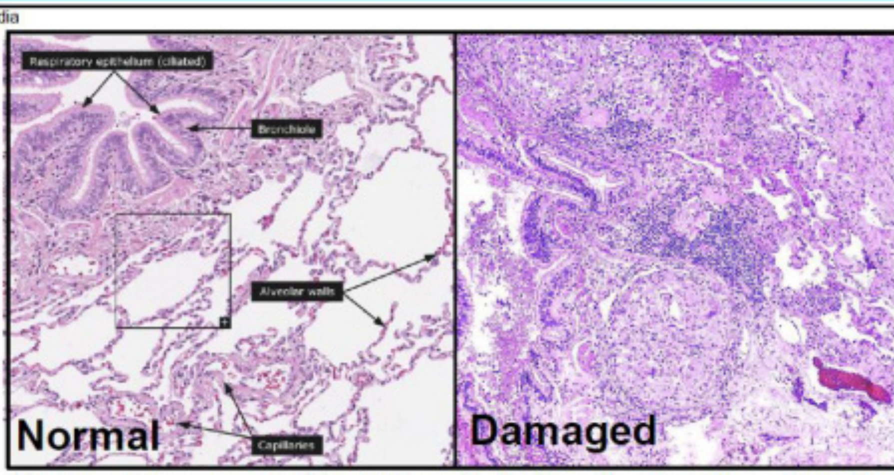
Damage to Lung Tissue

Wikipedia



Progressive amount of immune cells in the air sacs form onion-like (non-caseating) granulomas

Granulomas result in reduced oxygen uptake

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Genetic Susceptibility

LTWA ($\mu g/m^3$)	E69-	Single *02 Allele	Single $E69^{+}$ non-*02 Allele	E69 homozygote
0.02 $\mu g/m^3$	1.02 (1.00-1.03)	3.52 (1.45-8.57)	12.16 (5.20-28.45)	22.90 (7.11-73.83)
0.05 $\mu g/m^3$	1.04 (1.01-1.07)	3.61 (1.48-8.77)	12.45 (5.32-29.14)	23.46 (7.27-75.69)
0.10 $\mu g/m^3$	1.08 (1.02-1.15)	3.75 (1.54 -9.14)	12.96 (5.53-30.37)	24.41 (7.55-78.96)
0.20 $\mu g/m^3$	1.18 (1.04-1.32)	4.06 (1.66-9.95)	14.03 (5.95-33.07)	26.43 (8.11-86.11)
0.50 $\mu g/m^3$	1.49 (1.10-2.02)	5.16 (2.02-13.16)	17.82 (7.25-43.81)	33.56 (9.90-113.76)
1.0 $\mu g/m^3$	2.22 (1.21-4.07)	7.68 (2.63-22.43)	26.52 (9.38-75.02)	49.95 (13.07-190.88)
2.0 $\mu g/m^3$	4.91 (1.46-16.56)	17.01 (3.80-76.17)	58.77 (13.43-257.2)	110.7 (19.78-619.3)

Van Dyke et al. Am J Respir Crit Care Med, 2011

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Speciation of Beryllium and Risk

- Beryllium oxide
- Beryllium metal and alloys
- Beryllium salts

Risk is higher for exposure to oxides and metals

Risk

- Naturally occurring
  - Beryllium aluminum silicate
  - Beryllium silicate

No known CBD risk to naturally occurring beryllium

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Beryllium occupational exposure limits are based on weight or mass of beryllium inhaled

The limits are very very low

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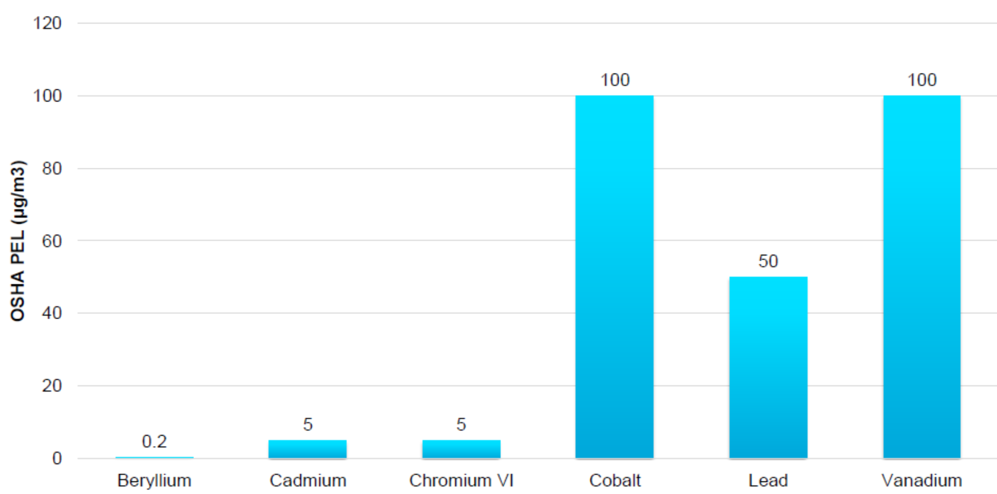
Comparison of OSHA Permissible Exposure Limits



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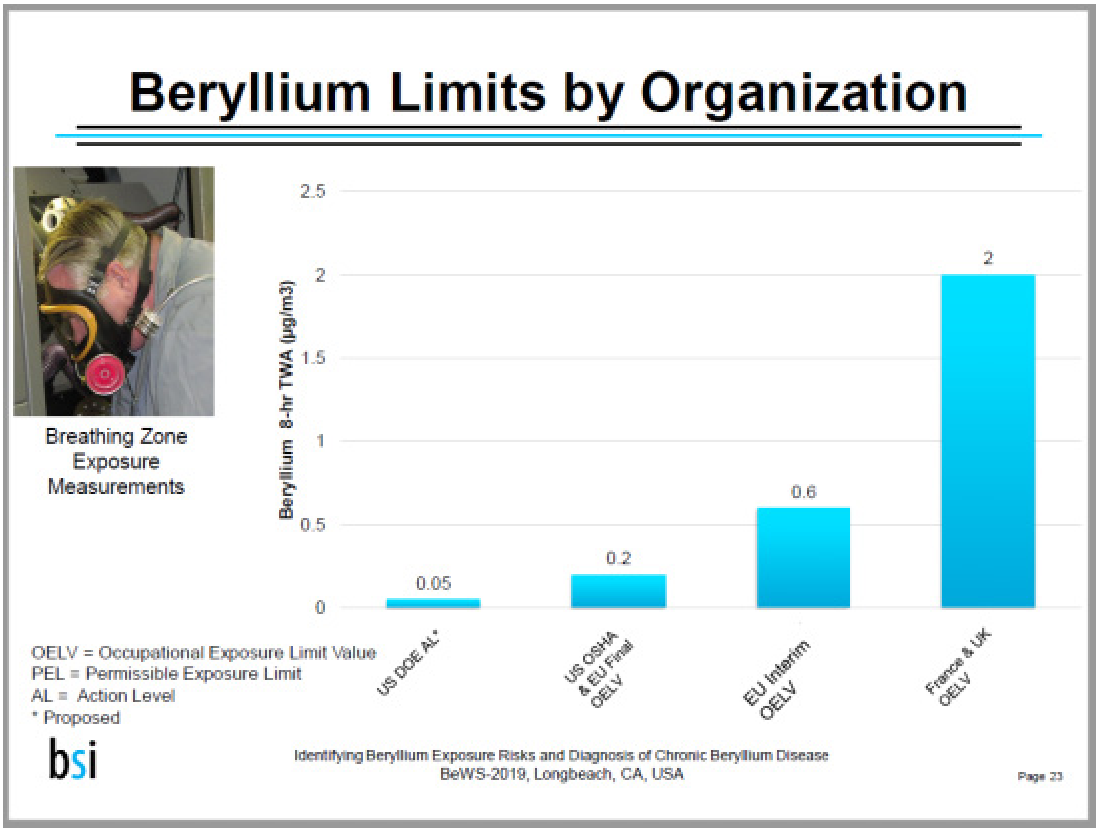
Beryllium Limits by Organization
Breathing Zone Exposure Measurements
Beryllium 8-hr TWA (µg/m3)
2.5
2
1.5
1
0.5
0
0.05
0.2
0.6
2
US DOE AL*
US OSHA & EU Final OELV
EU Interim OELV
France & UK OELV
OELV = Occupational Exposure Limit Value
PEL = Permissible Exposure Limit
AL = Action Level
* Proposed
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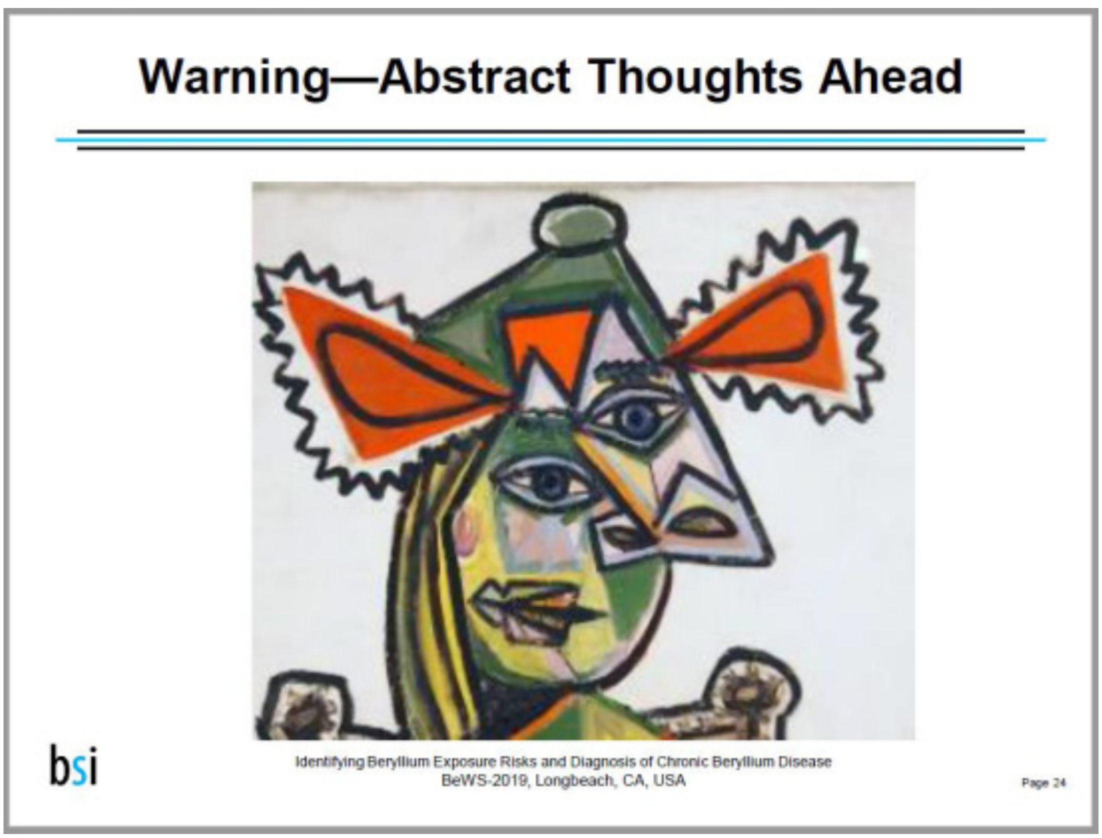
Warning—Abstract Thoughts Ahead
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Indicators of Risks

While exposure limits are based on weight or mass of inhalation, this may not be the best indicator of risk

Exposure Risk

Total beryllium particle number inhaled is a probable indicator of risk combined with skin exposure

M. McCawley et al., “Ultrafine Beryllium Number Concentration as a Possible Metric for Chronic Beryllium Disease Risk”, App. Occ. & Env. Hyg., 2001, 16(5)

This may be a matter of statistics from a macrophage finding a beryllium particle in the lung

Are These Airborne Particles?

Albuquerque Balloon Festival

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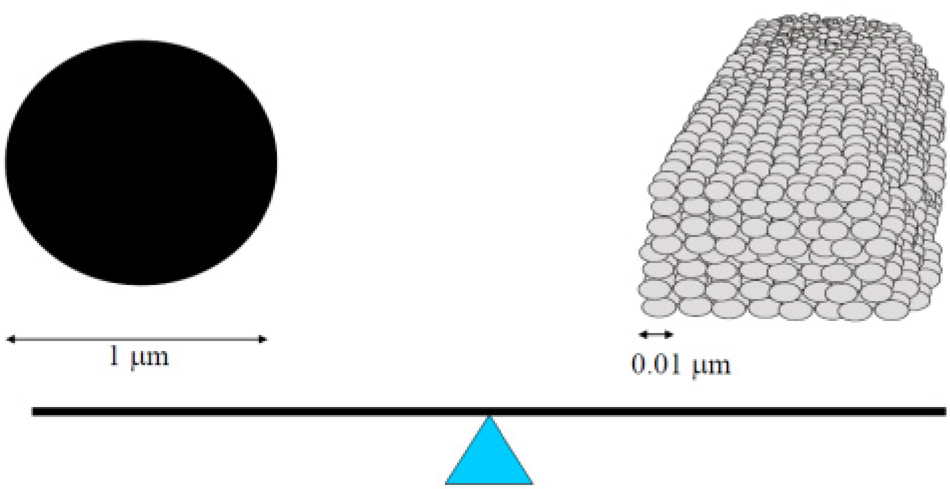
Ultra-fine Particles don't weigh much!

1 particle

1,000,000 particles



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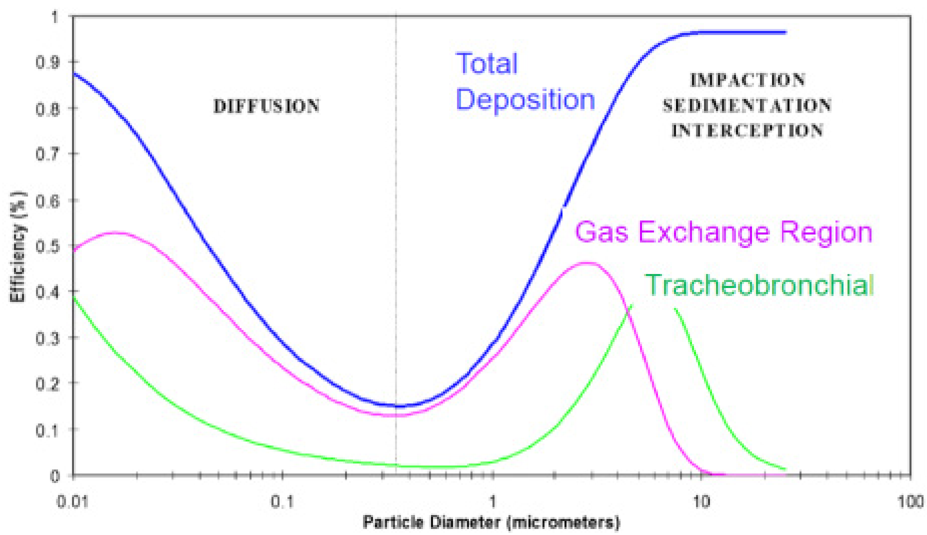
Lung Particle Deposition Curve



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NIOSH Study on Risk

M.M^cCawley, "Ultrafine "Beryllium Number Concentration as a Possible Metric for Chronic Beryllium Disease Risk", App. Occ. & Env. Hygiene, 2001

	Deposited Be Particles / cc	Average Be Mass ug / m^3
Oxide furnace area	9900	0.015
Fluoride furnace area (Average)	9700	0.25
Cast shop	3700	0.036
Hydrolysis / filling area	1800	0.19
Wet plant (Processing Facility)	1400	0.058
Reduction furnace area	300	0.059
Bulk pickle in strip mill	200	0.075
Ball mill (Processing Facility)	100	0.074
Beryl ore mine	9	0.24
Ore crusher	5	0.077
Milling operation	3	0.004
Administration and shipping	1	0.001

Increase in disease ↑

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Copper Beryllium Workplace Risk

- NIOSH study evaluated a copper beryllium strip and wire finishing facility
- The rates of BeS and CBD were 7% and 4% respectively

C. Schuler et al.,"Process-Related Risk of Beryllium Sensitization and Disease in a Copper-Beryllium Alloy Facility", *Am J Ind Med*, 2005, 47:195-205

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Identifying Risks in the Workplace

- Airborne exposure evaluation of job categories or locations that includes weight-based (compliance) and particle number based techniques
- Skin contamination evaluation
- Surface contamination evaluation in non-beryllium areas to protect support staff (secretaries, finance, human resource staff)
- Observation of worker habits and practices

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Diagnosing CBD

- Medical and work history with emphasis on past, present, and anticipated future exposure to beryllium
- Respiratory symptoms questionnaire
- Physical examination with special emphasis on the respiratory system, skin, and eyes
- Low dose computed tomography scan when recommended.
- Pulmonary function test (spirometry) for forced vital capacity and forced expiratory volume (FEV_1)
- Beryllium lymphocyte proliferation test (BeLPT)
- Other tests deemed appropriate by the examining physician for evaluating beryllium-related health effects
- Follow up testing for BeLPT+, borderline, or uninterpretable cases
- Medical follow up for BeLPT+ cases

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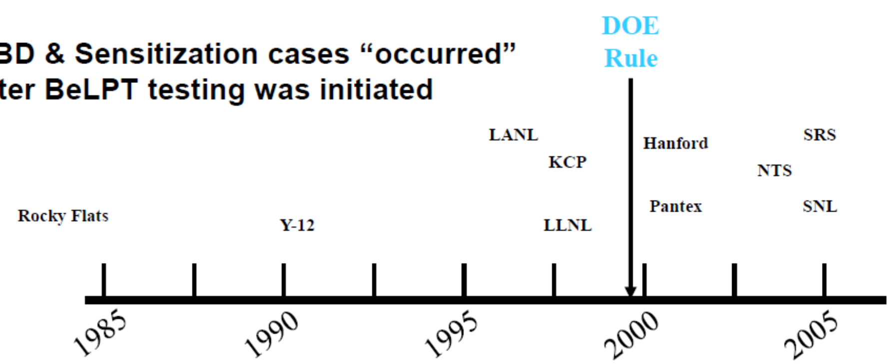
Chronology of Cases in DOE

Use of BeLPT is key for diagnosing CBD

CBD & Sensitization cases "occurred" after BeLPT testing was initiated



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And here we are today!

- Blood tests are a very common method for diagnosing illnesses and for the basic yearly occupational medical examination
- Governments excuse that a blood test is too invasive is questionable
- Efficacy of the BeLPT is comparable to screening tests for breast and pancreatic cancer so excuse of test not being sound is unjustified
- Only four medical laboratories are known in Europe to conduct the BeLPT

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Studies in France by INRS

Exhaled Breath Condensate (EBC) Analysis

- **Markers of oxidative stress**: 8-IP, MDA, NOx, 3-NT
- **Markers of inflammation**: total proteins, TNF-α

→ EBC shows potential for identifying body's reaction to beryllium in the lung

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Conclusions

- Body systems of the skin, immune system, and lung are involved in the development of CBD
- CBD can result from skin exposure and inhalation of particulate
- An effective risk evaluation should include particle number or assessment of ultra-fine particulate concentrations as well as the compliance weigh-based limit for effective risk
- The BeLPT should be used as a leading measure for control verification and for diagnosis of CBD

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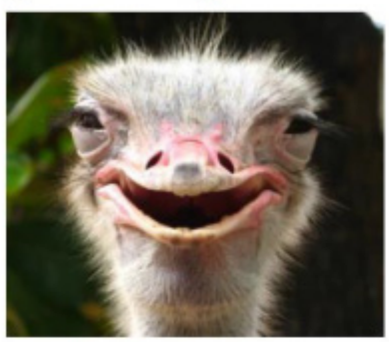
Thank you for your attention



www.beryllium-solutions.com
creek@beryllium-solutions.com

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Key Elements of a Successful Beryllium Control Program

Kathryn Creek, CIH

Beryllium Solutions International LLC, Los Alamos, New Mexico, U.S.A.

The regulatory requirements for beryllium control have increased in Europe and the United States. The European Commission has proposed the Directive 2004/37/EC for the protection of exposure to carcinogens, including beryllium with a lowering of the current exposure limit to 0.2μg/m^3 following a seven-year grace period at 0.6μg/m^3.

The U.S. Department of Labor, Occupational Safety and Health Administration (OSHA) published their expanded standards for General Industry, Construction, and Maritime Standards on January 9, 2018. Portions of the Standards came into effect in May 2018, including lower permissible exposure limit of 0.2μg/m^3 which is 10% of the previous limit.

Further requirements for the standard include establishing regulated areas, conducting exposure assessments and exposure monitoring, written exposure control plan, engineering and work practice controls, prohibition of worker rotation, respiratory and personal protection, hygiene areas and practices, housekeeping, medical surveillance and removal, and communications. Key elements of a successful program for protection of the worker and compliance with the OSHA standard are presented.

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Los Alamos, New Mexico 87544
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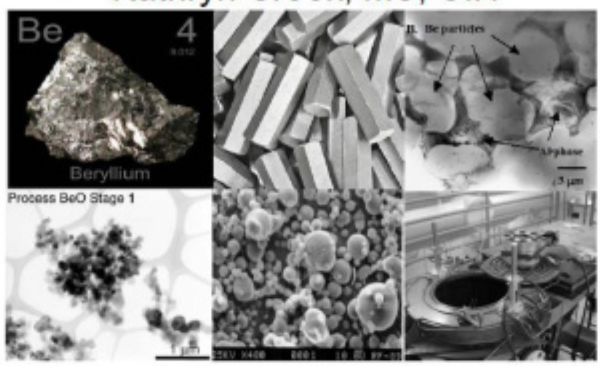
Key Elements of a Successful Beryllium Control Program

Kathryn Creek, MS, CIH



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Key Elements of a Successful Beryllium Control Program
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Page 1

A successful beryllium product manufacturing business requires control of worker exposures to skin and inhalation

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Page 2

Establish a Comprehensive Program

EU

Limit only

UK

Limit only

US-Expanded Standards

DOE

Subpart A—General Provisions

Sec.
850.1 Scope.
850.2 Applicability.
850.3 Definitions.
850.4 Enforcement.
850.5 Dispute resolution.

Subpart B—Administrative Requirements

850.10 Development and approval of the CBDPP.
850.11 General CBDPP requirements.
850.12 Implementation.
850.13 Compliance.

Subpart C—Specific Program Requirements

850.20 Baseline beryllium inventory.
850.21 Hazard assessment.
850.22 Permissible exposure limit.
850.23 Action level.
850.24 Exposure monitoring.
850.25 Exposure reduction and minimization.
850.26 Regulated areas.
850.27 Hygiene facilities and practices.
850.28 Respiratory protection.
850.29 Protective clothing and equipment.
850.30 Housekeeping.
850.31 Release criteria.
850.32 Waste disposal.
850.33 Beryllium emergencies.
850.34 Medical surveillance.
850.35 Medical removal.
850.36 Medical consent.
850.37 Training and counseling.
850.38 Warning signs and labels.
850.39 Recordkeeping and use of information.
850.40 Performance feedback.

DOL/OSHA

(a) Scope and Application
(b) Definitions
(c) Permissible Exposure Limits (PELs)
(d) Exposure Assessment
(e) Beryllium Work Areas and Regulated Areas (General Industry); Regulated Areas (Maritime); and Competent Person (Construction)
(f) Methods of Compliance
(g) Respiratory Protection
(h) Personal Protective Clothing and Equipment
(i) Hygiene Areas and Practices
(j) Housekeeping
(k) Medical Surveillance
(l) Medical Removal
(m) Communication of Hazards
(n) Recordkeeping

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Page 3

Establish Purpose of Program

To prevent CBD and BeS in the workplace and environment

- Minimize # of Be Workers, locations and items contaminated with beryllium
- Minimize # of transient workers
- Minimize non-essential beryllium containing components
- Keep contamination under company action limits (AL)*
- Minimize the potential for migration

* Consider airborne AL at 5-10% of the PEL or OEL

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Goals and Feedback

- Here are some examples
  - No CBD or BeS cases
  - No exposures above PEL
  - No exposures above the action level without respiratory protective equipment (RPE)
  - No nasal smears with detectable levels
  - Constant number of beryllium areas
- Evaluate and report on each year

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Scope

- All workers on site who have the potential for exposure to beryllium should be covered by the prime contractor's beryllium control program
- Include in the scope beryllium materials that are greater than 0.1% weight percent beryllium

Beryllium aluminum silicates should be addressed as naturally occurring beryllium that has no known health risk due to the beryllium

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Risk Assessment

- Conduct an evaluation of all operations using airborne contamination measurements in worker's breathing zone with weight-based methods
- Assess exposure to beryllium particle number by subjective or objective information/data
  - Does the operation involve high heat, high energy, or high speed of rotation? If so, there is probability that ultrafine particles are generated

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Follow the Hierarchy of Controls

Source:
- Elimination
- Substitution
- Source or process modification
- Automation
- Isolation / containment / enclosure
- Local exhaust ventilation

Path:
- General ventilation
- Increase distance
- Work scheduling

Worker:
- Good working practice
- Operating procedures
- Personal protective equipment

Prevention: Elimination, Substitution, Source or process modification

Engineering: Automation, Isolation / containment / enclosure, Local exhaust ventilation, General ventilation

Procedures: Increase distance, Work scheduling, Good working practice, Operating procedures

PPE: Personal protective equipment

Student Manual for Control of Hazardous Substances, HSE

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Page 8

Balance

Given the cost is high for beryllium controls, we should put our funds where they will be most effective and improve with time

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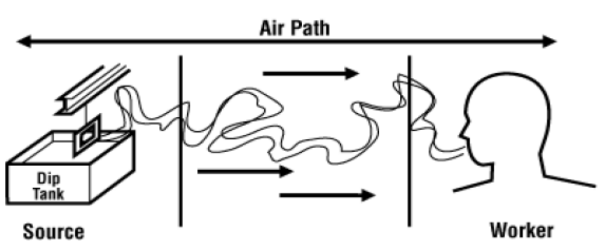
Page 9

Airborne Contamination Minimization

- Minimize the airborne exposure by applying engineering controls at the source
  - High Velocity Low Volume local exhaust extraction
  - Isolation
  - Exhaust of enclosures.
- Consider automation of high risk operations



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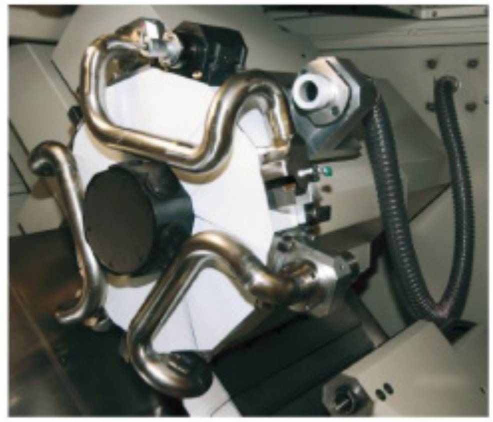
Source Capture for Lathe



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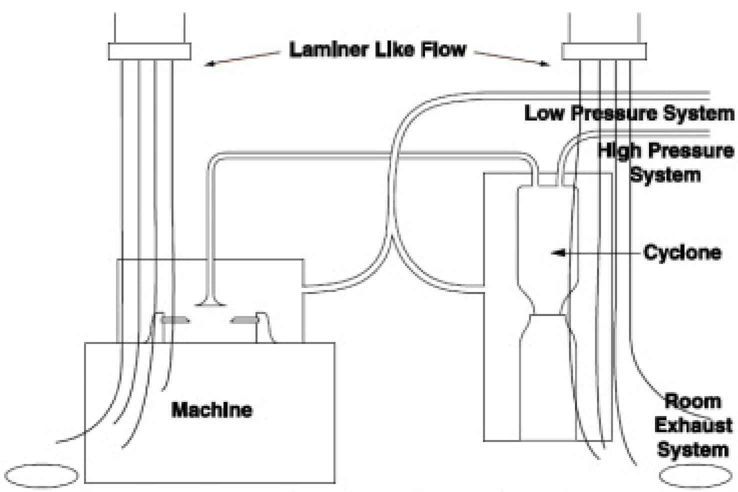
Source and Enclosure Exhaust



- High velocity capture at source
  - 7000 - 12000 fpm face velocity
  - Custom capture hoods
- Exhausted Enclosures
  - Provide 2° control
  - Custom designed each source

Use HEPA filtration and nuclear grade housings

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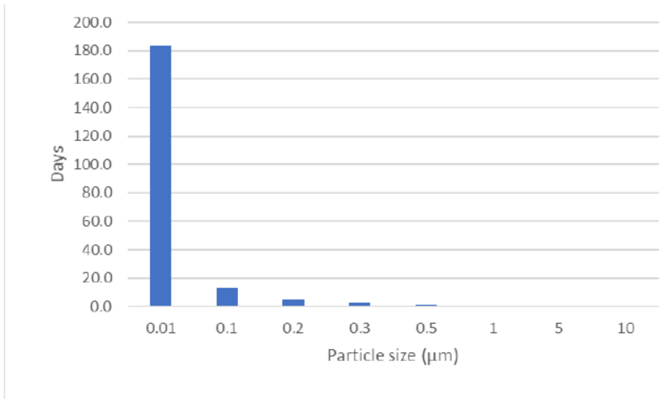
Page 12

General Exhaust

For airborne contamination that was not captured at the source and especially for ultrafine particulate, a high number of room air changes per hour is recommended



13 days for 0.1 μm particle to settle one meter

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Keep Beryllium inside Beryllium Areas

- Facility design should have import/export areas that minimize access to suppliers
- Consider confinement, airlocks, and pressure cascades with the lowest pressure in the highest risk area
- Keep process duct work and equipment inside beryllium areas
- Use Contamination Reduction Zones (for workers and equipment) when coming out of beryllium areas

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Operating Procedures and Work Permits

- High risk operations should have detailed operating procedures, work permits, and management of change
- Approved by IH/Safety and line management
- Particularly important for maintenance activities

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H&S Personnel Qualifications

Occupational Health Personnel qualifications should have **experience controlling beryllium operations**

Accept no substitute!

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Page 16

Conduct Exposure Monitoring

- Exposure monitoring should be used not only as a method of evaluation, but also as a control method to identify airborne contamination above expected levels on a daily basis
  - Personal air sampling or breathing zone
  - Nasal smears
  - Skin contamination monitoring (ASTM D7822-18)

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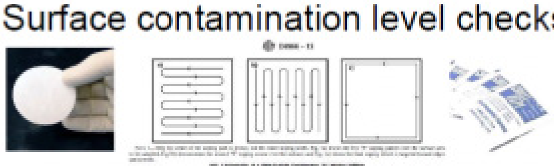
Page 17

Conduct Surveillance of Controls

- Facility, operations, and maintenance activities should be checked on a routine basis (daily, weekly, monthly)
- Checks of engineering control parameters
- Surface contamination level checks



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Key Elements of a Successful Beryllium Control Program
BeWS-2019, Longbeach, CA, USA

Page 18

Housekeeping Practices

- Use a dedicated trained staff
- Housekeeping schedule should include
  - Cleaning of all horizontal surfaces every year to minimize resuspension
  - Mopping floors on a routine basis
- Operators should clean their work area at the end of the shift

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Page 19

Standard PPE/RPE

- Use multiple levels of Personal and Respiratory Protection based on airborne and skin contamination risk
- Standard clothing
  - Eye protection (prevent eye exposure from contaminated hand contact)
  - Coveralls
  - Impervious gloves
  - Over boots
  - Company provided work shoes, modesty clothing, etc.
- Industry standard for respiratory protective equipment is PAPR hood-type respirator

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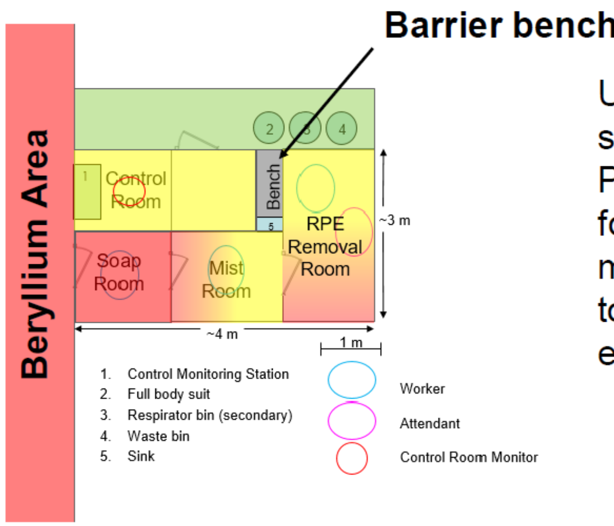
Contamination Reduction Zone



Use of foam soap on PPE/RPE followed by misting seems to be most effective

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Page 21

Medical Surveillance

- Routine exam (yearly for current, less frequent for former)
- Should include BeLPT testing
- Follow up exam for BeS (extensive workup)
- Wounds from inside or outside the beryllium zone
- Worker should report to medical after surgeries for limits on work in a beryllium area

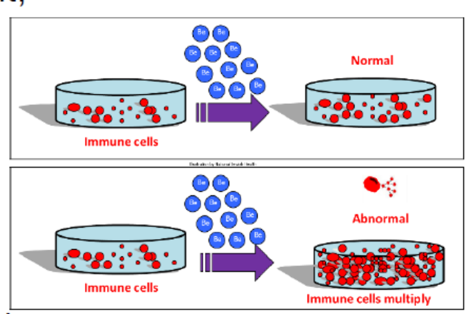

Image from National Jewish Health

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Page 22

Medical Removal & Counseling

- Final alternate work for individuals who are BeS or have CBD
- Notify management
- Include in program the requirements for modifying the worker's job (no beryllium exposure) and the timeframe that the employer must honor this
- Need to have counselors who help workers who are BeS or have CBD and to address health conditions and work changes

Emergency Procedures

- Medical emergency inside beryllium area
  - Trained for first aid
  - Limiting contamination risk offsite
  - Equipment for moving patient
- Need to consider how emergency personnel will be included in the program
- Injuries/wounds that occurred inside beryllium area should be considered an emergency with cleaning/sampling of the wound

Training on Beryllium Safety

- Beryllium Workers
  - Includes operators, crafts (maintenance), laboratory technicians/chemists, cleaning/decontamination personnel, supervisors, managers, contractors, etc.
- Managers
  - Include how to address workers that are BeS or have CBD
  - Know the elements of the program in sufficient detail that can ensure workers are following the requirements
- Design Engineers
- Mentoring of new workers is a very good practice

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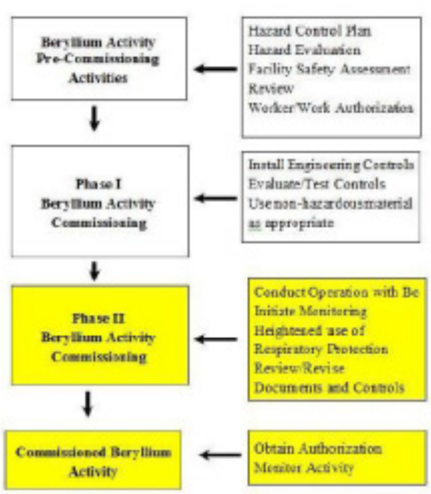
New Operations & Commissioning



Test operation with surrogate

Test operation with beryllium

Operation Authorization

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Key Elements of a Successful Beryllium Control Program
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Page 26

Hygiene Facilities and Practices

- Prohibited items and activities
  - No jewelry
  - No application of cosmetics
  - No personal clothing & shoes
  - No food, beverage, or tobacco
  - No chewing gum
- Showering should be conducted each time the worker leaves beryllium area
- Facilities
  - Change room/shower facility
  - Separate location free of contamination for storage of personal clothing
  - Lunchroom (ensure they are free of contamination)

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Page 27

Warning Signs and Labels

- Entrance to Beryllium Areas
  - Simple
  - Explanatory
  - Display requirements of PPE/RPE
- On beryllium materials and beryllium contaminated equipment/piping

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Page 28

Procurement of Beryllium & Services

- Include requirements for controlling risks to beryllium in your contracts
  - services inside a beryllium area (i.e., equipment repair or maintenance calibration)
  - laboratories, waste management, laundry, RPE cleaning

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Exceedances of Limits

- Notification to management
- Management/Safety Review
  - Fact finding
  - Corrective Action Plan
  - Lessons Learned
  - Notification of enhanced controls to worker's affected

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Release of Equipment from Be Areas

- Equipment should be sampled with results below company limits
- Sampling should be sufficient in number with all surfaces considered
- Company limits could be similar to DOE limits
  - Release limits for moving item to non-beryllium area of 0.2 μg/100cm^2
  - Release limits for moving item from one beryllium area to another of 3 μg/100cm^2
- Consider using tags for release of equipment

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Sampling Plans and Analysis

- Establish and implement sampling plans with schedules for beryllium zones and operations
  - Personal breathing zone sampling
  - Surface sampling in non-beryllium areas
- Use of approved methods and certified laboratories for beryllium analysis

NMAM

BERYLLIUM in Air
by Fluorometry — 7704

Be — MW: 9.0121 — CAS: 7440-41-7 — RTECS: DS1750000

METHOD: 7704, Issue2 — **EVALUATION:** FULL — **Issue 1:** 6 April 2007; **Issue 2:** 12 December 2015

Real-time beryllium monitoring is not available

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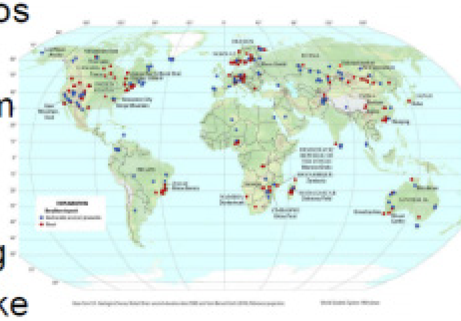
Background Beryllium

- Naturally occurring beryllium may interfere with sampling results
- Consider use of elemental ratios such as yttrium
- Example of Beryllium to Yttrium ratio
  - <1:5 Manmade
  - >1:5 Naturally occurring
- Need specific site study to make the ratio determination



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Page 33

Recordkeeping

- Establish procedures and possibly databases for the inventory, worker monitoring, facility surveillance, exceedances of controls, worker training, worker respiratory protection qualifications

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Basic Steps

- Develop an inventory (list of all operations and areas where there is a potential for beryllium exposure)
- Conduct risk assessments in relation to company action limits
- Control operations or areas based on outcome of assessments
- Design facilities for minimization of personnel
- Establish exposure monitoring and facility surveillance on a schedule
- Evaluate program based on goals

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Key Elements of a Successful Beryllium Control Program
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Conclusion

- Beryllium is a highly toxic metal; therefore addressing H&S is imperative to working with beryllium containing materials
- Design operations and facilities using engineering controls to minimize airborne beryllium contamination and the footprint
- Conduct monitoring and surveillance for control feedback
- Rely on skilled resources and development and implement an effective Beryllium Control Program!

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Key Elements of a Successful Beryllium Control Program
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Thank you for your attention

www.beryllium-solutions.com
creek@beryllium-solutions.com

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Surface Beryllium and Worker Exposures

Marc Kolanz and Theodore Knudson

Materion Corporation, Mayfield Heights, Ohio, U.S.A.

As a naturally occurring element which is ubiquitous in nature, surficial levels of beryllium may contribute to workers exposures to beryllium with potential health impacts. This presentation will discuss the sources of naturally occurring beryllium, the origin of surface limits, soluble versus insoluble compounds, surface contamination and its relationship to skin/inhalation exposures and potential health effects.

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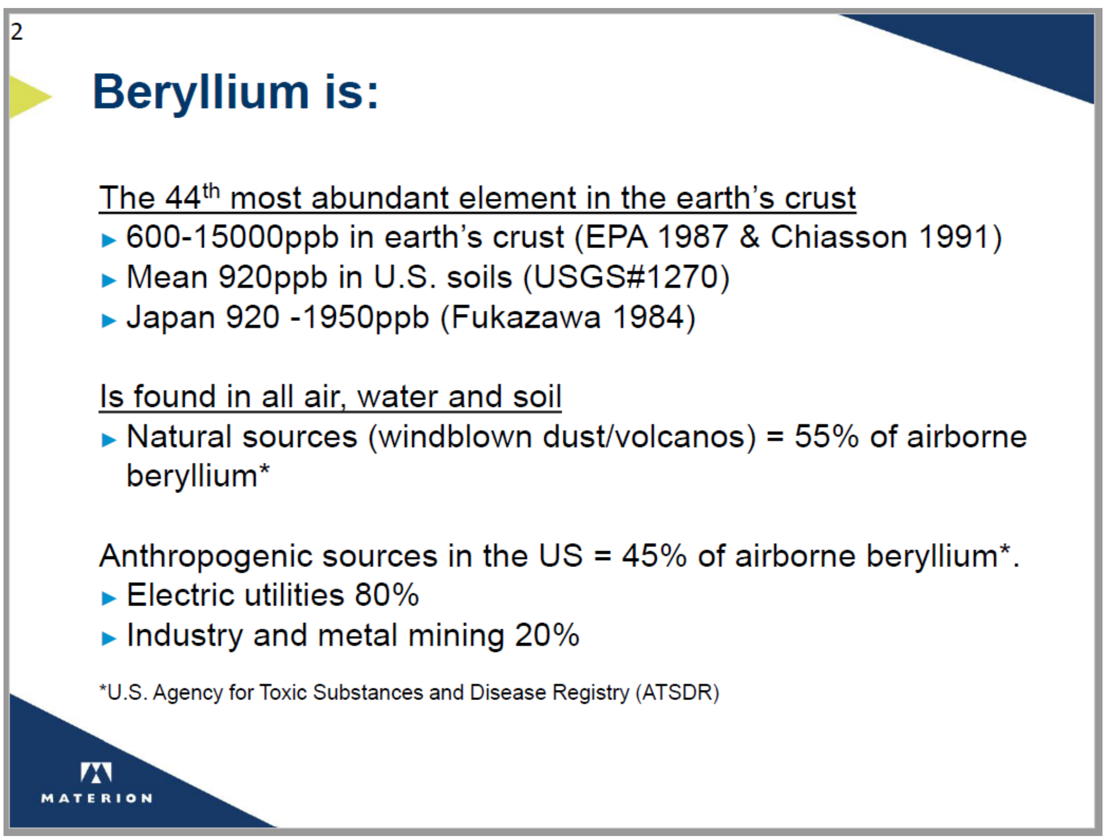
MATERION
Surface Beryllium and Worker Exposures
14th International Workshop on Beryllium Technology
Hotel Queen Mary, Long Beach, California, USA
October 24-25, 2019
Marc Kolanz, CIH
Vice President EH&S
Materion Corp.


2
Beryllium is:
The 44th most abundant element in the earth's crust
600-15000ppb in earth's crust (EPA 1987 & Chiasson 1991)
Mean 920ppb in U.S. soils (USGS#1270)
Japan 920 -1950ppb (Fukazawa 1984)
Is found in all air, water and soil
Natural sources (windblown dust/volcanos) = 55% of airborne beryllium*
Anthropogenic sources in the US = 45% of airborne beryllium*.
Electric utilities 80%
Industry and metal mining 20%
*U.S. Agency for Toxic Substances and Disease Registry (ATSDR)
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3

Beryllium is:

- Average US ambient air 0.03 nanograms/m^3*
- Annual Average US ambient air (cities) 0.2 nanograms/ m^3 *
- Range of Annual Average Urban air 0.1 -7.0 nanograms/m^3 **
- Average soils range from 0.3 – 2ppm
- US groundwater average is 13.6 µg/L (USEPA 2000)
- US surface waters average 23.8 µg/L*
- US drinking water average is 0.19 µg/L (range 0.01 – 1.22)*
- Germany (Rhine & Main rivers) <0.2 µg/L (Reichert 1973)
- Rainwater 0.05-0.08 µg/L (Meehan & Smythe 1967)

* ATSDR 2000

**EPA 1987

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Beryllium is:

Ubiquitous in soil

- A shovel full of typical soil is about 2 Kg.
- One Kg of typical soil contains about 1000-2000 µg beryllium. (Field 2011)

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National Uranium Resource Evaluation Data (NURE (detected 70 elements)

- Number of NURE Sample Points — 678,558
- Sample Points with detectable levels of beryllium — 206,168 - 30%

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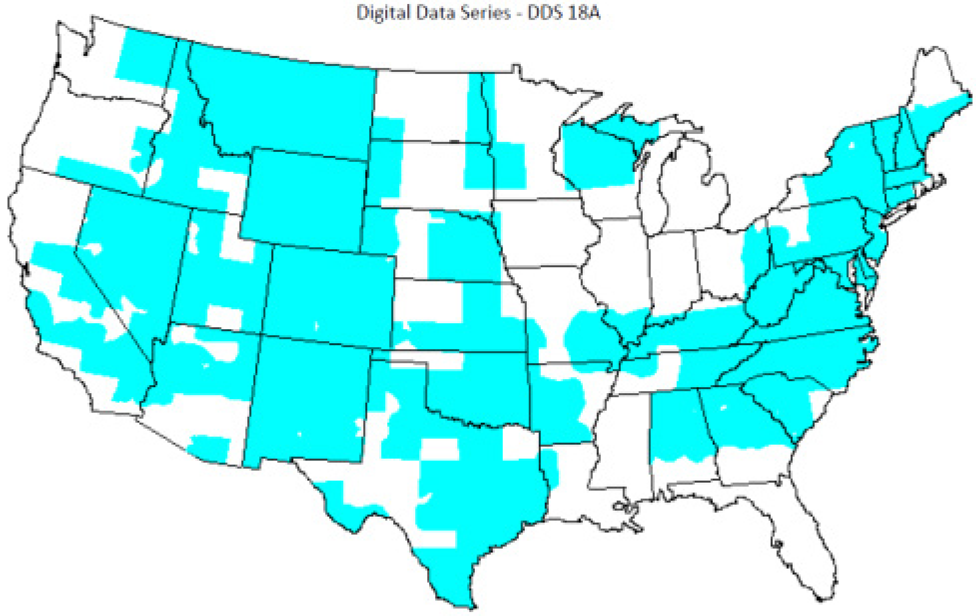
National Geochemical Database:

National Uranium Resource Evaluation / Hydrochemical Stream Sediment Reconnaissance

Digital Data Series - DDS 18A



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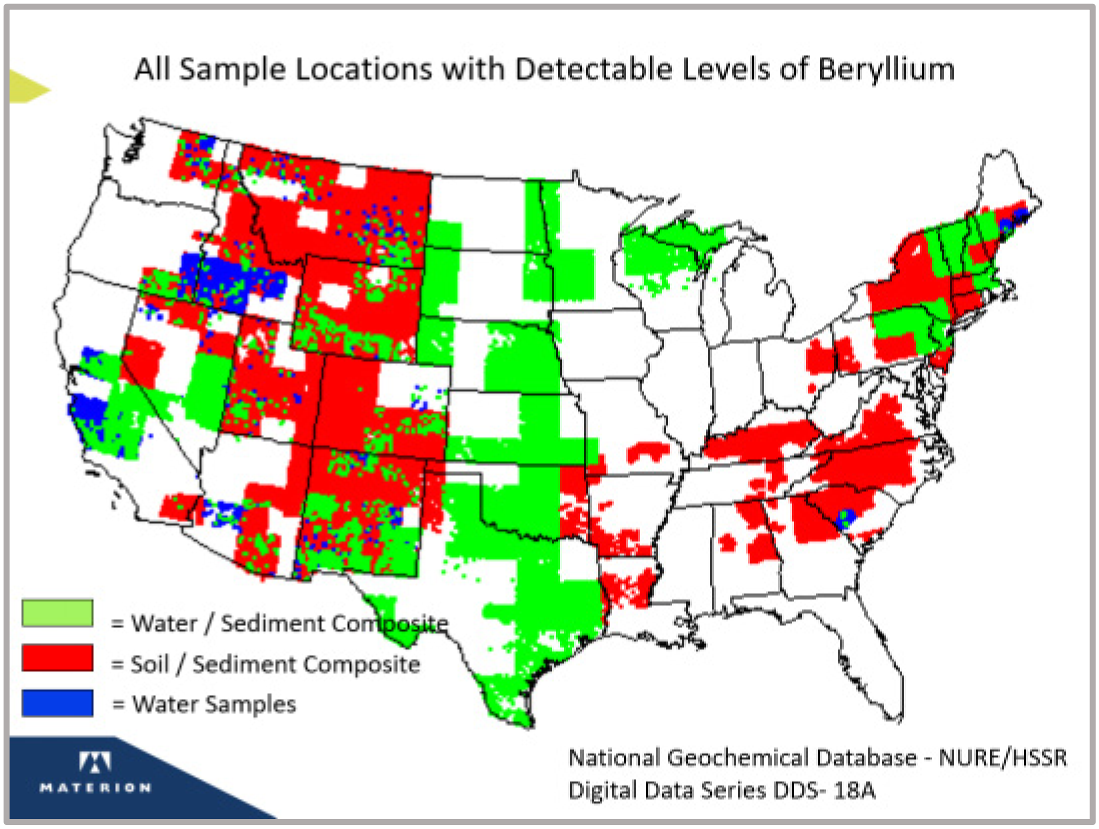
All Sample Locations with Detectable Levels of Beryllium
= Water / Sediment Composite
= Soil / Sediment Composite
= Water Samples
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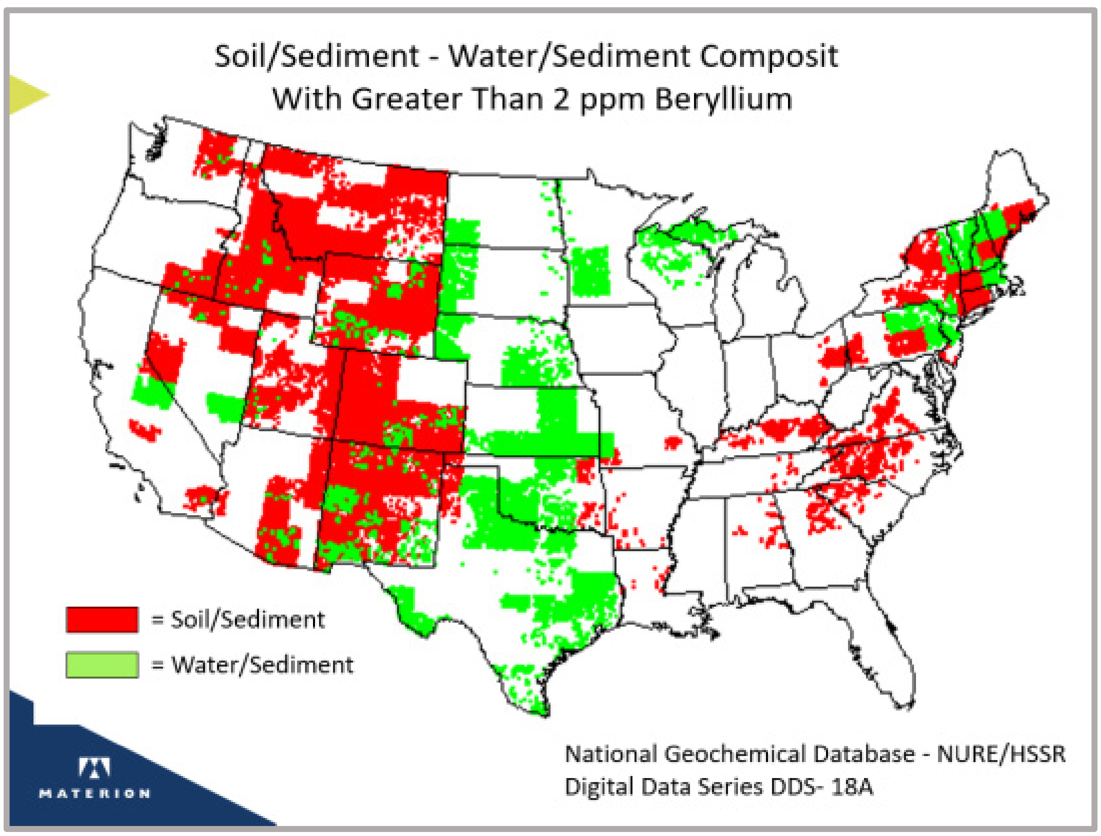
National Geochemical Database - NURE/HSSR
Digital Data Series DDS- 18A


Soil/Sediment - Water/Sediment Composit
With Greater Than 2 ppm Beryllium
= Soil/Sediment
= Water/Sediment
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National Geochemical Database - NURE/HSSR
Digital Data Series DDS- 18A

Beryllium in Non-occupationally Exposed Persons

- Daily intake 5-100 µg/day (ATSDR 2002)
- Lung burden 0.1-20 µg/kg (dry weight) (EPA 1987)
- Other organs <80 µg/kg

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Beryllium in Foodstuffs and Consumables (µg/kg or ppb)

Solids (all ATSDR 1991/2002 except as noted)

Cabbage	0.24	Green pepper	400
Mushrooms	1.58	Kidney beans	2200
Crabs	15	Carrots	25
Oyster flesh	2.0	Corn	25
Rice	80	Peas	109
Brown sugar	30	Potatoes	170
Refined sugar	2	Ground coffee	500 & 77.9 (Materion)
Crisp bread	112		
Lettuce	330	Chocolate bar	190 (Materion)
Eggplant	370	Flour	100 (Materion)

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Beryllium in Foodstuffs and Consumables

Liquids (µg/L)

Mineral water	0.94 (high) (Kilinc 2011)
Wine	0.16- 0.25 (Bayern 2010)
Whiskey	0.0003 (Woods 2011)
Apple juice	22.5 (ATSDR 2002) (detected in 58%)
Orange juice	2.8 (ATSDR 2002)
All juices	(detected in 38%) (Bayern 2010)
Tomato sauce	42.4 (ATSDR 2002)
Milk	0.2 µg/kg (ATSDR 2002)

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Beryllium in Foodstuffs and Consumables

Other (µg/kg or ppb)

Coal	2000 (ATSDR 2002)
Fuel oil	100-200 (ATSDR 1991)
Granitic Rock	6,000 (USGS #1214)
Orchard Leaves	26 (ATSDR 2002)
Fertilizers	<200 - 13,500 (ATSDR 2002)
Sewage sludge	40-2300 (dry weight) (USEPA 2009)
Copper ore	690 (Materion)
Cigarettes	ND - 0.74 µg/cigarette(2-10% in smoke) (ATSDR 2002)

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Beryllium in Clay & Mineral-based Materials

Household items examples* (µg/kg or ppb) (not intentionally added)

Cosmetics	206 & 1010
Detergents	430 & 690
BBQ Charcoal	197 & 300
Kitty litter	870 & 837
Aluminum foil	390
Fertilizer	510, 506, 184
Automotive disc brakes	180
Home vacuum dirt	165
Fireplace ash	350
Candy wrapper	120
Household dust (estimate)	60 (USEPA 1987)

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* Unpublished Materion

Beryllium in Clay & Mineral-based Materials

Construction/manufacturing Materials (ppb-not intentionally added)

Cement	1020 & 459 (Materion)
Clinker dust/fly ash	900/2300 (NIOSH 2005)
Ceiling tiles	1190 (Materion)
Emery cloth	85 & 930 (Materion)
Roofing shingle	1000 (Materion)
Glass wool	2000 (Materion)
Beach sand	132 (Materion)

Grinding wheels, hones, sticks 0.02%-0.14% (DOE Lessons Learned)

Abrasive blast media

Crushed glass 270 to 757ppb (9 samples-ABMA 2018)

Garnet <52ppb (Materion)

Coal based 4360, 4480ppb (Materion)

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Beryllium in Ores and Gems

Beryl	0.5 -- 3.5% Be
Beryllite	4.3 -- 5.6% Be
Bromellite	24 -- 36% Be
Bertrandite	0.5 -- 1% Be
Chrysoberyl	0.5 -- 7% Be
Danalite	0.5 -- 9% Be
Eudidymite	1.5 -- 3.7%Be
Helvite	2.5 -- 5.2% Be
Phenakite - (Phenacite)	8.5 -- 16.4% Be
Hambergite - (Homberlite)	9.5 -- 19.2% Be
Leucophanite	1.5 -- 10.0% Be
Meliphanite	1.5 -- 13.0% Be
Milarite	1.5 -- 1.8% Be
Taaffeite	0.5 -- 1.9% Be
Euclase	1.5 -- 6.1% Be
Roscherite	4.3 -- 5.6% Be
Gadolinite	1.5 -- 10.6% Be
Herderite	3.5 -- 15.1% Be

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Non-beryllium Manufacturing Facility Example

Initial surface sampling (smears) of 50 grinding machines revealed contamination of 5 machines > 0.05µg/100cm^2 analytical laboratory limit of quantification (LOQ).

Statistically-based, biased sampling of the 5 machines (59 smears) yielded 12 results >LOQ.
2/12 > 0.2µg/100cm^2.

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Non-beryllium Manufacturing Facility Example

Sampling of 27 businesses not specifically working with beryllium (Field 2014)

- Auto repair shop, coal power plant, flower shop, restaurant, fertilizer co-op, hog confinement, machine shop, electronics shop, dental lab, schools and bars etc.
- 137 samples of surfaces with visible dust (bookshelves, cabinet tops, ductwork, etc)
- 3 sites (11%) reported prior beryllium use (2 dental, machine shop)

78% above the LOQ (0.035 µg).
12% exceeded 0.2 µg/100 cm^2 (6/10 sites w/no beryllium use)
4% exceeded 3 µg/100 cm^2 (all Be use (recent or past) except fertilizer co-op)

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Non-beryllium Manufacturing Facility Example

Sampling of 27 businesses not specifically working with beryllium (Field 2014)

<u>Select interesting findings</u>
Sites without detectable Be:
- Computer recycling shop

Sites with detectable Be included:
- Flower shop
- Hog confinement
- Restaurant
- School
- Food processing co-op
- Fertilizer co-op
- Feed/fertilizer shop

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Surface Beryllium in Public Places

Atlanta, Georgia USA (Materion 2002-unpublished)

Surface wipe samples (µg/100 cm^2)	
Country Fried Chicken restaurant	0.036
City Hall newspaper box	0.082
Newspaper stand (downtown)	0.015
Eye exam office	0.012
Big Lot store shelf	0.0085

Soil and other Atlanta area samples (µg/kg)	
Residential vacuum cleaners	260 & 350
15 soil samples range	<100(2) - 1200
15 soil samples average	563

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Health Implications for Workers Exposed to Trace Beryllium (not intentionally added)

- No known case of CBD linked only to exposures to natural beryllium-containing materials or the use of processed materials where beryllium was not intentionally added for its unique properties; i.e., abrasive blast media, coal slag in road construction, etc.

- Naturally occurring minerals that contain beryllium involve a form of beryllium that is a beryllium aluminum silicate.

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Recommended Surface Limits (US) (not health-based, based on ability to clean to limit)

- 1950s U.S. Atomic Energy Commission site clean-up goal
  = 25 µg/ft^2 (dry wipe) (2.67 µg/100 cm^2)
- 1999 DOE post workday operations
  = 3.0 µg/100 cm^2 (dry)
- 1999 DOE Cleaning of contaminated equipment for release to non-beryllium areas or the general public (not applicable to buildings)
  = 0.2 µg/100 cm^2 (dry)

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French Nuclear Safety Authority ITER Regulations (scientific basis for criteria unknown)

Beryllium surface contamination exposure criteria*
(µg/M^2 converted to µg/100 cm^2)

Beryllium zone with respiratory protection	0.1 µg/100 cm^2
Beryllium controlled zone	0.001<[Be]< 0.1 µg/100 cm^2
Beryllium non-controlled zone	< 0.001 µg/100 cm^2

*ITER – Joelle Elbez-Uzan June 2017

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ITER Criteria versus Atlanta GA Public Places

Surface Wipe Samples (µg/100 cm^2)

Country fried chicken restaurant	0.036	**= ITER beryllium-controlled zone**
City Hall newspaper box	0.082	**= ITER beryllium-controlled zone**
Newspaper stand (downtown)	0.015	**= ITER beryllium-controlled zone**
Eye exam office	0.012	**= ITER beryllium-controlled zone**
Big Lots store shelf	0.0085	**= ITER beryllium-controlled zone**

Recommendation – ITER revisit its criteria basis with French Nuclear Safety Authority

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Beryllium in Air (scientific basis for criteria unknown)

French Nuclear Safety Authority ITER Regulations

Beryllium atmospheric concentration exposure criteria*

Beryllium zone with respiratory protection	>0.2 µg/m^3
Beryllium controlled zone	0.01<[Be]< 0.2 µg/m^3
Beryllium non-controlled zone	<0.01 µg/m^3

*ITER – Joelle Elbez-Uzan June 2017

NOTE:

French OEL = 2.0 µg/m^3 (total method?)
EU-wide OEL = 0.6 µg/m^3 (inhalable method = 0.2 total method)
US-OSHA OEL = 0.2 µg/m^3 (total method)

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Beryllium in Air

Work activities found to exceed US/EU beryllium OELs of 0.2/0.6 µg/m^3 due to exposures to airborne beryllium from working with materials containing trace beryllium that is ***not intentionally added***.

Painting	Road sweeper	Abrasive blasting	Carpentry	Fabricating
Cutting concrete	Road building	Building demolition	Electrical work	Welding
Dumping soils	Jack hammering	Boiler cleaning	Excavating	Sandblasting
Torch cutting	Welding	General labor work	Needle gunning	Shot blasting

Information Source is U.S. OSHA IMIS database

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Beryllium in Air

Non-beryllium manufacturing facility examples:

Roof coatings (tar-like) manufacturer (Materion study)

25 Air Samples Taken = <0.01-0.08ug/m^3

1 Bulk Sample of HVAC Filter = 175 ug/$ft.^2$

Cement plant (OSHA 2007)

Cement slag = >2.0ug/m^3

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Beryllium in Air

Revisiting OSHA IMIS data and non-beryllium manufacturing reveals that natural trace beryllium during “construction phase” or non-beryllium operational areas at ITER could result in areas being classified as a **“Beryllium zone with respiratory protection” or a “Beryllium controlled zone”**

Recommendation - ITER further clarify its application of beryllium zoning boundaries of what is meant by ***“starting with the construction phase”*** and in non-beryllium operational areas.

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Session 7: The BeYOND Industrial Forum

Overview of the BeYOND Workshop Series

A. Goraieb (KBHF, Germany) et al.

Overview of the BeYOND Workshop Series

Aniceto Goraieb[1] and Christopher Dorn[2]

[1]*Karlsruhe Beryllium Handling Facility (KBHF GmbH), Eggenstein-Leopoldshafen, Germany*
[2]*Be4FUSION LLC, Upland, California, U.S.A.*

In the technical field of beryllium, there are currently two workshops that take place in alternating years, the IEA International Workshop on Beryllium Technology (BeWS) in the odd-numbered years and the BeYOND Industrial Forum in the even-numbered ones. The name BeYOND is an acronym which stands for "Beryllium Opportunities for New Developments".

After trying various timing with respect to other conferences, it evolved that the BeYOND workshops would take place on alternate years from the BeWS, and normally held in conjunction with the SOFT Conference (Symposium on Fusion Technology), which is always held in Europe. Meetings to date:

- BeYOND-1: Karlsruhe, Germany, 2009
- BeYOND-2: Porto, Portugal, 2010
- BeYOND-3: Karlsruhe, Germany, 2011
- BeYOND-4: Karlsruhe, Germany, 2012
- BeYOND-5: Barcelona, Spain, 2013
- BeYOND-6: San Sebastian, Spain, 2014
- BeYOND-7: Berlin, Germany, 2016
- BeYOND-8: Karlsruhe, Germany, 2018

BeYOND was created with the idea of more emphasis on strengthening the connection between the beryllium research community and the beryllium industry, an aspect not always satisfied by the BeWS. Since BeYOND-5 in 2013, the workshop has generally also placed more emphasis on beryllium health and safety than typically found in the BeWS.

At this point, BeYOND-9 is planned for 17-18 September 2020, to be held at or near the ITER Organization site in Cadarache, France.

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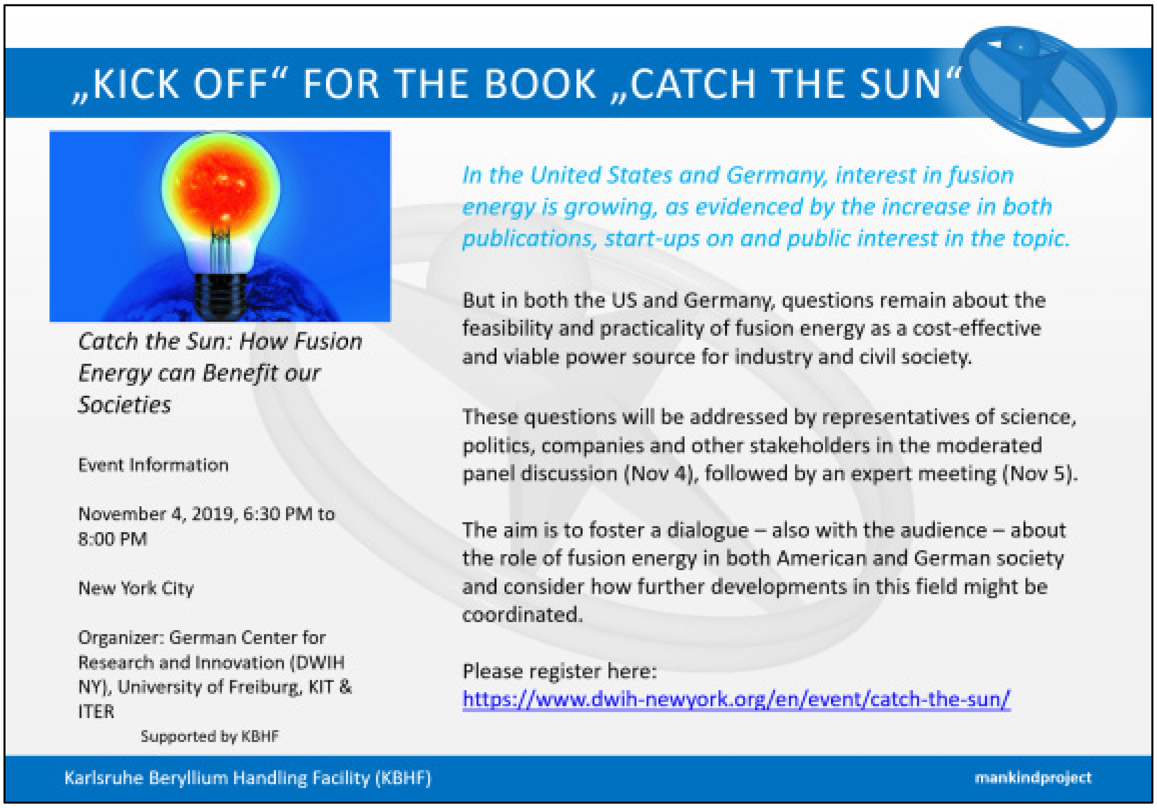
GERMANY


BeYOND 2020 will take place at ITER
17^{th} and 18^{th} September 2020
FUSION FOR FUTURE A MANKINDPROJECT
KBHF
Karlsruhe Beryllium Handling Facility
www.goraieb.de


TOPIC WILL BE: "HEALTH AND SAFETY"
Photomontage:
Picture of Mario Dalle Donne in a Diving Capsule, taken by me at the EPCOT Center
ICFRM Clearwater Florida 1991
On behalf of the IAC we hope to welcome You at BeYOND again!


„KICK OFF“ FOR THE BOOK „CATCH THE SUN“
Catch the Sun: How Fusion Energy can Benefit our Societies
Event Information
November 4, 2019, 6:30 PM to 8:00 PM
New York City
Organizer: German Center for Research and Innovation (DWIH NY), University of Freiburg, KIT & ITER
Supported by KBHF
In the United States and Germany, interest in fusion energy is growing, as evidenced by the increase in both publications, start-ups on and public interest in the topic.
But in both the US and Germany, questions remain about the feasibility and practicality of fusion energy as a cost-effective and viable power source for industry and civil society.
These questions will be addressed by representatives of science, politics, companies and other stakeholders in the moderated panel discussion (Nov 4), followed by an expert meeting (Nov 5).
The aim is to foster a dialogue – also with the audience – about the role of fusion energy in both American and German society and consider how further developments in this field might be coordinated.
Please register here:
https://www.dwih-newyork.org/en/event/catch-the-sun/
Karlsruhe Beryllium Handling Facility (KBHF)
mankindproject


Without you, FUSION will remain an Experiment, that is why we have to think BeYOND 2020
FUSION FOR FUTURE A MANKINDPROJECT
KBHF
Karlsruhe Beryllium Handling Facility
www.goraieb.de

Appendices

BeWS-14 Participants List

First Name	Family Name	Organization	Country
Eduardo	Alves	Instituto Superior Tecnico	Portugal
Raul	Amarelle	LEADING Enterprises	Spain
Vladimir	Chakin	KIT	Germany
Kathryn	Creek	Beryllium Solutions International	USA France
Mirjana	Damjanovic	CEA UK Atomic Energy Authority	France UK
Marta	Dias	Instituto Superior Tecnico	Portugal
Christopher	Dorn	UK Atomic Energy Authority Be4FUSION LLC	UK USA
Fred	Elsner	General Atomics	USA
Ramil	Gaisin	KIT	Germany
Aniceto	Goraieb	KBHF	Germany
Klaus	Hesch	KIT	Germany
Hidetaka	Kanawaza	Chiyoda Technol Corporation	Japan
Hiroshi	Kawamura	Chiyoda Technol Corporation Japan Atomic Energy Agency (Retired)	Japan
Jae-Hwan	Kim	QST	Japan
Michael	Klimenkov	KIT	Germany
Theodore	Knudson	Materion Corp.	USA
Andreas	Koester	TROPAG GmbH	Germany
Marc	Kolanz	Materion Corp.	USA
Viacheslav	Kuksenko	UK Atomic Energy Authority	UK
Glen	Longhurst	Southern Utah University (Retired) Idaho National Laboratory (Retired)	USA
Patrick	Lorenzetto	Fusion for Energy	Spain
Joey	Loyd	Hardric Laboratories	USA
Peter	Maehlmann	TROPAG GmbH	Germany
Walid	Mohamed	Argonne National Laboratory	USA
Keisuke	Mukai	Kyoto University	Japan
Masaru	Nakamichi	QST	Japan
Yuri	Natori	KAKEN Laboratories	Japan
Keigo	Nojiri	NGK Insulators, Ltd.	Japan
Yi-Hyun	Park	NFRI	Korea
Richard	Pearson	The Open University	UK
Dirk	Radloff	KIT	Germany
René	Raffray	ITER Organization	France
Rolf	Rolli	KIT	Germany
Hans-Christian	Schneider	KIT	Germany

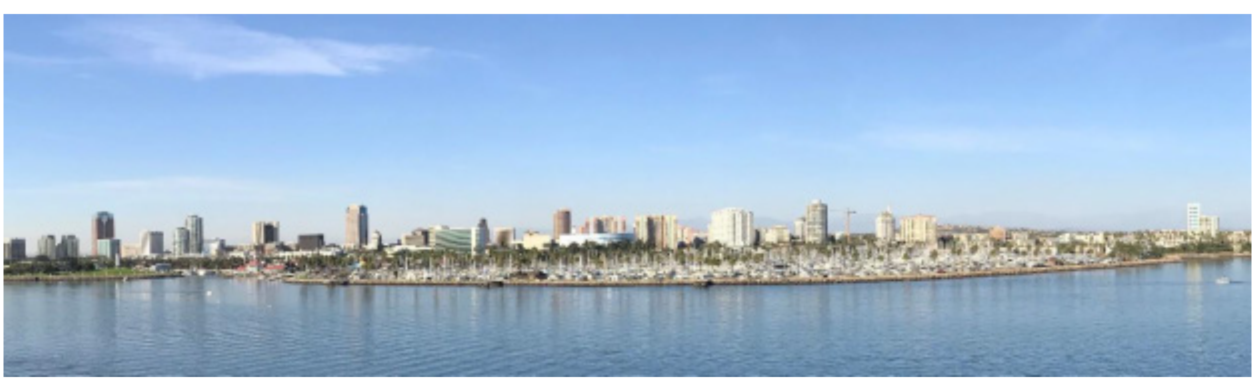
First Name	Family Name	Organization	Country
Keith	Smith	Materion Brush Inc.	USA
Gary	Solbrekken	University of Missouri	USA
Peter	Späh	KIT	Germany
Benjamin	Spilker	Forschungszentrum Jülich	Germany
Christopher	Stihl	KIT	Germany
Arman	Suleimenov	Ulba Metallurgical Plant	Kazakhstan
Ron	Townsend	Materion Brush Inc.	USA
Carole	Trybus	Materion Brush Inc.	USA
Lee	Vandermark	General Dynamics	USA
Pavel	Vladimirov	KIT	Germany
Beth	Walker	Cambridge Technology Ltd.	UK
Lance	Whalen	General Dynamics	USA
Sunyoung	Yang	Modern Science Tech Inc. Motion High Tech Co., Ltd.	USA Korea
Nikolai	Zimber	KIT	Germany
Milan	Zmitko	Fusion for Energy	Spain

Workshop & Venue Photos

The BeWS-14 was held at one of more unusual venues in its history, aboard the permanently moored retired ocean liner, the RMS Queen Mary, located in Long Beach since 1967. Today, the Queen Mary functions primarily as an event center and hotel.

View of The Queen Mary in her berth at the harbor and sign near the entrance to the ship.

The workshop was held in the former First-Class Smoking Lounge on board the ship. Note the Art Deco style, including the decorative mural at the back of the room, seen in the photo on the left.



Panoramic view of Long Beach harbor as seen from the decks of The Queen Mary.

Dr. Jae-Hwan KIM of QST in Japan, pictured on the right in both photos, was recipient of the 2019 Mario Dalle Donne Memorial Award for outstanding achievement in the field of beryllium research for fusion.

Left: Drs. Hiroshi KAWAMURA and Glen LONGHURST, recipients of special BeWS Lifetime Achievement Awards for being two of the co-founders of the workshop, pictured with BeWS-14 Chair, Chris DORN.
Right: Dr. Anton MÖSLANG of KIT, long-time BeWS IOC Chair, receiving his BeWS Lifetime Achievement Award from BeWS-14 Technical Chair, Dr. Pavel VLADIMIROV, in a ceremony held at KIT after the workshop.

Left: Aniceto GORAIEB of KBHF, who holds the distinction of having attended more Be Workshops than any other individual participant (13 of 14), discussing the awards presentations with Glen LONGHURST.
Right: Keith SMITH of the leading BeWS-14 sponsor Materion, using his company's poster at the workshop's vendor exhibition to illustrate a finer point of beryllium use in fusion research for one of the participants.