Proceedings of the 15th International Workshop on Beryllium Technology (BeWS-15)

September, 14-15, 2022, Karlsruhe, Germany

Pavel Vladimirov, Christopher Dorn, Ramil Gaisin (eds.)
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by
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Executive Summary

The 15th International Workshop on Beryllium Technology (BeWS-15) was initially planned to be held as a satellite meeting of the SOFT-32 in Dubrovnik, Croatia in 2021. Due to the global COVID pandemic the SOFT-32 conference was held as a virtual event only and the International Organizing Committee (IOC) of the BeWS-15 decided to postpone the workshop until in person participation will be possible.

Finally, the Workshop was held as a joint event combining both the BeWS-15 and an industrial forum BeYOND-IX during 14-15 September 2022 in Karlsruhe, Germany with a great success. The workshop was organized by the Karlsruhe Institute of Technology. A company Karlsruhe Beryllium Handling Facility (KBHF) headed by A. Goraieb and Dr. M. Lemmens was responsible for the industrial forum and helped us with the organization of joint social events as well as. Due to still difficult pandemic situation in Asia, it was decided to organize the workshop as a hybrid event, i.e., provide a possibility for the participants having difficulties with travel with online access.

Participants came mainly from Germany, the US, the UK, Kazakhstan, Latvia, Czech Republic, Japan, Sweden, France and China, totaling 55 persons, which was not expected immediately after the global pandemic.

The BeWS-15 program agenda comprised two keynote presentations, which were followed by five technical sessions.

The first session was devoted to the memory of our colleague and friend Prof. Dr. Glen Longhurst who passed away on May 12, 2021. The session was started with the presentation given by Dr. Milan Zmitko from F4E, who reported on the status of Be neutron multiplier materials for the ITER Test Blanket Module (TBM) program and overviewed developments and opened issues. The final keynote was presented by Dr. Jae-Hwan Kim, QST, Japan, who gave an overview on R&D on Neutron Multipliers in Japan.

The five technical sessions featured presentations on

- “News from Industry” with contributions from Materion, USA, and Ulba Metallurgical Plant, Kazakhstan;
- Reports from big facilities “DEMO, ITER & JET” with contributions on the current advanced EU DEMO HCPB breeding blanket design, regulatory situation of Beryllium in EU and France, as well as investigation of JET beryllium tiles;
- The session on “Beryllides” summarized activities in this area performed worldwide by various institutions in Kazakhstan, KIT, Germany and QST, Japan;
- In the session on “Modeling and experimental validation” both high-quality experimental and simulation results on beryllium and beryllides were presented;
- The session “Mechanical properties and irradiation damage” contained contributions on mechanical and microstructural changes of beryllium and beryllides after helium implantation, creep of beryllium pebbles after irradiation as well as thermos-mechanical properties of beryllide pebbles.

The last day of the joint event was devoted to the industrial forum BeYOND-IX where contributions from Fusion-Start-Ups and Health & Safety aspects of beryllium fabrication and study were interchanged. This day has started from a Lab Tour visit to the Karlsruhe Beryllium Handling Facility situated in KIT Campus North.

The Prof. Mario Dalle Donne Memorial Award (MDDMA) was presented for the first time at the BeWS-11 in Barcelona to recognize researchers with outstanding achievements in beryllium-related research. This year, the IOC of BeWS awarded this prize to Mr. Aniceto Goraieb, the KBHF leader, based on his numerous technological achievements in beryllium-related research over the past decades. For him it was a special pleasure to receive this award as he was a student of Prof. M. Dalle Donne.

The IOC of BeYOND industrial forum decided to establish a Glen Longhurst Award for people working on the edge between industry and technological applications of beryllium materials. The Prof. Glen Longhurst Award was presented for the first time to Mr. Christopher Dorn for his long-term support for R&D on beryllium materials for their application for fusion technology working as one of the chief managers at Materion Inc. in USA, keeping beryllium safety standards and services at ITER in France and at JET in UK, and running his own consulting company on beryllium business.
SESSION 1

In memory of Glen Longhurst
In memory of Glen Longhurst (1943-2022)

Pavel V. Vladimirov¹, Christopher K. Dorn²

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This presentation is devoted to the memory of our colleague and dear friend, Prof. Dr. Glen Longhurst, who passed away on May 12, 2021.

Together with Mario Dalle Donne, Hiroshi Kawamura and Vladimir Shestakov, Glen was one of the co-founders of the Beryllium Workshop in 1991. After that, he was a member of the International Organizing Committee (IOC) for many years, and he actively worked on further development of the Workshop Series. In 2005, Glen hosted the BeWS-7 in Santa Barbara, California, USA as a satellite meeting of the ICFRM-12 conference, and the workshop was a great success. In 2012 during the BeWS-10 in Karlsruhe, he suggested the establishment of the Mario Dalle Donne Memorial award to honor outstanding contributions by scientists working in beryllium area.

In the field of beryllium technology, Glen was well known for his investigations of beryllium behavior under irradiation, its handling, recycling, application for fusion and, especially, for his studies on the retention of accumulated tritium within beryllium. Glen developed the Tritium Migration Analysis Program (TMAP) for simulation of tritium transport and release, which has survived through seven major releases and is still regarded as the gold standard in the field today.

A special award for lifetime achievement, in particular, for the establishment and development of the BeWS series, was presented to Prof. Emeritus G. Longhurst during the previous BeWS-14 held in Long Beach, California, USA in 2019. The following slides share some memories from this event.

To honor Glen’s achievements, the IOC of the BeYOND Industrial Forum has decided to establish a Glen Longhurst Memorial Award for exceptional persons working on the edge between industry and technological applications of beryllium materials.

Glen will be forever enshrined in our hearts as an outstanding scientist, a great personality, and a wonderful friend to us all.

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In memory of Glen Longhurst

Pavel Vladimirov and Christopher Dorn

Dr. Glen Longhurst was one of the co-founders of the Beryllium Workshop in 1991. He also served on the IOC for many years and worked to develop the BeWS series.

Glen was well known for his investigations of beryllium behavior under irradiation and, especially, for his studies on the retention of accumulated tritium within beryllium. Glen developed the Tritium Migration Analysis Program (TMAP) for simulation of tritium release and transport, which has survived through seven major releases and is still regarded as the gold standard in the field today.

A special award for lifetime achievement, in particular, for the establishment and development of the BeWS series, was presented to Prof. Longhurst during the BeWS-14 held in Long Beach, California, USA in October 2019.

Glen Longhurst: 1943-2021
Lifetime achievement awards were presented to Drs. Longhurst and Kawamura, two of the original co-founders of the Beryllium Workshop series. The ceremony took place at the BeWS-14 workshop dinner aboard the Hotel Queen Mary, a former British passenger ship, in Long Beach, California, USA in October 2019.

Glen Longhurst, pictured here with Aniceto Goraieb, Managing Director of KBHF, the Karlsruhe Beryllium Handling Facility, after the awards ceremony at the BeWS-14 workshop dinner aboard the Hotel Queen Mary.
From left to right: Aniceto Goraieb of the KBHF in Germany, Glen Longhurst, and his wife Jean, shown here at the BeWS-14 workshop dinner aboard the Hotel Queen Mary.

The Last Slide from Glen's Last Presentation

Thank you for your attention
Best of luck and success in your future work and meetings

The HCPB Test Blanket Module: Current Status in Development and Qualification of Beryllium Materials and an Overview of Open Issues

Milan Zmitko¹, P. Vladimirov², V. Chakin², A. Spagnuolo³

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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, 300-500°C).

The paper gives a brief general description of the HCPB TBM design and the main design requirements including the requirements to beryllium multiplier material. The ITER HCPB TBM development and qualification plan with identification of the main milestones will be presented, taking into account recently signed EU-KO TBM Partnership agreement between Fusion for Energy and ITER Korea.

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 achieved results on fabrication technologies development, materials characterization and performance under TBM/DEMO relevant conditions, including the performance under neutron irradiation and thermo-mechanical performance will be briefly overviewed and a new neutron irradiation experiment, foreseen for the functional materials (i.e. beryllium materials and ceramic breeder pebbles), will be introduced.

A special attention will be focused on technical issues related to the HCPB TBM design and manufacturing, like for instance, Be/ceramic breeder pebbles filling procedure and realization of post-weld heat treatment operations during the course of TBM box manufacturing, and an assessment of their possible implications.

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The HCPB Test Blanket Module: Current Status in Development and Qualification of Beryllium Materials and an Overview of Open Issues

15th International Workshop on Beryllium Technology
Karlsruhe, Germany
14-15 September, 2022

M. Zmitko – presenting author
P. Vladimirov, V. Chakin
A. G. Spagnuolo

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

Test Blanket Systems testing at ITER

“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 (Collaboration Agreement with KODA – HCCP TBM)

EUROFER97 structural material (RAFM steel)
EU HCPB: Helium Cooled Pebble Bed TBM

**EUROFER97 structural material**

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

Reference CB material: Advanced CB = mixture of Li$_2$SiO$_4$ (LOS) with Li$_2$TiO$_3$ (LMT) (~25-35 mol%) as a secondary phase (improved mechanical properties); produced by melt-spraying process; total weight of ~80 kg

Beryllium neutron multiplier in the form of pebble beds; total weight ~190 kg

Reference NMM: 1mm Be pebbles produced by REP (NGK, JA)

Alternative NMM: (i) Be chips UMP, KZ; (ii) Be mini-spheres (FRM) MATERION, US

DEMO/Future TBM: Advanced Be material: beryllium alloys/beryllides (e.g. Be$_{12}$Ti) → lower swelling, better T release, better oxidation resistance, smaller reactivity with structural material

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

**For illustration**

Stiffening Rods concept changed for Crossing Vertical concept

**Extension of vSP up to BP5, so called Crossing vertical concept**

M. Zmitko et al., The HCPB TBM: Current Status of Beryllium Materials and Open Issues
EU-KODA TBS collaboration & New schedule

EU and KO toward a joint implementation of the TBM Program

- The goal is joint implementation
- Follow-up of the IC decision 2018 to reduce total number of TBM in ITER
- 2+ years negotiations EU-KO

2. Beryllium multiplier materials
Functional Requirements for Functional Materials

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 FM systems and EUROFER (up to max. T~ 550ºC), in the reference purge gas He+0.1vol%H₂
- **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)

Beryllium materials R&D activities: Material Assessment and Database Reports

**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
HIDOBE irradiation campaign & PIE

**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_{5at%}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

**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]

**HIDOBE Post-Irradiation Examination**
**Microstructure of Be pebbles**

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

Effect of irradiation temperature on surface oxidation layer

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

SESSION 1 IN MEMORY OF GLEN LONGHURST
HIDOBE Post-Irradiation Examination

Tritium release characteristics

- Tritium released predominantly as HT
- One broad peak for T (830-1070°C) independently of irradiation temperature
- Slow T release starts always at 670-730°C

- He released sometimes in several peaks
- He release starts at 830-930°C

Residual Tritium retention of 1 mm Be pebbles as a function of irradiation temperature (related to theoretically calculated values of T production)

Microstructure of Be pebbles

- Swelling depends significantly on irradiation temperature
- Swelling of Be pebbles remains under ~10%
- Swelling of beryllide pellets remains under ~5%
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 525°C. Irradiations at 650 and 750°C 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°C. However, strong oxidation occurs after irradiation at 650 and 750°C 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 ~900-1100°C 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 650°C; at 750°C, the residual Tritium measured in irradiated samples is 6 to 8 times lower than in samples irradiated at lower temperature.

Characterization of reference Be pebbles

Characterization of as-received Be pebbles supplied by NGK Insulators Ltd., Japan
- 1 mm Be pebbles produced by Rotation Electrode Process (REP)
- Over 90% of pebbles in the size range of 890-1220 μm

Chemical analyses obtained by Ion Beam (in ppm)

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<td>Ti</td>
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</tbody>
</table>

Be oxidation in steam

Interaction of Be pebbles with air and steam → reaction kinetic determination

Be specimens after reaction with steam at different temperatures

620°C  720°C  750°C  800°C
Complementary Be pebbles oxidation studies (REM / NGK & FRM / MATERION pebbles)

1. The 2 mm Be pebbles fabricated by the FRM show similar corrosion behavior to the 1 mm REM Be pebbles.
2. An intensive oxidation in air starts since 700-800°C.
3. In steam, both kinds of Be pebbles have a protective oxide surface layer up to 650-700°C. Since 700-750°C, the oxide layer is already not protective. The temperatures of 700-750°C are critical for the use of Be pebbles in contact with water steam even during a short time exposure.
4. The intensive reaction with water steam should be avoided in fusion reactors because it results in the hydrogen production bearing a risk of hydrogen gas explosion.

Tritium release characteristics (REM & FRM pebbles)

Tritium release rate from Ø 1 mm beryllium pebbles produced by REM and FRM

Total tritium accumulation from Ø 1 mm beryllium pebbles produced by REM and FRM

Temperature Programmed Desorption (TPD)
Future activities, incl. new neutron irradiation experiment

3.

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

The main scope of the activity is to define a roadmap to secure the delivery of sufficient amount and requested quality of the pure Be pebble material for the ITER-TBM. The following activities should be addressed (some of them have already been addressed):

Supply Capacity Development

- Determination of supply capacities for a (semi-industrial) production of Be pebbles (for HCPB/HCCP TBM ~200kg) - reference & alternative production routes and respective suppliers; elaboration of a roadmap for delivery of Be materials for HCPB/HCCP TBMs providing a sufficient amount and quality of the Be pebble material
- Define a Procurement specification for Be pebbles, including acceptable level of impurities
- Delivery of small amount of REM Be pebbles by NGK, JA and Be ‘chips’ from UMP, KZ for a complementary characterization → fabrication trials before a real production/supply for TBM
- Production and characterization of Be pebbles supplied for 1st HCPB/HCCP TBM
- Regulatory (dual-use) and legal aspects shall be investigated for the Be material (pebbles) procurement and supply for ITER-TBM purpose
- Functional Materials Development, Qualification, Supply and Regulatory aspects – Return of eXperience (RoX) for DEMO - Be (& ACB) pebbles
Development & Qualification

- Characterization of Be pebbles **produced with an alternative production route** based on fluoride reduction method (FRM, Materion, US) and Ulba Be ‘chips’ (UMP, KZ) → i.e. chemical composition, impurities, microstructure, porosity, thermo-mechanical properties, oxidation characteristics, tritium release characteristics, ...
  - Characterization of FRM Be pebbles supplied by MATERION, US → **high level of impurities detected** *(activity already performed)*

- Complementary **oxidation studies** of reference NGK Be pebbles in air and water vapor; experiments at temperatures not covered by the previous activity *(activity already performed)*

- **Update** of the existing Functional Materials properties database *(Version for HCPB/HCCP PDR in 2023)*
  - Definition and realization of a **new neutron irradiation campaign** for ACB and Be materials → **very important step** in the Functional Materials qualification for their use in the ITER-TBM
  - Production & characterization of NMM samples (Be pebbles & beryllides) for a **new irradiation experiment**
  
  - Experimental study on development of a **filling procedure** for CB and Be pebbles into the HCPB/HCCP Breeder Units, taking into account the TBM assembly sequence and post-weld heat treatments *(PWHT)*

- **Update** of the existing Functional Materials properties database *(Version for HCPB/HCCP FDR in 2027)*
  - Important activity for ACB *(for info): Modelling of dust formation/re-suspension mechanisms in the Breeder Unit and V&V with experiments → a safety-related aspect*

---

Functional Materials Neutron Irradiation

- **New neutron irradiation campaign** of the Advanced Ceramic Breeder (ACB) and Neutron Multiplier Material (NMM) → irradiation facility(ies) under discussion/assessment *(WWR-K, Almaty - Kazakhstan; BR2, SCK-CEN Mol – Belgium)*

- Definition of the **scope of the experiment**, experimental matrix, experimental conditions and PIE for both ACB and NMM *(on-going)*

- 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 & Ulba Be chips)*

- **New irradiation experiment realization** *(irradiation parameters and exp. conditions still TBD)*
  - Irradiation temperatures of the irradiated materials *(irradiation temperatures for the ACB in the range of 500-900°C, of 350-650°C for pure Be pebble materials and of up 1000°C for beryllides)*
  - Irradiation dose / neutron fluence *(target irradiation dose for the ACB is ~2-3 dpa in steel, for Be pebbles ~2 dpa)*
  - Type of the experiment *(i.e. materials irradiation for subsequent PIE, in-situ on-line tritium release measurement for the ACB, compatibility test with structural material)*

- Post-irradiation activities *(dismantling, retrieval of irradiation drums, transport, waste management and permanent storage)*

- Post-irradiation examination of Be materials
  - Be pebbles microstructure and morphology with OM, SEM, TEM
  - Dimensional stability *(swelling*, pebbles’ size distribution, close and open porosity
  - Mechanical properties *(e.g. Vicker’s hardness, creep behavior)*
  - Tritium and Helium release characteristics *(with Temperature Programmed Desorption (TPD) process)*
  - Be pebbles compatibility with EUROFER97 structural material
R&D activities on Be pebbles

Summary

4. Beryllium materials prepared for irradiation in the W7-AS.
<table>
<thead>
<tr>
<th>Summary</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Beryllium Multiplier Materials:</strong></td>
</tr>
<tr>
<td>• The 1 mm Be pebbles produced by REP are at present the reference multiplier material for the first HCPB/HCCP TBM breeder blanket concept</td>
</tr>
<tr>
<td>• The reference Be pebbles/pebble beds are widely characterized, both out-of-pile &amp; in-pile available data collected in the MAR and MDBR</td>
</tr>
<tr>
<td>• 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</td>
</tr>
<tr>
<td>• 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</td>
</tr>
<tr>
<td>• Irradiation temperature most significantly affects the development of the material properties microstructure, tritium retention and release (mainly in HT form)</td>
</tr>
<tr>
<td>• Identification of an alternative production route for Be pebbles, including necessary characterization and qualification for ITER TBM use</td>
</tr>
<tr>
<td>• Identification of supply capacities and elaboration of a delivery roadmap for both reference and alternative Be pebbles to be used in ITER TBMs</td>
</tr>
<tr>
<td>• New neutron irradiation experimental campaign under definition &amp; planning; testing of alternative Be pebbles and beryllides (together with advanced ceramic breeder pebbles)</td>
</tr>
</tbody>
</table>

---

**Thank you for your attention**

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Overview of R&D activities on Neutron Multipliers in QST

Jae-Hwan Kim, Taehyun Hwang, Yutaka Sugimoto, Suguru Nakano, Yoshiaki Akatsu, Masaru Nakamichi

National Institutes for Quantum 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.

Since beryllides are too brittle to produce 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 beryllide pebbles using the plasma-sintered beryllide rod, a 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 general industry. The result of beryllide granulation revealed that the prototypic beryllide pebbles with 0.5 mm to 2.5 mm in average diameter were successfully fabricated. In this study, the recent progress on R&Ds of beryllides as the advanced neutron multipliers in Japan will be presented.

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Overview of R&D activities on Neutron Multipliers in QST

Outline
1. Necessity of advanced neutron multipliers
2. Synthesis of single-phased beryllides
3. Granulation of beryllide pebbles
4. Characterization of beryllide pebbles
5. A joining process to F82H by plasma sintering
6. Summary

Jae-Hwan Kim, Taehyun Hwang, Yutaka Sugimoto, Suguru Nakano, Yoshiaki Akatsu, Masaru Nakamichi

National Institutes for Quantum Science and Technology, QST

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Beryllium metal (Be) is a candidate material. There are some issues under high neutron fluence at high temperatures.

- Swelling
- Hydrogen generation with water vapor

Increase of swelling

Change of swelling

Change of \( H_2 \) generation rate

Temperature (˚C)

Be

\( Be_{12}Ti \)

\( Be_{12}Ti \)

Beryllide has a good potential for high temperature use.

Based on the common interests of the EU and JA, beryllide R&D is carried out as a part of the BA activities.

Plasma sintering is a non-conventional consolidation process, consisting of plasma generation, resistance heating and pressure application.

The plasma discharge results in particle surface activation that enhances sinterability and reduces high temperature exposure.

Pressure application assists the densification process by enhancing sintering and thus further reducing the high temperature exposure of the consolidation powders.

Comparison of methods

1. Joule heating by DC
   Die acts as a heating element

2. Heating by heater located outside of mold

3. Heating by heater with pressure of isostatic gas

Application of:
1) Uniaxial pressure
2) Plasma generation for powder surface activation
3) Resistance heating

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)


Plasma sintering process:
1) Uniaxial pressure
2) Plasma generation for powder surface activation
3) Resistance heating

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.

This has successfully fabricated by the plasma sintering.

Plasma sintering process is a simple, easily controllable process, which has short synthesis time (30% less than HIP) and good cost performance.
Single-phased beryllides (Be\textsubscript{12}Ti, Be\textsubscript{12}V, and Be\textsubscript{13}Zr) with high density have been successfully synthesized by a plasma sintering process. The synthesis was conducted at a temperature of 1000 °C, with a time of 20 min, and a pressure of 50 MPa. The density of Be\textsubscript{12}V was 2.37 g/cm\textsuperscript{3}.}

**Synthesis of single-phased Be\textsubscript{12}V beryllides**

**Plasma sintering**

- Temperature: 1000 °C
- Time: 20 min
- Pressure: 50 MPa

**SEM and Optical images**

**X-ray diffraction**

Single-phased beryllides (Be\textsubscript{12}Ti, Be\textsubscript{12}V, and Be\textsubscript{13}Zr) with high density have been successfully synthesized by a plasma sintering process.
Novel granulation method of Be$_{12}$V beryllide

**Synthesis of beryllide**

**Granulation using beryllide rod**

**Beryllide rod has successfully fabricated by the plasma sintering.**

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

**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

**Pebbles list that we have successfully fabricated so far.**

Be, Be$_{12}$Ti, Be$_{12}$Zr, Be-Ti-V, Be-Ti-Zr, Be-V-Zr beryllides

M. Nakamichi, Novel granulation process of beryllide as advanced neutron multipliers, FED 88 (2013) 611-615
Novel granulation method of Be$_{12}$V beryllide

*Be$_{12}$Ti pebble has a porous body by the homogenization treatment.
*To prevent increase of H$_2$ generation associated with increase of the specific surface area, other compositions of beryllides have been surveyed.


Be$_{12}$V composition was selected,
* No peritectic reaction,
* similar nuclear property to Be-Ti beryllide

Jae-Hwan Kim | BeWS-15, KIT | 14th 15th Sept. 2022 | page (10/24)
Optimization of granulation process

Pebble size is under control by a rotating speed

Pebble packing factor (targeting to 80%)

<table>
<thead>
<tr>
<th></th>
<th>Pebble size</th>
<th>Container dia., mm</th>
<th>Packing factor%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single packing</td>
<td>0.3-0.5</td>
<td>10</td>
<td>64.9</td>
</tr>
<tr>
<td></td>
<td>0.425-0.6</td>
<td>10</td>
<td>64.8</td>
</tr>
<tr>
<td></td>
<td>2.36-2.8</td>
<td>30</td>
<td>63.5</td>
</tr>
<tr>
<td>Binary packing</td>
<td>2.38-2.8</td>
<td>30</td>
<td>81.0</td>
</tr>
<tr>
<td></td>
<td>/0.3-0.5</td>
<td></td>
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</tr>
</tbody>
</table>

J.-H. Kim et al., Overview of R&D Activities On Neutron Multipliers In QST
Thermal conductivity of Be and Be$_{12}$V pebble beds

It was clear that the effective thermal conductivity of the Be$_{12}$V pebble bed was lower by 33% than that of Be pebble bed at 873 K.

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$_2$O (1~15%) were examined using a Thermo-Gravimetry/Differential Scanning Calorimetry apparatus connected with a gas chromatography.

Schematic flow diagram of test apparatus

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$_2$O
- Temperature: ~1473 K

(Ar gas flow during temperature ramping)
H₂ generation with 1% water vapor of Be₁₂V pebbles

- 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

Be₁₂V beryllide showed lower H₂ generation ratio than Be₁₂Ti and Be.


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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6. Summary
**Application of joining process**

Beryllium and tungsten joining

**Beryllium tiles**

Cu alloys

SUS

**Many applications**

**Beryllium and tungsten joining**

divertor

Many applications

**Sintering temp.**

923, 1023, 1123 K

**Sintering time**

30m, 60m, 90m

**Sintering pressure**

15 kN

**Dimension**

3 mm x 3 mm x 35 mm

**Sample number**

N = 3

**Bonding strength was measured**

---

**Results (1) : Mapping and activation energy for growth**

**Line analysis**

**Mapping**

**Arrhenius Plot**

116.2 kJ/mol

In the reaction layer, it was found out that Be-Fe compounds, Be$_{12}$Fe, Be$_5$Fe, Be$_2$Fe were formed.

By the thicknesses of joined samples at 923, 1023 and 1123 K for 90 min, the growth rate was evaluated, indicating the activation energy is 116.2 kJ/mol.
The bonding strength of the joined materials was inversely proportional to the thickness of the reaction layer. The difference in the fracture behaviour induced by either the delamination of grains or delamination along grain boundaries is attributed to the difference in the bonding strength.

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$_2$O 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.
• A joining process between Be and F82H by plasma sintering was tried. It was clear that the bonding strength of the joined materials was inversely proportional to the thickness of the reaction layer.

Thank you for your kind attention
SESSION 2

News from Industry
Overview of the United States Beryllium Industry - 2022 Update

Keith Smith¹

¹ Materion – Brush Inc Elmore, Ohio USA

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 updated overview of the US beryllium industry including mining, manufacturing, and fabrication capabilities. Beryllium raw material availability, ITER relevant First Wall beryllium grades, 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 including activities related to the manufacturing and purification of beryllium containing molten salt (FLIBE) be addressed.

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USA
Overview of the United States Beryllium Industry – 2022 Update

Keith J. Smith   VP Nuclear & Science Materion-Brush Inc. Elmore, Ohio  USA
Andreas Frehn PhD    Director of Technology & Innovation Materion-EMEA  Germany
14 September 2022

Materion at a Glance…
A global high-tech solutions provider of performance alloys, precision optics and advanced materials

- 100+ years of materials knowledge
- Publicly traded since 1972 - NYSE (MTRN)
- In excess of $1 billion in sales
SESSION 2  NEWS FROM INDUSTRY

Our People Make the Difference

- Materials scientists
- Chemists & physicists
- Applications & sales engineers
- Manufacturing & process engineers
- Customer-focused teams
- Technical support staff
- Supply chain, logistics, & materials management experts


**OUR BUSINESS SEGMENTS**

**Electronic Materials**
- Precious and engineered materials for sputtering and evaporative coating
- Advanced chemicals and powders
- Microelectronic packaging materials
- Large area targets for sputtering
- Precious metal life cycle management and refining services

**Performance Materials**
- Specialty engineered alloy systems
- Beryllium and composites
- Engineered clad and plated metal solutions
- Ceramics
- Advanced performance parts

**Precision Optics**
- Precision filters and optical coatings
- Wafer level coatings
- Specialty thin films
- Projection display components
- Optical assemblies
- Roll-to-roll thin film deposition and conversion

Plus recent acquisitions of Balzers Optics and H.C. Starck (tantalum and niobium business)
OUR GLOBAL MARKETS

51% Performance Materials
34% Electronic Materials
15% Precision Optics

Beryllium Supply Chain
Materion – Beryllium Processing

- Materion is the largest producer of beryllium containing materials in the world. Be mining – Be extraction – Be production – Be machining
  - We operate and invest in the most modern beryllium production facilities.
  - Over $100,000,000 (US) has been invested over the past 15 years.
  - New investments: New Machining facility, Upgrade of the Primary Beryllium Facility, FLIBE Prototype Manufacturing Facility, Mining Facility Upgrades.

Materion – Beryllium Mining

- 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$_4$Si$_2$O$_7$(OH)$_2$).
- Materion holds estimated ore reserves for 70 years of production.
  - USGS estimates 21,000 tons of Be reserves @ Utah.
Materion Resources, Delta, Utah

Materion Resources, Delta, Utah
Beryllium Mine Production

<table>
<thead>
<tr>
<th>World Mine Production and Reserves:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
<tr>
<td>United States</td>
</tr>
<tr>
<td>Brazil</td>
</tr>
<tr>
<td>China</td>
</tr>
<tr>
<td>Madagascar</td>
</tr>
<tr>
<td>Mozambique</td>
</tr>
<tr>
<td>Nigeria</td>
</tr>
<tr>
<td>Rwanda</td>
</tr>
<tr>
<td>Uganda</td>
</tr>
<tr>
<td>World total (rounded)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Reserves*</th>
</tr>
</thead>
<tbody>
<tr>
<td>The United States has very little beryl that can be economically hand sorted from pegmatite deposits. The Spor Mountain area in Utah, an epithermal deposit, contains a large bertrandite resource, which is being mined. Proven and probable bertrandite reserves in Utah total about 20,000 tons of contained beryllium. World beryllium reserves are not available.</td>
</tr>
</tbody>
</table>

U.S. Geological Survey, Mineral Commodity Summaries, January 2022

Performance Materials

- The only fully integrated manufacturer of Beryllium and Beryllium containing materials in the world.
  - Mining
  - Extraction
  - Conventional Metal Processing - CuBe/ CuNiSn/NiBe
  - Ceramic processing (BeO, Al₂O₃, others)
  - Powder metallurgy (Be, Al-Be, others)
  - Metal Matrix Composites (Al-B₄C, Al-SiC, others)
  - Clad and Plated Metal Strip
  - Machining - Fabrication
Materion Performance Materials

- Advanced Alloys provides upstream product lines in ingot, billet and plate forms
- Specialty Metals provides precision strip, rod and wire products
- Performance Solutions provides end-products and finished machined components

- Beryllium
- AlBeMet®
- E-Materials
- Ceramics
- Metal Matrix Composites
- BeCu
- Toughmet

Beryllium Products

Beryllium:
- Seven commercial grades of Beryllium metal:
  ▪ 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
S-65 Vacuum Hot Pressed Beryllium

- A Materion standard product produced for over 40 years.
- Fully qualified for ITER.
- Baseline FW beryllium for ITER.

Beryllium Fabrication

- Materion – Fabrication and Program Management
  - Fabricated Solutions Group – Elmore, Ohio
  - Application engineering
  - Program management.

- US Domestic Fabrication Capabilities
  - All fabricators maintain various levels of program management and applications engineering support to customers.
US Beryllium Fabrication Sources

- Materion – Elmore, OH
- General Dynamics – Cullman, AL
- LA Gauge – Sun valley, CA
- Peregrine Falcon – Pleasanton, CA
- Hardric Laboratories, Inc. – Waltham, MA
- WessDel – San Jose, CA
- Rev Manufacturing – Plamdale, CA
- Coherent, Inc. – Richmond, CA
- Skinner Machining – Cleveland, OH
SESSION 2  NEWS FROM INDUSTRY

Continuing to Accelerate Our Transformation Strategy

WHERE WE WERE
(Before 2017)
- Origins as a mining and metals-focused producer in 1931
- Strong heritage of beryllium expertise
- Several acquisitions broaden portfolio, creating the foundation for Electronic Materials and Precision Optics segments

WHERE WE ARE
(2017 - 2021)
- Becoming a global leader in advanced materials solutions through organic initiatives and strategic acquisitions
- Implemented One Materion strategy focused on driving profitable growth
- Increased investment in R&D, fueling innovation; significantly expanding growth pipeline through customer collaborations
- Transformed financial profile, doubling margins and returns

WHERE WE ARE GOING
(2021+)
- Accelerating global leadership in advanced materials solutions for evolving growth megatrends
- Delivering step-change organic and inorganic growth initiatives, driving transformative profitable growth
- Investing to deliver next generation solutions to support customer roadmaps
- Building on our leading positions in all major geographies
- Driving above-market top line growth and mid teens margins with strong ROIC

Materion: Value-added Partner with Long-term Relationships

We work with our customers to understand their current and future challenges...

...Applying our core competencies and expertise...

...To develop advanced materials with differentiated performance characteristics...

...That enable solutions supporting global growth megatrends

Customer Technology & Product Roadmaps

- Materials Science
- Compositional Synthesis
- Applications and Process Know-How

- Exceptional performance at extreme conditions
- High purity materials for demanding applications
- Superior thermal and electrical conductivity
- Highest-precision light management and sensing
- Unparalleled mechanical properties

Smart Devices
High Speed Connectivity (5G)
Clean Energy
Artificial Intelligence
Cloud Computing
Space
Sensing
Healthcare

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FLiBe Molten Salt Production

- Materion and Kairos Power are collaborating to produce FLiBE, a molten fluoride salt, as a coolant for the Kairos Power Fluoride salt-cooled High temperature Reactor (KP-FHR).

- The facility has been constructed at Materion’s Elmore, Ohio plant.

- Materion will operate the facility and provide both BeF$_2$ and LiF.

Molten Salt Purification Plant

➢ FLiBe Purification Facility

➢ Kairos Power / Materion Joint agreement to produce FLiBe (BeF$_2$ + LiF)
Additive Manufacturing

- Materion has initiated a comprehensive Additive manufacturing program.
- State of the art AM development center has been built at the Materion Technology and Innovation Center in Ohio.
- More detail will be discussed in a separate presentation this week.
Newton Acquisition Overview

OUR TEAM: 140 Employees

OUR EXPERIENCE: 70+ Years

OUR SITE: Located in Massachusetts, USA
ISO 9001 certified
20,300 m²

OUR METALS: Tantalum
Niobium

OUR MARKETS: Electronics
Chemicals & Superconductors
Aerospace & Defense

Next Gen Nuclear Energy

• Niobium rod preforms are critical superconductor components of high strength magnets required for Tokomak (magnetically confined) fusion energy

• Niobium and Tantalum play a key role in fusion energy as the global scientific community drives to demonstrate its viability, sustainability, and ultimately commercialization
Superconductor Industry

Applications
• MRI and NMR
• Mass spectroscopy
• Nuclear fusion energy
• Magnetic levitation
• Particle accelerators

Niobium and Tantalum Products
• Sheet
• Rod
• Wire

JWST – First Images

The James Webb Space Telescope is an all-Beryllium telescope.

The mirrors and structures are beryllium. The company also supplied additional beryllium components, electronic packages, and optical filters and coatings.
Explore, Inspire, Deliver, Repeat™

- Guided by a culture of continuous improvement that is creative, agile, and responsive
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- Partnering with our customers to deliver game-changing innovations
- Taking a systematic approach to delivering breakthrough solutions that create scalability
Beryllides - experience of UMP JSC in development and testing

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Intermetallic compounds of beryllium (beryllides) have outstanding characteristics in terms of heat resistance, hardness, and resistance to oxidation. For this reason, beryllides have potential applications in nuclear and thermonuclear energy, aerospace, instrumentation and other industries.

To date, the process of producing billets and products from beryllides has not passed into the stage of stable industrial production.

In recent years, UMP JSC has been making efforts to develop and implement technologies for producing billets and products from various beryllides, as well as testing and studying the properties of beryllides.

This article presents information on the results of producing billets and products from tantalum, titanium and chromium beryllides, as well as resource thermocyclic tests under conditions simulating the thermal modes of operation of helium-cooled blanket modules of the DEMO/may be ITER reactor.

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Ulba Metallurgical Plant, Kazakhstan

Uranium
Beryllium
Tantalum & Niobium
HF acid

Big press
Beryllium smelting furnace

Sergey Udartsev
Ulba Metallurgical Plant
**History of Be production in Ulba**

- 1951 $\text{Be(OH)}_2$ and $\text{BeO}$
- 1955 Metallic $\text{Be}$
- 1958 $\text{BeO}$ ceramics
- 1963 Be billets and parts
- 1971 Be powders
- 1973 Hot working of Be
- 2001 Beryllium-copper alloys (beryllium bronze)
- **2015 Beryllides**

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**Advantages of beryllides**

- High melting point
- Heat resistance
- High specific strength
- Hardness
- Resistance to oxidation at high temperature
- High $(n,2n)$ reaction rate for $\text{MBe}_{12}$ type of beryllides
Tantalum beryllide $\text{Ta}_2\text{Be}_{17}$

- Density: $5.05 \text{ g/cm}^3$
- Temp: $1980^\circ \text{C}$
- $D_{\text{max}} = 45 \text{ mm}$
- $H_{\text{max}} = 50 \text{ mm}$
- $T_{\text{th}}_{\text{max}} = 4 \text{ mm}$

CrBe$_{12}$ neutron multiplier blocks for DEMO fusion and fission reactors

- Density: $2.44 \text{ g/cm}^3$
- Temp: $1337^\circ \text{C}$
- Billet: $2.42 \text{ g/cm}^3$, $\phi 90\times90 \text{ mm}$
- Trefoil shaped block
- X-Ray phase analysis
TiBe\textsubscript{12} neutron multiplier blocks for fission reactors and DEMO fusion reactor

2.26 g/cm\textsuperscript{3}

Tmp = 1600°C

N\textsubscript{Be} = 92.3% at.

Hexagonal closed-shaped block

Hexagonal fragmented block

Billet

2.23 g/cm\textsuperscript{3}

ϕ150×170mm

Industrially manufactured TiBe\textsubscript{12} neutron multiplier block for DEMO fusion reactor ϕ150×170 mm
Looking for non-nuclear application of TiBe$_{12}$

Billets processing facility

Six-bladed TiBe$_{12}$ impeller $\varnothing$ 50x75 mm

Ulba Metallurgical Plant
Sergey Udartsev

Thermal cycling tests of TiBe$_{12}$

The heating from 200°C to 900°C
In 60 sec
(11.7 °C/sec)

The exposure at 900°C
for 45 sec

The cooling from 900°C to 200°C in 60 sec
(11.7 °C/sec).

No fracture, no cracks, no surface changes

$\varnothing$40x20 mm
50 cycles

$\varnothing$20x20 mm
3 cycles

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Thermal cycling tests of TiBe\textsubscript{12}

Thermal cycling of large TiBe\textsubscript{12} samples

150×30×40 mm

T_{\text{min}} 350°C
T_{\text{max}} 850°C
The heating rate 1.4, 0.3, 0.6 °C/sec
The cooling rate 0.6 °C/sec in air atmosphere

No fracture,
no cracks,
no surface changes
Thermal cycling of full-size closed-shape block

- \( T_{\text{init}} = 300^\circ \text{C} \)
- \( T_{\text{fin}} = 800^\circ \text{C} \)
- Heating rate – 0.3\(^\circ\)C/sec
- Water cooling rate – 200 ml/sec
- Helium atmosphere

The block fractured during the first cycle into several big pieces

Thermal cycling of full-size fragmented block according to the updated design

No fracture, no cracks

200 cycles
Long-term thermal cycling of the full-size fragmented block

The purpose: 3,000 cycles

After 200 cycles

After 300 cycles

No fracture, no cracks, no decrease in density

Accelerated time-laps video of one thermal cycle
Conclusions

Test prototypes for the high temperature application were manufactured from Ta$_2$Be$_{17}$.

Sufficiently dense homogeneous products from chromium beryllide CrBe$_{12}$ with a size of Ø 90×90 mm were successfully fabricated. Production can be scaled up to much larger workpieces.

Sufficiently dense homogenous titanium beryllide TiBe$_{12}$ billets Ø150×170 mm and various products for nuclear and non-nuclear applications can be manufactured by UMP on industrial facilities.

Thank you for your attention!

We invite you to cooperation!
Beryllium Additive Manufacturing

FritzCarl Grensing\textsuperscript{1,2}, Andreas Frehn\textsuperscript{3}, Carole Trybus\textsuperscript{2}, Andrew Ruzek\textsuperscript{2}, Rhea Christopherson\textsuperscript{2}, Jacob Huxol\textsuperscript{2}

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Beryllium components have historically been produced via powder metallurgy processes followed by subtractive machining operations. In the last 25 years, rapid advances in additive manufacturing have occurred in aluminum, titanium and many other materials while little to no work was done on beryllium. In 2021 Materion installed the first dedicated beryllium additive manufacturing laboratory with the goal of both developing additive technology for beryllium materials and understanding and mitigating the Environmental, Health and Safety issues associated with additively processing beryllium. Metallurgical results obtained will be described as well and the EHS data.

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Beryllium Applications

Electro-Optics
- Visible Light & IR Mirrors
- Optical Support Structures
- Instrumentation

Nuclear
- Fusion Reactor Walls
- Neutron Moderators
- Nuclear Reactor Coolant

X-Ray
- Radiation Windows
- Beam Pipes

Alloying Element
- Non-Sparking, Non-Magnetic Tools
- Molding Dies
- Corrosion Resistant Heavy Equipment

F. Grensing et al., Beryllium Additive Manufacturing
Potential Materials for AM

Beryllium (Be)
- Current technology has very high buy to fly ratios, long lead times, and limited flexibility
- Best AM value proposition

Aluminum-Beryllium (AlBe)
- Alternative to Be in lightweight and stiff applications
- Lower material and machining costs than Be
- More damage tolerant than Be
- Used in sensors, electronics, and housings

Beryllides (Beryllium-transition metal alloys)
- Lightweight with very high melting points
- Significant research on binary systems

Beryllium Oxide (BeO)
- Thermally conductive but electrically insulating ceramic
- Thermal spreaders and power applications

History of Be-Based AM

2004-2008: Materion led E-Beam Powder Bed Fusion Efforts on AlBe
- Very early ARCAM machine with minimal control systems
- Lack of sufficient thermal control resulted in defects
- Materion concluded that AM system controls needed further maturation

2008-2018: Materion continued to keenly follow and assess the technical developments in additive manufacturing

2019: Materion concluded sufficient advancement in AM has been achieved to deliver the required quality and flexibility to produce parts on a commercial scale
- Performed in-depth review of the AM industry, evaluated 92 equipment vendors on a ranked matrix and determined the best available technology for Be-based AM

2020: Commissioned our Safe Be Materials AM Lab

2021: Work on Be materials began
Materion Additive Manufacturing Laboratory

Materion has built a laboratory to investigate and develop multiple additive manufacturing processes for Beryllium containing and other advanced materials.

Due to health and safety considerations for Be, the laboratory has strict access, ventilation, and environmental controls.

In addition to developing the technology, the laboratory will validate the appropriate health and safety procedures for Additively Manufacturing Beryllium and other advanced materials.

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Materion’s Additive Manufacturing Development Plan

**Phase 1 – Patterns and Fixtures (Since 2013)**
- AM investment castings (IC) patterns for AlBeCast® product line
- Plastic AM printed parts for simple fixtures and demonstration parts
- IC customers requesting optimized topology

**Phase 2 – Process and Specification Development (2020-2024)**
- Operate prototype AM Development Lab to evaluate technologies
- Obtain customer support and acceptance of Materion AM-based parts
- Capture external funding for the AM lab
- Identify and select production-capable AM technologies

**Phase 3 – Full Production and Commercialization (2025 and Beyond)**
- Production AM capability
- AM of Beryllium-containing components
- Design for AM will provide highest performance solutions to our customer base
Laser Powder Bed Fusion (LPBF) Of Beryllium

Beryllium LPBF had never been attempted before, so fundamental development necessary.

- Melt Pool Size
- Layer Thickness
- Energy Density
- Scan Velocity
- Stress Relief

These parameters require optimization based on the characterization of the resultant material.

- Volumetric Density
- Microstructural Analysis

Current best recipe has minimal porosity and an intertwined columnar grain structure.

- Result after hundreds of samples
- Still significant room for improvement
- Initial mechanical property testing is underway

Surface roughness, support structures and other build requirements need to be developed.
Binder Jet Printing of Beryllium

Parallel efforts to development binder jetting printing of beryllium.

- Green components can demonstrate through features (Walls and Gaps).

Densification upon sintering remains a challenge.
- Density is not expected to match that of laser powder bed fusion, however, further densification is desired.
- Trials to improve densification are ongoing.

Supporting Capabilities and Heritage Systems

- Gas Atomization, Powder Processing and Characterization
- Hot Isostatic Pressing, Post Processing and Machining
- Chemical, Mechanical and Microscopic Analysis
- Metrology, NDT and Radiography
- Beryllium Metallurgical Expertise
- Labs and Testing Capabilities
Conclusions

• Materion is developing additive manufacturing for beryllium and other specialty engineered materials.
• Current efforts are in the laboratory phase and not yet commercialized.
• As additive manufacturing of beryllium approaches maturity, industrial partners and future customers will be needed for trade studies, pathfinder parts and specification evaluation.
• The goal is to bring a robust beryllium additive manufacturing solution to market and change the paradigm of beryllium utilization.
SESSION 3

DEMO, ITER & JET
Current design of the EU DEMO Helium Cooled Pebble Bed breeding blanket

Guangming Zhou¹, Francisco A. Hernández¹

¹Karlsruhe Institute of Technology

In the Work Package Breeding Blanket (WPBB) of the European DEMO program, the Helium Cooled Pebble Bed (HCPB) breeding blanket is one of the two driver-blanket candidates for the European DEMO and to be tested as test blanket module (TBM) in ITER. In the Pre-Concept Design (PCD) phase (2014-2020), within the framework of the EUROfusion consortium in Europe, the design of the HCPB breeding blanket has been changed to address various challenges facing the HCPB blanket concept. One of the big challenges was the use of Beryllium pebbles as the neutron multiplier in the previous design. Irradiation campaign showed that the tritium retention in the Be pebbles could impose severe safety issues and exceed the tritium limit of EU DEMO. Beryllides, on the other hand, have better properties in terms of volumetric swelling, tritium retention, irradiation and melting temperature.

This talk will focus on the current design status of the European DEMO HCPB breeding blanket and conclude with future activities in the Concept Design phase (2021-2027).

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Current design of the EU DEMO Helium Cooled Pebble Bed breeding blanket

Dr. Guangming Zhou
Lead Engineer of HCPB Breeding Blanket

Outline of content

• Status at the end of Pre-Concept Design Phase (2014-2020)
• Identified risks
• Design activities to address the risks
• Outlook
Status at the end of Pre-Concept Design Phase (2014-2020)

- Coolant: He @80 bar, 300-520°C
- Fuel-breeder pins containing advanced ceramic breeder (ACB) pebble bed
- Pins inserted into blocks of Be12Ti neutron multiplier
- Structural steel: Eurofer97
- Purge gas: He + 0.1vol% H2 @2 bar
- Easier manufacturing, easier filling of pebbles
- NA, TH & TM; TBR = 1.20; Ppump per blower < 6 MW; satisfying shielding

Identified risks related to HCPB BB

1. Low reliability of BB system under DEMO conditions [due to welds failure]
2. Loss of structural integrity of beryllide blocks
3. High pressure drops in coolant loop contributing to total high pumping power
4. Large tritium permeation rates at the interface of breeder-coolant loop
5. Low BB shielding capability
6. Degradation of Eurofer at contact with pebbles in purge gas environment
7. Reduction of structural integrity of blanket during shutdown due to Eurofer irradiation embrittlement
8. Low TRL of Codes & Standards for design of DEMO components
Proposed design changes for improvements

- Equalize purge gas and coolant pressure to eliminate in-box LOCA welds to improve reliability
- Increase ΔT (300°C-530°C) to further reduce pressure drop
- Re-arrange flow scheme to cool key structure with fresh coolant
- Shape of Be12Ti block to square

Tritium breeding ratio (TBR) optimization

P1. Study influence of ACB in back side of the pin (whole length of back side of pin)
P2. Study reduction of the front pin cladding distance to FW
P3. Study influence of Be12Ti radial length
P4. Study influence of Be12Ti block gaps
P5. Introduction of a Be12Ti rod in the inner tube
P6. Introduce Be12Ti in ACB pebbles (on both sides)
P7. Like P6, Introduce Be12Ti in ACB pebbles (only on inner side)
P8. Combined the positive effects

• Combined all positive effects, TBR = 1.17 > 1.15
Thermal and structural analysis

FEM model
Temperature field
Power density
Stress field (P)

Tin / Tout = 300 / 530 °C
Temp. within design limits
Stresses of steel are within allowables of code

Assessment of pebble-Eurofer interaction

- Acc. to [1], the fatigue lifetime reduced due to interaction between pebbles and Eurofer97

- Creep-Fatigue-Assessment tool [2] used to assess different design options (2 bar vs 80 bar purge gas)

- Along the indicated paths, most regions failed to withstand the required 7787 cycles
- Along the indicated paths, most regions succeeded to withstand the required 7787 cycles

- New design able to improve lifetime.

Shielding design (1/2)

- Parametric neutronics analysis [3]
  3D MCNP model by SuperMC
  - Baseline: 15 cm Eurofer
  - v1: 1 cm B$_4$C, 14 cm Eurofer
  - v2: 2 cm B$_4$C, 13 cm Eurofer
  - ...-v5: 5 cm B$_4$C, 10 cm Eurofer
  - ...-v10: 10 cm B$_4$C, 5 cm Eurofer
- Tritium and helium production in B4C

Nuclear heating in B4C and Eurofer used as input for structural design of the shield.

At least 9 cm B4C is needed for meeting all the requirements. Due to fragmentation of B4C, container of B4C is needed.

Maximum T and He production is in v10, 1.84 mole (5.52 g) T per FPY, 500 mole (2 kg) He per FPY in EU-DEMO.

Negligible, 120 kg T/fpy in EU-DEMO $\ll 1 \times 10^{-11}$ Pa m$^3$/s m$^2$ Outgassing limit 1e-11

Shielding design (2/2)

- Structural design
  To confine the fragmentation, B4C is designed to be contained.

Concept 1: Radiation, shield fixed to cover plate

Concept 2: Contact, shield fixed to BSS backplate

Concept 3: Contact, shield fixed to BSS backplate with external clamping
Global segment hydraulics

Porous media approach [4]
Totally in COB
1232 pins
860 FW channels
Pressure drop: 0.96 bar
Flow distribution relative OK

Tritium Extraction and Removal (TER) system

• Reference design
➢ Two stages in series
   First the adsorption of Q2O on the Reactive Molecular Sieve Bed (RMSB), thereafter the adsorption of Q2 on the Cryogenic Molecular Sieve Bed (CMSB) at 77 K.
➢ Tritium recovered via isotope exchange on RMSB and by heating-up of the CMSB.
➢ Extrapolated to DEMO scale is realized with industry.

• Outlook
➢ 80 bar purge gas, introduced to improve reliability of BB, results show that TER operating at 80 bar not a issue.
➢ CMSB requires large amount of liquid N2, getter bed is explored as alternative.
➢ Wetted purge gas to have a higher isotopic exchange rate compared to H2 and oxidized Q2, reducing permeation.

Tritium permeation analysis

- 3D component level solver [6]
  ➢ Developed based on the OpenFOAM and benchmarked with TMAP 7
  ➢ T release model
    Grain surface release model based on irradiation T release experiment [7]

- T permeation analysis
  ➢ T permeation analysis under 2 bar pressure purge gas vs 80 bar pressure purge gas, with same H2 partial pressure
  ➢ Wetted purge gas vs dry purge gas

<table>
<thead>
<tr>
<th>Purge gas</th>
<th>Permeation to coolant</th>
<th>Wall T inventory</th>
</tr>
</thead>
<tbody>
<tr>
<td>200Pa H2, no H2O</td>
<td>0.077% of T generation 290 mg/d</td>
<td>85 ng</td>
</tr>
<tr>
<td>200Pa H2 + 200Pa H2O</td>
<td>0.022% of T generation 83 mg/d</td>
<td>19.2 ng</td>
</tr>
</tbody>
</table>


Outlook

- At end of 2022, the milestone of preliminary conceptual design of the HCPB blanket shall be reached.
- At second half of 2024, the milestone of reference conceptual design for the HCPB blanket shall be reached, together with R&D programme.
- At the end of 2024, the driver blanket for EU-DEMO will be selected from the HCPB and WCLL concepts.
- From 2025 to 2027, the selected blanket will be further consolidated and qualified via design and R&D activities.
Contributors & Acknowledgements

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Since the last Beyond event that I attended in Karlsruhe, Germany, in 2018, the regulatory situation of beryllium in EU has evolved.

At EU level, a harmonized Occupational Exposure Limit (OEL) for beryllium has been adopted in July 2019. Member States had 2 years to implement it in their national law. Furthermore, Beryllium has been assessed for an eventual restriction in the Electrical and Electronic Equipment (EEE) under the RoHS Directive (Restriction of Hazardous Substances in EEE). The assessment started in February 2018 and the conclusion has been published in March 2021 by the European Commission: it is not recommended to restrict beryllium. Another important point is that it was decided to keep beryllium in the list of Critical Raw Materials (CRM) for the EU (last list published in September 2020). Lastly, this presentation could be the opportunity to introduce new concepts currently discussed in the frame of the green deal and the Chemicals Strategy for Sustainability, including the ongoing revision of the main EU chemicals regulations (REACH and other sectorial directives). Essential Use Concept, Safe Use Concept or General Risk Approach are emerging new concepts which could have impacts on beryllium.

At French level, ITER being based in France, it is important to mention the implementation of the EU OEL in France since the 1st March 2022 (Decree No. 2021-1849 of December 28, 2021).

In a second part of the presentation, I suggest to introduce the Be Responsible Program (www.berylliumsafety.eu) developed by the Beryllium Association (BeST) in which NGK is involved and our actions to promote these good practices to the users of our beryllium-containing products. I was honored to present our beryllium good practices in the frame of trainings for ITER beryllium workers organized by INSTN (Institut des Sciences et Techniques du Nucléaire), several sessions in French and English. As French SME processing articles in copper-beryllium alloys, we also were very honored to share our experience during the conference on occupational cancers organized as part of the French Presidency of the European Union on March 07 and 08, 2022 in Paris.

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1. Regulatory situation of beryllium: last update
   ☑ Context reminder (main uses, risk & CLP classification)
   ☑ RoHS Directive (Restriction of Hazardous Substances in EEE): Be evaluation (2020)
   ☑ EU CRM (Critical Raw Materials) list: Be still CRM in 2020, next list 2023
   ☑ Chemicals Strategy for Sustainability CSS (Green Deal): ongoing REACH revision and new concepts

2. Good Practices Program Be Responsible: last update
   ☑ Be Responsible Program developed by BeST www.berylliumsafety.eu
   ☑ Webinars
   ☑ Trainings for ITER workers
   ☑ NGK Beryllium testimony at the French Presidency of the EU
Risk and classification:

- Risk by inhalation of fine airborne particles (< 10 microns). Risk limited to the workplace. Individuals who are sensitized (allergic) to beryllium and exposed can develop a chronic lung disease: Chronic Beryllium Disease (CBD) also called berylliosis. This is the consequence of a reaction of the immune system at the level of pulmonary alveoli (formation of granulomas). CBD can be treated but is not curable. It can be fatal.

- EU CLP Regulation (Classification): Beryllium is classified Carcinogen 1B (H350i: can cause cancer by inhalation) for all forms insoluble (metal) and soluble (compounds).

- Recent epidemiologic studies (Dr. Paolo Boffetta) found no excess cancer risk in workers at plant sites that only used insoluble forms of beryllium. The Beryllium association BeST aims to reclassify beryllium metal as Carcinogen 2 differently from beryllium compounds. Ongoing revision of the EU CLP is an opportunity.

- REACH evaluation (Risk Management Option Analysis RMOA published by BAuA in 2016): Beryllium is not Substance of Very high concern SVHC on the candidate list, not under authorization, not under professional restriction.

New EU and French Beryllium Occupational Exposure Limit OEL:

- Beryllium has been included in the Carcinogen and Mutagen Directive CMD on 11 July 2019 (3rd revision): Directive (EU) 2019/983 amending 2004/37/EC: 0.6 µg/m³ to be followed by 0.2 in July 2026

- National transposition of the EU CMD Directive in France: French Decree 2021-1849 published on 28 December 2021 and entered into force on 1st March 2022
  - French OEL previously indicative at 2 µg/m³ now binding at 0.6 µg/m³
  - 0.2 µg/m³ in 2026
**Beryllium evaluation under the RoHS Directive:**

(Restriction of Hazardous Substances in Electrical and Electronic Equipment EEE)

- Beryllium is not restricted under RoHS (Annex II of RoHS: 10 substances restricted to date including lead, mercury, cadmium and hexavalent chromium)
- A new evaluation of Beryllium has been launched in February 2018 for a possible restriction under RoHS (Pack 15 featuring 7 substances including beryllium)
- The evaluation report dated November 2020 has been published by the European Commission in February 2021
- Conclusion: « the inclusion of beryllium and BeO in ANNEX II of RoHS is currently not recommended »
- Excerpts: « High technological importance of beryllium for the EEE sector » « especially as it comes to medical and safety relevant products » « the health hazards of beryllium and BeO, in particular CBD and even beryllium sensitisation can be regulated through an Occupational Exposure Limit » « progress in implementing exposure controls in order to meet established OELs of 0.6/0.2 µg/m³ for airborne beryllium at mechanical and thermal plants throughout the EU » « same measures can reduce the release of other pollutants »
- Ongoing RoHS general review expected for 2023

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**Beryllium Critical Raw Material (CRM) to the EU:**

- Beryllium is in the CRM list since the first list in 2011
- CRM List updated every 3 years: 2011 (14 CRMs) - 2014 (20) - 2017 (27) – 2020 (30)
- New list expected in 2023 (BeST involved in ongoing workshops: SCREEN, MSA)
- 3 criteria:
  - Supply risk
  - Economic importance
  - Non-substitutability
- BeST member of the CRM Alliance: « need of a unique approach in regulation and policymaking when addressing CRMs to avoid overregulation, innovation barriers, loss of EU competitiveness and societal well-being »
Chemical Strategy for Sustainability CSS (EU Green Deal):

➢ Ongoing review of REACH and CLP regulations + RoHS and ELV Directives
➢ REACH and RoHS revision proposals expected for 2023. BeST proactive in workshops and public consultations
➢ 2 new concepts to be introduced:
   - Essential Use Concept EUC
   - Generic Risk Management Approach GRA
➢ Simplistic hazard-based approaches in which the real risks are not considered \( \text{Risk} = \text{Hazard} \times \text{Exposure} \). Impact on innovation, competitiveness of the EU industry and achievement of Green Deal objectives.
➢ BeST is defending a « safe use approach », i.e. a risk-based approach versus a mere hazard-based approach. Common position of EU industry (ASMOR : Alliance for Sustainable Management of chemical Risk)
➢ More than 80 actions in the CSS are to be implemented by 2024. Other vigilance point: Sustainable Product Initiative & Circular Economy Action Plan (Safe and sustainable by Design SSdB concept, mandatory recycling targets)
➢ BeST message:

"BeST stresses the need for a coherent and balanced approach. The regulatory actions stemming from the CSS should be developed in coordinated manner, coupled with proper impact assessments to determine benefits and drawbacks. To meet the green deal challenges, we will need to use hazardous chemicals. Simplistic and unscientific statements on phasing out hazardous chemicals will be counterproductive. A real risk-based approach is essential: Risk = Hazard x Exposure."

2. Good Practices – Beryllium Product Stewardship Program – Be Responsible

☑ Be Responsible Program www.berylliumsafety.eu
➢ Launched by BeST in 2017
➢ Website accessible (free of charge) in English : www.berylliumsafety.eu
➢ Summarized web version in all European languages including French : www.berylliumsafety.eu and German www.berylliumsicherheit.de
➢ Kit of 12 guides available in English and French (+ DE, IT, SP) :
   ✓ 3 generic guides : Health and Safety / Hygiene & Personal Protective Equipment / Exposure assessment
   ✓ 9 guides for the most frequent operations in industry
     Low inhalation concern operations (CNC machining / Sawing / Stamping)
     Likely inhalation risk operations (Machining by Electro-erosion EDM / Sandblasting, grinding, rectification, polishing / Fusion / Foundry / Heat Treatment / Forging / Welding)
Identification of sources of exposure

Beryllium worker protection model based on 8 principles: limiting the emission and dispersion of fine particles

1. Keep Beryllium Out of the Lungs
2. Keep Beryllium Off the Skin
3. Keep Beryllium Off the Clothes
4. Keep Beryllium at the Source
5. Keep Beryllium in the Work Area
6. Keep Beryllium on the Plant Site
7. Keep Beryllium Work Areas Clean
8. Keep Beryllium Workers Prepared
Risk Management Measures

- **Personal Protection:** Exposure Assessment & Personal Protective Equipment (PPE) at the workplace.
- **Cleanliness:** appropriate Housekeeping & Hygiene Measures.
- **Work Area:** Access & Engineering Controls (exhaust ventilation, wet process etc.).

**Controlling Dust Emission & Dispersion**

- **Be Responsible Program – Webinars by BeST « Working safely with Beryllium »**

Since 2020, BeST has proposed 3 up to 4 webinars /year: 2 in English, 1 in French and 1 in German - Next in German scheduled on Thursday 20 October 2022.

- Communication: Newsletters, Easy Guide Blast, Web etc..
Beryllium Training courses for ITER workers (INSTN – CEA)

- In cooperation with the ITER occupational physician

Testimony as part of the French Presidency of the EU (conference on occupational cancers – French Labor and Health Ministries on 07 & 08 March 2022 in Paris)

- Testimonial from a company that has conducted a prevention initiative: NGK BERYLCO France (Beryllium sector)
Beryllium in JET with the ITER-Like Wall: Fuel retention, oxidation, melt erosion, dust

M. Rubel¹, A. Widdowson², I. Jepu², L. Dittrich¹, T.T. Tran³, J. Grzonka⁴,⁵, E. Fortuna-Zaleśna⁴, S. Moon¹, P. Petersson¹ and JET Contributors*

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Joint European Torus (JET) functions with the ITER-Like Wall (JET-ILW): beryllium in the main chamber and tungsten in the divertor. Be is used in the form of castellated bulk metal limiter blocks and evaporated coatings on the inner wall cladding (IWC) tiles. The operation with Be plasma-facing components (PFC) involves a spectrum of nuclear safety issues arising from plasma-material interactions: erosion, fuel retention, co-deposition with oxygen, melt damage including metal splashing and melt layer propagation, dust generation and mobilization, behaviour under massive water (liquid, vapour) or air leak, impact on invessel diagnostic components.

All above mentioned aspects of Be erosion and migration have been studied in detail with a range of methods including high-resolution in-vessel photographic survey, over twenty exsitu ion-, electron-, photon-based material analysis techniques and dedicated laboratory experiments performed on materials retrieved from JET. Results are summarized as follows.

- Be-based dust occurs in two forms: co-deposited layers peeled off from PFC and droplets released from the limiters following high-heat loads and consequential target melting.
- Be splashes stick firmly to surfaces thus creating a minimal risk for further mobilization.
- The amount of loose Be dust after a full experimental campaign comprising 20 h of plasma operation is assessed at the level of 0.05-0.1 g, i.e. 5-10% of the total mass of dust.
- Deuterium content in co-deposits on so-called wall probes is 1-2x10¹⁷ cm⁻², i.e. it is low.
- Total D retention in Be coatings on IWC, 5.3×10²², is on the same level as on Be limiters.
- Optical performance of test mirrors in the divertor is fully degraded by Be deposition, while mirrors in the main chamber wall maintained reflectivity.
- Depth profiles of Be and oxygen in co-deposits are of the shape indicating the in-vessel origin of O (gettering) but not post exposure oxidation when PFC was in contact with air.
- No mobilization of Be dust was detected in connection with the operation of remotely handled robotic arm used for in-vessel work at shutdowns.
In summary, all these points answer specific questions formulated at JET and ITER. A comprehensive report will contain research details accompanied by a critical assessment of results with emphasis of their weight and possible impact on the use of Be in future devices.

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Beryllium in JET with the ITER-Like Wall:
*Fuel retention, oxidation, melt erosion, dust*

Marek Rubel, Anna Widdowson, Ionut Jepu, Laura Dittrich, Tuan T. Tran, Justyna Grzonka, Elzbieta Fortuna-Zalesna, Sunwoo Moon, Per Petersson and JET Contributors

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JET tokamak with the ITER-Like Wall:
Plasma-facing components: Beryllium and Tungsten

All categories of PFC tiles have special structural features.
**Motivation and Aims**

**Driving force in studies: Safety & economy of reactor operation.**

Operation with Be wall components involves a spectrum of nuclear safety issues arising from plasma-wall interactions:

- Erosion, migration re-deposition (co-deposition),
- Fuel retention,
- Melt damage, melt layer propagation & metal splashing,
- Dust generation and mobilisation (disruptions),
- Impact of erosion products on in-vessel diagnostics,
- Risks related to massive coolant (water, vapour) leaks.

All processes need to be studied and (if possible) quantified to enable reasonable predictions for a reactor.
Handling beryllium and tritiated components
Beryllium Handling Facility (BeHF) at JET

All JET materials go through BeHF before shipment for ex-situ studies.

Analysis: A mosaic of methods
A "mosaic" of over 40 different methods has been used in our studies of wall components.
Beryllium Limiters

Fuel retention on Plasma-facing surfaces

**Aim:** Comparison of retention after campaigns finished with different fueling.

**ILW 1:**
- D: $90 \times 10^{15}$ cm$^{-2}$,
- H: $61 \times 10^{15}$ cm$^{-2}$,

→ D content higher than H

**Messages:**
- The content of hydrogen isotopes in Be limiters is small.
- Main impurities: O, C, N and Ni (*Inconel components*)
- Operation with H$_2$ fuel at the end of ILW-2 eliminated D from the surface layer.

**ILW 2:**
- D: 0 cm$^{-2}$,
- H: $185 \times 10^{15}$ cm$^{-2}$,

→ No deuterium in the surface layer.

Laura Dittrich, 47th EPS, 2021
Be upper dump plate ILW1-3: Melt zone composition

Motivation: Analyses of MkI-Be divertor tiles (1995) suggested possible higher fuel retention in molten Be areas.

Aim: To determine the composition of the melt zone.

Main results
- On average 99 at.% Be
- ~1 % at. oxygen
- No co-deposited N, C, Ni
- < 0.1 % at. H isotopes

Messages:
- Melt zones are cleaner than not melted Be areas.
- They are cleaner than unexposed reference samples.

Beryllium-coated Inner Wall Cladding

Laura Dittrich, 47th EPS, 2021
Be-coated Inconel: Inner Wall Cladding

**Aim:**
Assess the Be erosion and fuel retention, the share in the total retention in JET

**Total Be-coated IWC area** $5.36 \text{ m}^2$

**Analyzed tiles:**
- Reference: unexposed
- Tile 403 exposed to JET-ILW 1-3
- Tile 106 exposed to JET-ILW 1-2
- Tile 412 exposed to JET-ILW 1-3

---

**Be erosion and deuterium content.**

**Messages:**
- Small variations in the Be layer thickness.
- No erosion through the whole layer.

- D content: $0.2 - 4.1 \times 10^{18} \text{ cm}^{-2}$.
- Averaged D content: $1.0 \times 10^{18} \text{ cm}^{-2}$.
- Extrapolated D inventory in IWC:
  $5.3 \times 10^{22} \text{ D}$ corresponding to 176 mg D.
- This is on the same level as in all limiters.

**Conclusion:**
Retention in the coating cannot be neglected in the accountancy of fuel inventory in the entire tokamak.

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L. Dittrich et al., Phys. Scr. 96 (2021)
A. Widdowson et al., Phys. Scr. 96 (2021)
**Be-coated Inconel: Erosion of Inner Wall Cladding**

*Structure of initial and power-tested Be layers.*

<table>
<thead>
<tr>
<th>Initial</th>
<th>Test at 1.8 MW/m²</th>
<th>Test at 2.55 MW/m²</th>
</tr>
</thead>
</table>

After exposure in JET.

Note: rounded edges with bright rims and a network of small “dots” on Be platelates.

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**Inner Wall Cladding: Erosion pathway of Be coatings**

*Message:*

Layer erosion and disintegration by the formation of tube-like Be-O structures.

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L. Dittrich et al., Phys. Scr. 96 (2021)
Beryllium melting, splashing and dust

Dust Collection: Facts, Advantages, Drawbacks

Vacuum cleaning: 360°

Message:
Around 1 g found after each campaign (20 h of plasma)

Drawbacks:
Composition: a mix of all particles.
Not possible to associate dust morphology with place.

Local sampling: sticky tape

Advantage: Detailed local analyses.
Drawbacks:
The top layer of samples represents the bottom of deposits.
Force applied during collection; more than just dust may be collected.

Dust monitors

Message:
Tiny amount collected.

Advantages:
Collection of undisturbed mobilized particles.
Precise analyses.

Silicon plate

Message:
Tiny amount collected.
Be upper dump plate ILW-2: Melting and splashing

Note: the melt layer propagates upwards.

Diagnostic mirror: Au-coated steel

damaged area

intact

Be splashes from molten limiters

On the mirror at the top of JET.

Main chamber: mirror at the equatorial plane

Be splashes from molten limiters

On the mirror at the top of JET.

Main chamber: mirror at the equatorial plane

M. Rubel et al., Beryllium in JET with the ITER-like Wall

Droplets and bubbles? (boiling beryllium)
Be droplets and splashes from molten limiters

On dust monitors above the divertor

On the tungsten divertor plates: bottom of JET

**Messages:**

- Be droplets and splashes adhere well to surfaces → not decisive for loose dust.
- Splashes are mostly round and only very thinly coated by deposits.

Search for mobilized Be dust during in-vessel interventions
Dust on the remotely handled (RH) robotic arm

Exercise performed on the request from the ITER Safety Division.

- Aluminium is the main species: from the RH equipment.
- Other species: carbon, copper (NBI), steel/Inconel
- Only ONE Be-containing particle was found.

Main message: No massive Be mobilization.

Concluding Remarks: specific to JET

- Over 2 tons of beryllium PFC are installed in the main chamber of JET with the ITER-Like Wall.

- JET operation with metal walls has led to a significant decrease of fuel inventory and dust production in comparison to the operation with carbon walls.

- Beryllium splashes stick firmly to surfaces and do not form loose dust.

- Fuel retention in Be coatings is comparable to that in Be limiters. In both cases it is small: < 200 mg after three ILW campaigns (63 h operation).

- In-vessel operation with a robotic arm does not cause mobilisation of Be dust. The study was performed to respond to the request of the ITER Safety division.
Recent papers on Be

Data on erosion and hydrogen fuel retention in beryllium plasma-facing materials
G. De Temmerman, K. Heinola, D. Borodin, S. Brezinsek, R.P. Doerner, M. Rubel, E. Fortuna et al.,

Dust generation and accumulation in JET-ILW: morphology and stability of co-deposits on main plasma-facing components and wall probes.
E. Fortuna-Zalesña, T. Płociński, S.W. Moon, P. Petersson, M. Rubel, A. Widdowson
*Phys. Scr.* 96 (2021) 124038 [https://doi.org/10.1088/1402-4896/ac2979](https://doi.org/10.1088/1402-4896/ac2979)

Fuel retention and erosion-deposition on inner wall cladding tiles in JET-ILW
L. Dittrich, P. Petersson, M. Rubel, T.T. Tran, A. Widdowson, I. Jepu, et al.,
*Phys. Scr.* 96 (2021) 124071 [https://doi.org/10.1088/1402-4896/ac379e](https://doi.org/10.1088/1402-4896/ac379e)

Evaluation of tritium retention in plasma facing components during JET tritium operations
A. Widdowson, J.P. Coad, Y. Zayachuk, I. Jepu, E. Alves, N. Catarino, V. Corregidor et al.,
*Phys. Scr.* 96 (2021) 124075 [https://doi.org/10.1088/1402-4896/ac3b30](https://doi.org/10.1088/1402-4896/ac3b30)

An overview of tritium retention in dust particles from the JET-ILW divertor
T. Otsuka, S. Masuzaki, N. Ashikawa, Y. Torikai, Y. Hatano, M. Tokitani, Y. Oya et al.,
*Phys. Scr.* 97 (2022) 024008 [https://doi.org/10.1088/1402-4896/ac445b](https://doi.org/10.1088/1402-4896/ac445b)

Tools: Erosion-Deposition Diagnostics in JET-ILW

**Marker Tiles** and **Probes**

The aim is to have a complete overview of material migration and material damage, not just a number (even large) of analysis points and isolated findings.
ILW 2013-2014: Dust monitors

Monitor in the inner divertor

- no exposure due to inner frame of box
- exposure to impurities - probably Be deposition

visible dust particles - origin unknown

clean area - no exposure due to inner frame of box
deposited area - exposure to impurities - probably Be deposition

A. Widdowson, NOT published images

Be Limiters: Deposition inside Castellations

Aim: Determine surface composition inside the castellations.

Dump Plate (DP):
- O, N, decrease slightly with increasing depth.
- C at $0.5 \times 10^{17}$ cm$^{-2}$

Outer Poloidal Limiter (OPL):
- No D detected close to the surface.
- C at $1 \times 10^{17}$ cm$^{-2}$

M. Rubel et al., Beryllium in JET with the ITER-like Wall 2017 Nucl. Fusion 57 066027
Thermal desorption of tritium from beryllium plasma-facing components of the JET ITER-like wall

Aigars Vītiņš1, Elīna Pajuste1,2, Juris Jansons1, Gunta Kīzāne1 and JET Contributors*

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* See the author list of E. Joffrin et al., 2019 Nucl. Fusion, 59, 112021.

The JET ITER-like wall (ILW) has beryllium in the main plasma chamber and tungsten in the divertor as foreseen for the ITER vacuum vessel. Limiters and an upper dump plate of the JET vacuum vessel are bulk beryllium tiles. The goal of the ILW experimental campaigns is to characterize plasma-wall interactions and to compare plasma performance with the previous, carbon wall. One of the scientific objectives of the ILW experimental campaigns is to demonstrate sufficiently low fuel retention and, in particular, to demonstrate ITER-relevant tritium retention mitigation. The aim of the present study is to assess tritium accumulation in the beryllium tiles from the three ILW campaigns (2011-2012, 2013-2014, 2015-2016) and to investigate its thermal desorption patterns with respect to development of possible detritiation techniques. Tritium is a minor component of the fuel in the present study as tritium has not been introduced within the ILW campaigns 2011-2016. Tritium accumulation in plasma-facing tiles can occur as a result of its co-deposition with eroded material and implantation/diffusion in the bulk of the tiles.

Thermal desorption of tritium was performed in a He + 0.1% H₂ flow at 4.8 K/min from 290 to 1300 K. For inner wall samples, the surface concentration of tritium decreased in the three ILW campaigns from 83E11 (2011-2012) to 6E11 (2015-2016) atoms / cm². The temperature of 50% detritiation was 697 K and 880 K for these two samples respectively. For outer wall samples, the maximum value of the surface concentration of tritium 13E13 atoms / cm² was found for a centre sample of the 2015-2016 campaign, but the minimum value of that 0.52E13 atoms / cm² was for a right-hand wing sample of the 2013-2014 campaign. The temperature of 50% detritiation was 966 K and 840 K for these two samples respectively. For the 2011-2012 and 2013-2014 campaigns, both inner and outer wall samples from a tile middle part had higher temperatures of their 50% detritiation than those of the respective wing samples. The different values of the surface concentration of tritium and dissimilarity of tritium desorption patterns of the samples investigated indicate both quantitative and qualitative differences in tritium accumulation in the samples.

Acknowledgment: This work has been carried out within the framework of the EUROfusion Consortium, funded by the European Union via the Euratom Research and Training Programme (Grant Agreement No 101052200 — EUROfusion). Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the European Commission. Neither the European Union nor the European Commission can be held responsible for them.
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Thermal desorption of tritium from beryllium plasma-facing components of the JET ITER-like wall

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* See the author list of E. Joffrin et al., 2019 Nucl. Fusion, 59, 112021.

Outline

• Introduction
• Experimental procedure
• Results and discussion
• Conclusions
The JET ITER-like wall (ILW)

- Limiters and an upper dump plate of the JET vacuum vessel are castellated bulk beryllium tiles.
- One of the scientific objectives of the ILW experimental campaigns is to demonstrate sufficiently low fuel retention and, in particular, to demonstrate ITER-relevant tritium retention mitigation.
- The aim of the present study is to assess tritium accumulation in the beryllium tiles from the three ILW campaigns (2011-2012, 2013-2014, 2015-2016) and to investigate its thermal desorption patterns with respect to development of possible detritiation techniques.
Tiles investigated

Samples for this study were cut out of the following bulk beryllium tiles of the JET ITER-Like Wall (ILW) main chamber after the JET-ILW campaigns 2011-2012 (ILW1), 2013-2014 (ILW2) and 2015-2016 (ILW3):

- Inner Wall Guard Limiter (IWGL) 2XR10
- Outer Wide Poloidal Limiter (WPL) 4D14, 4D15
- Upper Dump Plate (DP) 2BC2

Tritium as a minor component of the fuel

- Tritium is a minor component of the fuel in the present study as tritium has not been introduced within the ILW campaigns 2011-2016 with deuterium plasma. Tritium has remained in the vacuum vessel since D-T experiments (the last of them was in 2003). Tritium can be produced in the D-D reactions and in the beryllium reactions with neutrons.

- Tritium accumulation in plasma-facing tiles can occur as a result of its co-deposition with eroded material and implantation/diffusion in the bulk of the tiles. Subsequently, tritium-containing material can be eroded and re-deposited.
Sample preparation

- 10 mm thick beryllium samples have been prepared by cutting individual castellations (about 12 x 12 mm) from the beryllium pieces that make up each of the tiles in the main chamber of JET.
- These samples were further cut into two parts: one for a dissolution experiment and another one for temperature-programmed desorption (TPD) of tritium.
- TPD of tritium was performed for single quarters of samples 547, 563 and 598 and for three quarters of sample 691 separately for each quarter.
Gas exchange time of the tritium TPD setup

- Quartz tube of 156 cm³: 72 cm³ - before the sample; 59 cm³ - after the sample; 25 cm³ – the zinc compartment.
- Column of 74 cm³ for silica gel.
- Cold trap of 66 cm³.
- Gas filter of 20 cm³.
- Volume of these items of the tritium TPD setup together is about 320 cm³.
- Purge gas He + 0.1% H₂ 14-15 L/h or about 250 cm³/min.
- The gas exchange time of the tritium TPD setup itself is about 1.3 min.

- Operating volume of tritium detector DDH 32 is 300 cm³.
- Purge gas He + 0.1% H₂ 14-15 L/h or about 250 cm³/min.
- Counting gas Ar + 10% CH₄ 42-45 L/h or about 750 cm³/min.
- The gas exchange time of the tritium detector itself is about 18 seconds.
- According to the data of the manufacturer of DDH 32 (“MAB”, Germany), there will be 62% of the fresh gas mixture in the detector after 18 s, but 94% will be after 54 s.

- Therefore, the released tritium activity was measured repeatedly with the measuring time of 2 min.
Temperature program and temperature measurements

- Samples were heated at a rate of 4.8 K/min to 1300 K and then kept at that temperature for 1 h.
- The temperatures of the sample, the zinc bed and the cold trap were continuously measured with an Agilent 34970A multichannel digital voltmeter with an Agilent 34902A multiplexer and recorded with a PC using the Agilent BenchLink Data Logger 3 software using type S and K thermocouples.

Rationale of the tritium TPD experiments

- As tritium was a minor component of the fuel in the JET-ILW operation, the beryllium samples to be investigated may have a low tritium activity and a low tritium release.
- A blank experiment with an unirradiated beryllium sample in a quartz boat was performed as the first experiment in the series in order to test the system background.
- The count rate was corrected for the system background for ≥ 1 h time as determined at the start and the end of each experiment.
- The tritium release rate of the sample was calculated from the corrected count rate and the measured flow rate of the purge gas. The calibration factor for tritium was found to be 107.8 (kBq / m³) / cps.
- The total released tritium activity was calculated by integrating the release rate over the time.
- The radioactivity of tritium released was calculated as kBq/cm² to 1 cm² of the plasma-facing surface area of the sample. The respective number of tritium atoms was found by dividing their activity [Bq] with $\lambda = 1.782E-9$ s⁻¹.
- Subsequent second heating of the same sample in the same setup with the same temperature program to 1300 K caused no significant tritium release.
- As most of tritium released during the temperature ramp of 4.8 K/min to 1300 K, the release rate and the sum release were plotted as functions of temperature.
- In order to take tritium natural decay into account, the number of tritium atoms has been recalculated to the respective JET shutdown date.
Outline

- Introduction
- Experimental procedure
- Results and discussion
- Conclusions

TPD of tritium from the part of ILW3 Be sample 469 (2015-2016: IWGL 2XR10: R5-C2 of RH wing) (1/2)

Plasma-facing surface (PFS): 0.81 cm²
Initial background: 0.6239 cps (78'-178')
Final background: 0.6133 cps (448'-598')
TPD of tritium from the part of ILW3 Be sample 469 (2015-2016: IWGL 2XR10: R5-C2 of RH wing) (2/2)

Plasma-facing surface (PFS): 0.81 cm²

Initial background: 0.6239 cps (78'-178')
Final background: 0.6133 cps (448'-598')

IWGL: Tritium fractional release rate at 4.8 K/min

BeWS-15, Karlsruhe, Germany, 14 September 2022
IWGL: Released fraction of the total tritium

IWGL 2XR10: Thermal desorption of tritium
Dump plate 2BC2, R4-C3: Thermal desorption of tritium at 4.8 K /min

TPD of tritium from quarters 1-3 of ILW1-3 Be sample 691 (2011-2016: WPL 4D15: R6-C2 of RH wing) (1/2)
TPD of tritium from quarters 1-3 of ILW1-3 Be sample 691 (2011-2016: WPL 4D15: R6-C2 of RH wing) (2/2)

WPL: Tritium fractional release rate at 4.8 K/min
A. Vitiš et al., Thermal Desorption of Tritium From Beryllium Plasma-Facing Components

WPL: Released fraction of the total tritium

WPL: Thermal desorption of tritium
Conclusions about IWGL 2XR10 samples

1. For the inner wall samples investigated in the present study, the surface concentration of tritium gradually decreased in the three ILW campaigns. The maximum of 83E11 atoms / cm² was found for the LH Wing sample of ILW1 (2011-2012), and the minimum of 6E11 atoms / cm² was for the RH Wing sample of ILW3 (2015-2016). The temperature of 50% detritiation was 697 K and 880 K for these two samples respectively.

2. The samples had quite different thermal desorption spectra of tritium indicating different tritium accumulation in the samples. The ILW2 (2013-2014) samples had by 17-43 K higher temperature of 50% detritiation than the respective ILW1 (2011-2012) samples. For ILW1 and ILW2 samples, the temperature of 50% detritiation gradually decreased from the centre towards the wings by 24-88 K. For all the samples, the temperature of 50% detritiation was in the range of 697-880 K.
Conclusions about Upper Dump Plate 2BC2 samples

1. The ILW2 (2013-2014) sample had by a factor of >3 lower surface concentration of tritium than that of the respective ILW1 (2011-2012) sample: 1.5E12 and 5.1E12 atoms / cm².
2. The tritium release curves had a single main maximum, but the temperature of 50% detritiation of the ILW2 (2013-2014) sample, 796 K, was by 49 K higher than that of the ILW1 (2011-2012) sample, 747 K.

Conclusions about WPL samples

1. The surface concentration of tritium of the 4D14 samples depends strongly both on the ILW campaign and on their toroidal position. The highest value of 130E12 atoms / cm² was found for the Centre sample of ILW3 (2015-2016), and the lowest value of 5E12 atoms / cm² was for the RH Wing sample of ILW2 (2013-2014). The temperature of 50% detritiation was 966 K and 840 K for these two samples respectively.
2. The 4D14 samples had different thermal desorption spectra of tritium indicating different tritium accumulation in the samples. For all the 4D14 samples investigated, the temperature of 50% detritiation was in the range of 754-1003 K. At 1181 K, all the 4D14 samples investigated had ≥90% detritiation.
3. The three quarters of castellation R6-C2 of RH Wing of tile 4D15 from the ILW1-3 (2011-2016) campaigns had following values the total tritium: 8.7E12, 13.2E12 and 11.0 E12 atoms / cm². Their temperatures of 50% detritiation were 889-894 K. But their temperatures of 90% detritiation were in a broader range of 1009-1078 K.
Acknowledgements

• This work has been carried out within the framework of the EUROfusion Consortium, funded by the European Union via the Euratom Research and Training Programme (Grant Agreement No 101052200 — EUROfusion).

• Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the European Commission. Neither the European Union nor the European Commission can be held responsible for them.
A Study on Technician Variability in Wipe Sampling for Beryllium & Potential Contributions to Robotic Sampling Equipment

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The Joint European Torus (JET) is the largest operational magnetically confined fusion research reactor in the world, beginning operations in 1983. Beryllium (Be) was first installed in JET in 1989, which gives the UKAEA a high level of experience and expertise in the safe handling, storage, and disposal of this high-performance but toxic material.

The UKAEA’s Health Physics Group (HPG) is comprised of a team of trained technicians and analysts who support an on-site Be handling facility and operate a Be analysis laboratory, in which accredited processes are certified to UKAS ISO 17025. The laboratory capabilities enable the UKAEA to carry out technical studies involving beryllium in a controlled, safe environment by using established air and surface sampling techniques to monitor conditions over a study’s specified experiments and any related activities.

To date, surface sampling for beryllium, known on the Culham site as “smearing”, has been performed by HP technicians trained in defined, manual processes. Others’ studies in recent years have looked at collection efficiencies between the sampling media being used and the type of beryllium compound being sampled. Given the manual nature of the task, natural variations occur from person to person, and building on the aforementioned research, UKAEA has decided to investigate the impact of force exerted by a technician during the sampling process.

This presentation gives an overview on the history of beryllium use at JET, surface sampling processes used on site, a summary of the pilot study in progress, and how these manual studies are contributing to further research into robotic sampling equipment in challenging environments.

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Presentation Plan

- **Part 1: Introduction / Background**
  - Presenter: Chris Dorn
  - Overview of the Joint European Torus (JET)
  - History of Beryllium Use in JET
  - Sampling Programme at UKAEA

- **Part 2: Technician Variability Study**
  - Presenter: Eilish McKeon
  - Assumptions, Impetus & Prior Work
  - Purpose & Scope
  - Project Outline & Milestones

- **Part 3: Robotic Surface Wiping**
  - Presenters: Maddy Kearney & Jon Verdon
  - Background: Robotics & AI in Nuclear (RAIN)
  - Glovebox Wiping
    - Tool Design & Trials
    - Future Work
Part 1: Introduction / Background
Presenter: CHRIS DORN

Overview of the Joint European Torus (JET)

- **1978**: JET construction starts
- **1983**: First plasma in JET
- **1989**: First installation of beryllium in JET
- **1991**: First experiments with tritium
- **1997**: High-performance, full deuterium-tritium experiments. JET achieves world record fusion power of 16.1MW.
- **2009-10**: JET installs ITER-Like Wall: beryllium & tungsten
- **2019-20**: Preparations well advanced for new deuterium-tritium experiments, designed to sustain high fusion performance for longer periods.

[1] E. McKeon et al., A Study on Technician Variability in Wipe Sampling for Beryllium
History of Beryllium Use in JET

- More than just laboratory-scale use of beryllium in fusion research began at JET in 1989
- UKAEA understood the need to monitor for airborne beryllium to comply with applicable UK standards
- Based on experience in radiation protection, UKAEA also foresaw the need to monitor for beryllium surface particulate
- As a result, UKAEA developed a surface sampling method for beryllium, in conjunction with Sandia National Lab (USA)
  - Dry wiping using Whatman Grade 1 filter papers
  - Analysis of collected samples done on site
- Surface sampling is just one part of the overall UKAEA Beryllium Management Programme

Sampling Programme at UKAEA

- Unlike for airborne beryllium, there are no national or international standards for beryllium surface contamination
- UKAEA created its own internal standards and action levels for beryllium surface contamination
  - Transfer of items from one designated area to another
  - Clearance of items from designated areas entirely
  - Action levels: trigger the creation of designated areas
- UKAEA has used the same beryllium surface sampling method to support JET operations, but we have also developed other processes for external customers
Part 2: Technician Variability Study

Presenter: EILISH McKEON

Background: Assumptions, Impetus & Prior Work

- **Original Baseline Assumption**
  - Cleaning effect: each successive sample for removable beryllium taken from the same area should give a progressively lower result
  - Does not take into account samples collected from solid Be surfaces

- **Impetus**
  - Long-term study with surface sampling as integral part
  - Due to staff changes over the duration of the study, several technicians were used to collect surface samples
  - Results did not always consistently follow expectations

- **Prior Work**
  - Damjanovic, 2019: Comparison of Collection Efficiencies of Whatman No. 1 Filter Paper and Ghost Wipes for Loose Beryllium Surface Contamination (presented at BeWS-14) [6]
  - Glass plates spiked with beryllium-containing solution in known amounts were used in the previous study [6]
Purpose & Scope

- **Purpose of the Study**
  - Determine an uncertainty factor that can be applied to surface sampling results for removable beryllium that incorporates the effects of pressure exerted during the sampling process.

- **Scope of Work**
  - Procuring pressure-measuring equipment
  - Manufacturing a pressure rig (test fixture)
  - Spiking a defined surface with known concentrations of beryllium
  - Measuring pressure ranges exerted by a number of individual technicians
  - Measuring the amount of beryllium detected
  - Determining the results are correlated and if so, determining an uncertainty factor which can be applied to analytical results incorporating the uncertainty from the sampling and analytical processes. (Analytical uncertainty is already accounted for.)

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Project Outline & Milestones

Deliverables have been defined for the project as follows:

- **Preparatory Stage**
  - Experiment Design
  - Intellectual Property Investigation
  - Procurement & Manufacture of Components
  - Data Protection Requirements & Permissions

- **Experimental Stage**
  - Gathering Data
  - Analysing Data

- **Reporting Results**
  - Internal Report Write-Up
  - Authoring a Scientific Paper
  - Peer Review & Publication

- **Future Work**
  - Consider Investigation of Other Variables
Human-Robot Interaction
- The HRI working group is focused on the interactions with robotics and autonomous systems such as trustworthiness, perception and communication as well as explorations of the HRI systems including virtual and augmented reality, teleoperation, haptics, shared control and testing methods.

Remote Handling
- The Remote Handling Working Group is developing technology to take hands out of gloveboxes, making nuclear decommissioning safer, faster and cheaper.

Remote Inspection
- The Remote Inspection Working Group (RIWG) focuses on developing robotic and AI technology for both the characterisation of unknown nuclear environments and change or anomaly detection of previously characterised environments.

Standardisation
- The Standardisation working group focused on bringing academic and industrial experts together to create new baselines in specific areas such as operator-facing human-machine interfaces (HMI), extensible modular software systems, and nuclear telemanipulation systems.

Verification and Autonomy
- The V&A working group are focused on engagement with regulators and work that can be achieved through simulation. Including the assurance of autonomous Systems for safe use in hazardous environments.
Glovebox Wiping Tool Design & Trials (1)

1. Final prototype wiping tool with robotic interface

2. Remote Handling Operator using teleoperation to operate the robot

Glovebox Wiping Tool Design & Trials (2)

3. Robot demonstrating that it can wipe all surfaces

4. Comparison of forces from a robotic wipe and trained professionals’ wipe.
**Future Work (1)**

- **Redesigning the current hook and loop attachment system:** need to overcome the following issues:
  - Creation of high spots
  - Trapping of contaminants by high surfaces
  - Inability to work with wet media
- Plan to design a quick-release system that will not require the wipe (i.e., the media) to be modified

**Future Work (2)**

- **Design full life cycle of operation:** involves robotically cleaning an area, swabbing, analysing, and reading results, all within a glovebox using robotics.
  - Picking-up tool
  - Wiping surfaces
  - Removal of wipes
  - Safe disposal of wipes with no human contact
Future Work (3)

- Using Health Physics results to automate wiping.
  - Data taken from observing and recording experienced technicians
  - With automation, all parameters can be controlled.
    - Force
    - Speed
    - Wiping pattern

- Comparing humans, automation, and teleoperation (future paper).
  - Speed
  - Pick-up
  - Forces

References & Credits

SESSION 4

Beryllides
Overview of activities in Kazakhstan related to study of beryllium and beryllium compounds

A. Shaimerdenov¹,², T. Kulsartov²,³, I. Kenzhina¹,²,⁴*, Zh. Zaurbekova²,³, Y. Kenzhin⁴, Y.V. Chikhray², S. Udartsev⁵

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³Institute of Atomic Energy of the National Nuclear Center of the Republic of Kazakhstan, Kurchatov, Kazakhstan
⁴Kazakh-British Technical University, Almaty, Kazakhstan
⁵Ulba Metallurgical Plant, Ust-Kamenogorsk, Kazakhstan

Kazakhstan is one of the world leaders in the production of beryllium and beryllium products. Since recently, the Ulba Metallurgical Plant has set up production of beryllium-based intermetallic compounds; in particular, beryllides of titanium, chromium, molybdenum, etc. are produced. Beryllides are candidate materials for fusion plants as a neutron multiplier. In addition, beryllides are considered as a material for hydrogen storage. On the basis of research reactors in Kazakhstan, studies are being conducted on the effects of neutron irradiation on the properties of beryllium and beryllides. This paper provides an overview of research programs in Kazakhstan to study the radiation resistance of metallic beryllium of different grades and beryllides.

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Overview of activities in Kazakhstan related to study of beryllium and beryllium compounds

A. Shaimerdenov¹², T. Kulsartov²³, I. Kenzhina¹²⁴, Z. Zaurebekova²³, Y. Kenzhin⁴, Y. V. Chikhray², S. Udartsev⁵

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³ - Institute of Atomic Energy of the National Nuclear Center of the Republic of Kazakhstan, Kurchatov, Kazakhstan
⁴ - Kazakh-British Technical University, Almaty, Kazakhstan
⁵ - Ulba Metallurgical Plant, Ust-Kamenogorsk, Kazakhstan

Disclaimer:
This presentation summarizes the results of a study of the effect of neutron irradiation on the properties of beryllium and plans for reactor studies of titanium beryllides.
**IGR RESEARCH REACTOR**

- Type: pulse
- Thermal power: 1 GW
- Moderator: graphite
- Reflector: graphite
- Coolant: graphite/helium
- Pressure: atmospheric
- Type of coolant: natural convection
- Core size: 1400 mm
- Core height: 1463 mm
- Fuel: UO$_2$(NO$_3$)$_2$+C (HEU)
- Maximum of thermal neutron flux: $7 \times 10^{16} \text{ cm}^{-2}\text{s}^{-1}$

**IVG1.M RESEARCH REACTOR**

- Type: loop
- Thermal power: 6 MW
- Moderator: light water
- Reflector: beryllium
- Coolant: light water
- Pressure: atmospheric
- Type of coolant: forced
- Core diameter: 548 mm
- Core height: 800 mm
- Fuel: UZr (LEU)
- Maximum of thermal neutron flux: $1.5 \times 10^{14} \text{ cm}^{-2}\text{s}^{-1}$
**WWR-K RESEARCH REACTOR**

- **Type:** tank
- **Thermal power:** 6 MW
- **Moderator:** demineralized water
- **Reflector:** demineralized water and beryllium
- **Coolant:** demineralized water
- **Pressure:** atmospheric
- **Type of coolant:** forced
- **Coolant circuit:** two
- **Core diameter:** 720 mm
- **Core height:** 600 mm
- **Fuel:** UO₂+Al matrix (LEU)
- **Maximum of thermal/fast neutron flux:** $2 \times 10^{14}/8 \times 10^{13} \text{ cm}^{-2} \text{s}^{-1}$

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**EXPERIMENTAL POSSIBILITIES OF WWR-K**

**Two kind of hot cells:**
- Concrete shielding (5 cells)
- Steel shielding (4 cells)

**Critical assembly**

- Zero power reactor
- Maximum thermal power: 100 W
- Reflector: deionized water and/or beryllium
- Moderator: deionized water
- Fuel composition: UO₂+Al
- Enrichment in U-235: 19.7 % (since 2012)
- Max. thermal neutron flux density: $10^9 \text{ cm}^{-2} \text{s}^{-1}$
- Diameters of experimental channels: 65, 96 and 140 mm.

**Hydraulic transfer system**

Utilized to load and unload of irradiation ampoule to/from the core during reactor operation

**Pneumatic transfer system**

Installed at beam tube #3, allows transfer of ampoule with a sample from laboratory room to the core and return back for gamma spectrometry measurement (neutron activation analysis)

**Gas-vacuum loop facility**

Gas-vacuum Loop Facility is designed to create a vacuum or gas environment in a sample during in-pile test
VIKA experimental facility

Schematic circuit

PT – thermocouple vacuum gage;
PA – ionizing vacuum gage;
NS – magnetic-discharge pump NORD – 100;
NS2 – magnetic-discharge pump NORD – 250;
S1 – omegatron RMO – 13;
S2 – gage of quadrupole analyzer for remaining gases (RGA-100);
NV – forepump NVR – 5 DM;
BL – liquid nitrogen trap;
CV – vacuum chamber;
V – vacuum tap;

External view

Technical parameters

➢ Range of temperatures  
30 ÷ 1500 °C;
➢ Pressure in vacuum chamber at crucible temperature 1500 °C,  
10⁻⁴ Pa;
➢ An accuracy of temperature auto control ± 0,5 °C;
➢ A range of heating rate  
2 ÷ 50 °C/min.

Complex for in-reactor gas release analysis (CIRRA)

Block scheme

High-vacuum station (Agilent Technologies)
Pressure gauge
Record and archive system
Mass spectrometer (Stanford Research System)

Temperature control method: by changing gas pressure in capsule gap and using electrical heater;
Heating method: radiation heating and electrical heater;
Features: the experimental facility can be connected to the experimental devices installed in any cell of the reactor core; irradiation parameters (temperature, pressure, the neutron flux relative density, etc.) are recorded and archived.

Irradiation temperature: 50-1500 °C;
Measurement of elements mass: up to 100 a.e.m.;
Measurement method: determination of a separating fluid at the condition of vacuum extraction under uninterrupted exhaustion;
Study of DV-56 and TShG-200 beryllium grade

**DV-56 grade irradiated beryllium.**
Irradiation site – BN-350 reactor (Aktau, RK)
Fluence - $5 \cdot 10^{21}$ n/sm² (E > 1 MeV)
Year of unloading – 1996

<table>
<thead>
<tr>
<th>Chemical composition</th>
<th>Be</th>
<th>Si</th>
<th>Fe</th>
<th>Al</th>
<th>C</th>
<th>BeO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass fraction impurities, %</td>
<td>98.64</td>
<td>0.013</td>
<td>0.055</td>
<td>0.02</td>
<td>0.072</td>
<td>1.2</td>
</tr>
</tbody>
</table>

**TShG-200 grade irradiated beryllium.**
Irradiation site – IVG reactor (Kurchatov, RK)
Fluence ~ $10^{19}$ n/sm² (E < 0.1 MeV)
Year of unloading – 2010

<table>
<thead>
<tr>
<th>Chemical composition</th>
<th>Be</th>
<th>Fe</th>
<th>Al</th>
<th>Si</th>
<th>Cr</th>
<th>F</th>
<th>O</th>
<th>C</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass fraction impurities, %</td>
<td>97.8</td>
<td>0.25</td>
<td>0.03</td>
<td>0.04</td>
<td>0.05</td>
<td>0.002</td>
<td>1.3</td>
<td>0.12</td>
<td>0.04</td>
</tr>
</tbody>
</table>

Samples for TDS investigations:

<table>
<thead>
<tr>
<th>Sample shapes</th>
<th>Half-disc</th>
<th>Disk</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average size, mm</td>
<td>4.5</td>
<td>9-10</td>
</tr>
<tr>
<td>Width, mm</td>
<td>1-1.5</td>
<td>1-3</td>
</tr>
</tbody>
</table>

Experimental results:

Time dependences of sample temperature and partial gas pressure changes

**DV-56 grade**

Gas release from irradiated beryllium of DV-56 grade
Heating rate 10 °C/min, sample width – 1 mm

**TShG-200 grade**

Gas release from irradiated beryllium of TShG-200 grade
Heating rate 10 °C/min, sample width – 1.3 mm
Results of microstructural investigations

Results of microstructural investigation of DV-56 beryllium grade after TDS-experiments

![Surface of beryllium sample](image)

![Surface of the sample's cross section](image)

![Scaled up view of pore in a sample](image)

Main parameters of tritium interaction with investigated samples

<table>
<thead>
<tr>
<th>Parameter</th>
<th>DV-56 grade</th>
<th>TShG-200 grade</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diffusion coefficient $D$, m²/s</td>
<td>$(9,0\pm0,2)*10^{-2}$</td>
<td>$150\pm5$</td>
</tr>
<tr>
<td>Activation energy of diffusion $E_D$, kJ/mole</td>
<td>$(3,4\pm0,4)*10^{-4}$</td>
<td>$90\pm5$</td>
</tr>
</tbody>
</table>

Analysis of experimental results

A histogram of relative gas release from two-types samples during the different temperatures ranges of TDS spectrum

**High temperatures**

- DV-56 beryllium grade
- TShG-200 beryllium grade

**Low temperatures**

- DV-56 beryllium grade
- TShG-200 beryllium grade

Relative contribution of integral tritium-containing release to total tritium release from investigated samples
The beryllium grades were the following:
- traditional grade of beryllium, S-200F, which is produced by the vacuum hot pressing (VHP);
- the grade S-65H, which is produced by hot isostatic pressing (HIP); it is characterized by a higher degree of purity;
- the grade I-220H, which is also produced by the HIP technique but is characterized by higher mechanical strength and better extent of isotropy.

### Scope of study:
- measurement of dimensions and weight;
- hardness test;
- SEM observation;
- TDS experiments for measurements of Helium/Tritium contents.

### Irradiation conditions
- Samples were irradiated in the WWR-K reactor core;
- Two neutron fluence were reached: \( \approx 1.5 \times 10^{24} \) and \( \approx 4 \times 10^{24} \) \text{n/m}^2 (\( E_n > 1 \text{MeV} \));
- Iron fluence monitor was used for measurement of accumulated neutron fluence;
- Temperature of irradiation: 50ºC;
- Samples environment: helium;

<table>
<thead>
<tr>
<th>Grade</th>
<th>Shape</th>
<th>Dimension (mm)</th>
<th>Mass (mg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-200F</td>
<td>Disk</td>
<td>⊙10×1.5</td>
<td>217.96</td>
</tr>
<tr>
<td></td>
<td></td>
<td>⊙9.98×1.51</td>
<td>218.3</td>
</tr>
<tr>
<td>S-65H</td>
<td>Disk</td>
<td>⊙10×1.51</td>
<td>219.47</td>
</tr>
<tr>
<td></td>
<td></td>
<td>⊙10.0×1.51</td>
<td>219.04</td>
</tr>
<tr>
<td>I-220H</td>
<td>Disk</td>
<td>⊙10.01×1.51</td>
<td>219.04</td>
</tr>
</tbody>
</table>
**Microstructure study**

On all samples there is pitting corrosion over the entire surface of the sample.

The most pronounced corrosion pitting are observed on a sample of S200F beryllium; less pronounced on the sample of beryllium grade S65H; and the least pronounced on the I220H beryllium grade sample.

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**Gas release results (1)**

Sample heating is carried put in two stages.

- First stage: a sample is heated, by linear law, at a specified rate, to the temperature level close to the sample melting point ($T \approx 0.98T_{mel}$).

- Second stage: the sample heating rate is decreased, and the sample is heated to the beryllium melting point ($T \approx 1283°C$).

Variations in partial pressures of gases was studied (helium $^3$He (a.m.u. 3), $^4$He (a.m.u. 4), and tritium (a.m.u. 6)) in the continuously exhausted chamber of the experimental facility are recorded.
Gas release results (2)

Release of tritium and helium for samples S-200F, I-220H - the main amount of gas (both helium and tritium) is released close to the melting temperature and during melting (while in previous experiments with samples irradiated to a lower fluence, noticeable amounts of released tritium were observed in the temperature range of 900-1150 °C). Beryllium sample of grade S-65H, a noticeable amount of gas is released up to the melting point (with a peak for tritium in the region of about 1100 °C). (A similar picture was observed with samples irradiated to a lower fluence).

<table>
<thead>
<tr>
<th>Beryllium grade</th>
<th>Tritium mole T&lt;sub&gt;2&lt;/sub&gt; ppm</th>
<th>Helium mole He ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-65H</td>
<td>4.55·10⁻⁹</td>
<td>1.5</td>
</tr>
<tr>
<td>S-200F</td>
<td>4.45·10⁻⁹</td>
<td>1.5</td>
</tr>
<tr>
<td>I-220H</td>
<td>4.23·10⁻⁹</td>
<td>1.4</td>
</tr>
</tbody>
</table>

Temperature dependencies of tritium flux from beryllium

Plans for study of radiation resistance of titanium beryllide (1)

In this regard, Kazakhstan has great opportunities, since on its territory Ulba Metallurgical Plant (UMP) is located (a unique enterprise engaged in the production of beryllium, uranium and tantalum products for the needs of nuclear energy). UMP employees have developed a technological method for obtaining titanium beryllide in large quantities by vacuum hot pressing→ Mr.Udartsev was reported.

Titanium beryllide is a candidate material for a neutron breeder of a fusion reactor blanket. This is due to its improved mechanical properties compared to metallic beryllium.

Project named as “Influence of reactor irradiation on physical and mechanical properties and gas generation in titanium beryllide” (grant #AP14871445) has been accepted by Kazakhstan government for funding
Scope of study:
- Neutron irradiation by accumulation of two neutron fluence ($5 \times 10^{20}$ cm$^{-2}$ and $\sim 10^{21}$ cm$^{-2}$);
- Samples density (unirradiated and irradiated);
- Hardness (unirradiated and irradiated);
- Microstructure (unirradiated and irradiated);
- Magnetic permeability (unirradiated and irradiated);
- Phase structure (unirradiated);
- TDS experiments for gas release measurements (irradiated);

Samples dimension: 8x8x1.5 mm (LxWxT)

Project will started since October 2022

Summary

There are three research reactors in Kazakhstan where material science research is being carried out.

The research centers of Kazakhstan employ highly qualified specialists who have many years of experience in researching materials for fusion.

Two reactors have facilities for TDS experiments (in-situ and PIE). The availability of such an infrastructure makes it possible to study materials in high level, in particular, beryllium and beryllides.

In Kazakhstan, there is a unique UMP plant that manufactures products from beryllium and can manufactured beryllides. With this in mind, studies of beryllides manufactured at the UMP begin in Kazakhstan.

Taking into account the above, we invite you to cooperation.
Beryllides as advanced materials for neutron multiplication

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The neutron multiplier is an essential component of the blanket of future thermonuclear reactors, which should provide the tritium breeder with a sufficient amount of neutrons of a certain energy. Among all chemical elements, only beryllium and lead have an advantageous ratio of high neutron multiplication reaction at low neutron absorption rates. However, pure metals – beryllium and lead – for various reasons cannot be used in the harsh operating conditions of a fusion reactor blanket. Intermetallic compounds of beryllium – beryllides have a number of advantages over pure beryllium and are currently considered to be the reference neutron multiplication material for the Helium Cooled Pebble Bed (HCPB) breeding blanket concept of EU DEMO fusion reactor. Recently, a batch of full-size beryllide blocks has been manufactured on an industrial scale in cooperation with the Ulba Metallurgical Plant. The present work is devoted to the characterization and analysis of these beryllide blocks so that the material could be used for the manufacture of a blanket.

Titanium beryllide (TiBe$_{12}$) blocks are hexagonal prisms with an internal hole, while chromium beryllide (CrBe$_{12}$) blocks are solid prisms of complex shape. The resulting blocks have a single-phase structure of the corresponding beryllide with a small impurity in the form of beryllium oxide. One of the titanium beryllide blocks also has about 7% residual beryllium phase. Grains of titanium beryllide have an average size of about 7–8 µm, while grains of chromium beryllide are much larger and reach 40–50 µm. Mechanical compression and bending tests of beryllides showed their very high strength, which is maintained up to 1000°C. In terms of specific compressive strength, the single-phase TiBe$_{12}$ surpasses all materials, except diamond, in the 700–1000°C temperature range. Chromium beryllide and titanium beryllide with 7% of the beryllium phase have lower strength, but higher ductility. Corrosion tests were carried out in air and in He + 2% water vapor at 800–1200°C. Beryllides have high corrosion resistance similar to Ni-base superalloys and high temperature ceramics. Long-term thermal cycling tests with rapid heating and cooling, simulating operation in a fusion reactor, showed high resistance of beryllides to thermal shocks. The results obtained are also discussed from the point of view of the application of beryllides in other areas.

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Neutron multiplier materials for breeding blanket

Demonstration power plant: DEMO

Deuterium-Tritium tokamak

Fusion power 2 GW = 111 kg of tritium/year
Neutron multiplier materials for breeding blanket

Demonstration power plant: DEMO

Deuterium-Tritium tokamak

Fusion power 2 GW = 111 kg of tritium/year

DEMO must breed required tritium
Selection of neutron multiplier material

Relative reaction rates

NMM should have:
- large \((n,2n)\) cross-section
- low parasitic neutron absorption

Threshold energy
\((n,2n)\) threshold energy for Be is much lower than for Pb
Beryllium as NMM

**ADVANTAGES:**

+ The best neutron multiplier, dual function as multiplier and fair moderator
+ The most compact T-breeding blanket
+ Allows working with relatively low Li6 enrichment (≈60%)
+ Avoids problems of liquid metal blankets (corrosion, embrittlement, bubbles, magnetohydrodynamics)
+ Can operate above 400°C, thus avoiding irradiation embrittlement of steels

**SHORTCOMINGS:**

− High chemical reactivity with steam and air
− High swelling under neutron irradiation above 650°C
− High tritium retention

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Solution - Beryllides

- Beryllium forms intermetallic compounds (beryllides) with almost all metals
- Typical compositions: $\text{MBe}_{12}$, $\text{MBe}_{22}$, $\text{MBe}_{13}$, $\text{M}_2\text{Be}_{17}$
- High corrosion resistance
- Low swelling and low tritium retention
Selection of beryllides compositions

- Neutron performance – high Be content
- High melting point
- High corrosion resistance
- Reasonable strength
- Low radioactivation

![Graph showing TBR and melting point of beryllides]

**Titanium and chromium beryllides**

**TiBe$_{12}$**
- Be-7.7at.%Ti or Be-30.8wt.%Ti
- $T_m=1550^\circ$C
- $\rho=2.28$ g/cm$^3$
- High corrosion resistance
- Reasonable thermal conductivity

**CrBe$_{12}$**
- Be-7.7at.%Ti or Be-32.5wt.%Ti
- $T_m=1340^\circ$C
- $\rho=2.44$ g/cm$^3$
- Simple phase diagram, no peritectic reaction

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**Limits on the use of elements**

![Diagram showing limits on the use of elements]

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Helium-cooled pebble bed blanket design

- DEMO sector
- Ø144mm × 150mm
- Ø85mm × 80mm
- Li_4SiO_4
- Li_2TiO_3
- Beryllide blocks
- Li_2SiO_4
- Li_2TiO_3

Beryllides manufactured by VHP

- TiBe_{12}
- CrBe_{12}

- Hexagonal block Ø144mm × 150mm
- Complex-shape block Ø85mm × 80mm
Requirements for beryllides as neutron multiplier materials

- High tritium breeding ratio – maximum possible content of Be
- Minimum content of impurities that form long-lived isotopes under irradiation (e.g. Uranium)
- Fine grain structure for easy tritium release and reasonable mechanical properties
- Beryllide blocks must retain their shape and not fracture during operation
- Low corrosion in the air and purge gas atmosphere
- Low interaction with structural materials (e.g. EUROFER steel)
- No fracture or cracks during rapid heating/cooling due to pulsed operation of DEMO

TiBe\textsubscript{12} manufactured by VHP. XRD
Requirements for beryllides as neutron multiplier materials

- High tritium breeding ratio – maximum possible content of Be
- Minimum content of impurities that form long-lived isotopes under irradiation (e.g. Uranium) – as low as possible activation
- Fine grain structure for easy tritium release and reasonable mechanical properties
- Beryllide blocks must retain their shape and not fracture during operation
- Low corrosion in the air and purge gas atmosphere
- Low interaction with structural materials (e.g. EUROFER steel)
- No fracture or cracks during rapid heating/cooling due to pulsed operation of DEMO

Chemical composition

<table>
<thead>
<tr>
<th>Material</th>
<th>Ti</th>
<th>Cr</th>
<th>C</th>
<th>N</th>
<th>O</th>
<th>Mg</th>
<th>Al</th>
<th>Si</th>
<th>Ca</th>
<th>Fe</th>
<th>U, ppm</th>
<th>Be</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiBe₁₂ UMP</td>
<td>29.6</td>
<td>-</td>
<td>0.038</td>
<td>0.0801</td>
<td>0.597</td>
<td>&lt;0.0002</td>
<td>0.0222</td>
<td>0.0213</td>
<td>0.01</td>
<td>0.12</td>
<td>4</td>
<td>0.51</td>
</tr>
<tr>
<td>TiBe₁₂ + 7vol.% Be UMP</td>
<td>27.8</td>
<td>-</td>
<td>0.034</td>
<td>0.106</td>
<td>0.686</td>
<td>&lt;0.00005</td>
<td>0.020</td>
<td>0.0162</td>
<td>0.015</td>
<td>0.11</td>
<td>8</td>
<td>0.395</td>
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<td>CrBe₁₂ UMP</td>
<td>-</td>
<td>30.8</td>
<td>0.0346</td>
<td>0.0177</td>
<td>0.555</td>
<td>&lt;0.0002</td>
<td>0.0156</td>
<td>0.0237</td>
<td>0.0088</td>
<td>0.11</td>
<td>4</td>
<td>0.54</td>
</tr>
<tr>
<td>TiBe₁₂ HIP</td>
<td>29.11</td>
<td>-</td>
<td>0.0774</td>
<td>0.0028</td>
<td>0.219</td>
<td>0.0355</td>
<td>0.037</td>
<td>0.0215</td>
<td>0.0018</td>
<td>0.10</td>
<td>2</td>
<td>19.3</td>
</tr>
</tbody>
</table>

Almost no uranium in beryllides from UMP
Requirements for beryllides as neutron multiplier materials

- High tritium breeding ratio – maximum possible content of Be
- Minimum content of impurities that form long-lived isotopes under irradiation (e.g. Uranium)
- Fine grain structure for facilitated tritium release and reasonable mechanical properties
  - Beryllide blocks must retain their shape and not fracture during operation
  - Low corrosion in the air and purge gas atmosphere
  - Low interaction with structural materials (e.g. EUROFER steel)
  - No fracture or cracks during rapid heating/cooling due to pulsed operation of DEMO

Microstructure of TiBe$_{12}$

Single phase TiBe$_{12}$
- Density $\rho=98.6\%$
- Mean grain size $D = 7\pm3$ $\mu$m

TiBe$_{12}$ + 7 vol.% Be
- 7 vol.% of Be phase (black)
- Density $\rho=98.3\%$
- Mean grain size $D = 7\pm3$ $\mu$m
Microstructure of Ti and Cr beryllides

TiBe$_{12}$

- Single phase
- TiBe$_{12}$
- $\rho=98.6\%$
- $D = 8\pm3.5 \, \mu m$
- EBSD

CrBe$_{12}$

- Single phase
- CrBe$_{12}$
- $\rho=98.5\%$
- $D = 42\pm18 \, \mu m$
- SEM

Requirements for beryllides as neutron multiplier materials

- High tritium breeding ratio – maximum possible content of Be
- Minimum content of impurities that form long-lived isotopes under irradiation (e.g. Uranium)
- Fine grain structure for easy tritium release and reasonable mechanical properties
- Beryllide blocks must retain their shape and not fracture during operation.
- Low corrosion in the air and purge gas atmosphere
- Low interaction with structural materials (e.g. EUROFER steel)
- No fracture or cracks during rapid heating/cooling due to pulsed operation of DEMO
Compression tests

Specimens from Ø2.2mm×2.6mm to Ø4mm×6mm

Cracked Si₃N₄ platens

Compressive properties of TiBe₁₂

TiBe₁₂

TiBe₁₂ +7%Be
Compressive properties of Ti and Cr beryllides

![Compressive stress vs. compression percentage for TiBe$_{12}$ and CrBe$_{12}$](image)

Beryllides can be plastically deformed above 1200°C

Specimens after compression at 1200°C, $\varepsilon \approx 20\%$

![Specimens of TiBe$_{12}$, TiBe$_{12}+7\%$Be, and CrBe$_{12}$](image)

Beryllides can be plastically deformed above 1200°C
Compressive strength of beryllides

3 point bending tests
3 point bending tests

TiBe$_{12}$+7%Be

Walsh, Beryllium Chemistry and Processing, 2009
Morrell, Handbook of Properties of Technical & Engineering Ceramics, 1985
Requirements for beryllides as neutron multiplier materials

- High tritium breeding ratio – maximum possible content of Be
- Minimum content of impurities that form long-lived isotopes under irradiation (e.g. Uranium)
- Fine grain structure for easy tritium release and reasonable mechanical properties
- Beryllide blocks must retain their shape and not fracture during operation
- Low corrosion in the air and purge gas atmosphere
  - Low interaction with structural materials (e.g. EUROFER steel)
  - No fracture or cracks during rapid heating/cooling due to pulsed operation of DEMO

Corrosion tests in air and He + 2% H₂O
**Hot corrosion of TiBe$_{12}$ +7%Be in air**

![Graph showing mass loss with temperature](image1)

- Beryllium
- 1200°C
- 1000°C
- 800°C

**Hot corrosion in He + 2% H$_2$O**

![Graph showing mass loss with temperature](image2)

- TiBe$_{12}$ + 7vol.% Be
- CrBe$_{12}$
- 1200°C
- 1000°C
- 800°C
- 750°C
Hot corrosion in He + 2% H₂O

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>800°C</td>
<td>CrBe₁₂</td>
</tr>
<tr>
<td>1000°C</td>
<td>CrBe₁₂</td>
</tr>
<tr>
<td>1100°C</td>
<td>CrBe₁₂</td>
</tr>
<tr>
<td>1200°C</td>
<td>TiBe₁₂ + 7vol.% Be</td>
</tr>
</tbody>
</table>

Requirements for beryllides as neutron multiplier materials

- High tritium breeding ratio – maximum possible content of Be
- Minimum content of impurities that form long-lived isotopes under irradiation (e.g. Uranium)
- Fine grain structure for easy tritium release and reasonable mechanical properties
- Beryllide blocks must retain their shape and not fracture during operation
- Low corrosion in the air and purge gas atmosphere
- Low interaction with structural materials (e.g. EUROFER steel)
- No fracture or cracks during rapid heating/cooling due to pulsed operation of DEMO
Compatibility of Ti and Cr beryllides with EUROFER

900°C, 1000 N, 100 h

TiBe$_{12}$+7%Be
CrBe$_{12}$
Requirements for beryllides as neutron multiplier materials

- High tritium breeding ratio – maximum possible content of Be
- Minimum content of impurities that form long-lived isotopes under irradiation (e.g. Uranium)
- Fine grain structure for easy tritium release and reasonable mechanical properties
- Beryllide blocks must retain their shape and not fracture during operation
- Low corrosion in the air and purge gas atmosphere
- Low interaction with structural materials (e.g. EUROFER steel)
- No fracture or cracks during rapid heating/cooling due to pulsed operation of DEMO

Thermal cycling of test sample

TiBe$_{12}$ Ø40×20

After thermal cycling
Simulation of DEMO pulse using induction heating

8760 cycles

Credit: F.A. Hernández

Thermal cycling of full-sized blocks

TiBe$_{12}$ Ø144×100

Credit: KIT
Thermal cycling of full-sized blocks

Before test

After 200 thermal cycles

Thermal cycling of blocks with updated design

Credit: G. Zhou

Credit: G. Zhou
Conclusions

- Single-phase TiBe$_{12}$ meets all the basic requirements for NMM.
- CrBe$_{12}$ is not yet well studied in terms of irradiation and thermal cycling, but may be a good alternative due to better corrosion resistance.
- The presence of a beryllium metal phase in beryllide increases ductility, but can cause accelerated corrosion or interaction with other materials.

Outlook

- New irradiation campaign up to 2-3 dpa and post irradiation examination
Potential applications of beryllides in extreme environments

Neutron multiplier material in fusion power plant blanket
- High-dose irradiation (20-50 dpa) in high-energy neutrons
- High temperature up to 1000°C
- High heating/cooling rates
- Potentially corrosive atmosphere

Structural material for the skin of spacecraft and hypersonic vehicles
- High temperatures of 1000°C and above
- Thermal shocks
- Corrosive atmosphere
- High loads
- Collision with debris

Neutron reflector material in fission reactors
- High-dose irradiation in low-energy neutrons
- Low temperature (70-150°C) – cracking / swelling
- Water environment

Thank you for your attention!
Beryllium (Be) has been a promising functional material for the breeding blanket of a fusion reactor. However, Be has disadvantages such as the sharp increase of swelling above 600°C and deterioration for mechanical properties and thermal conductivity due to irradiation damage. Be intermetallic compounds (beryllide) such as Be$_{12}$Ti and Be$_{12}$V are considered as promising advanced neutron multipliers for DEMO instead of Be, because of its excellent high-temperature stability and low reactivity with water vapor. Besides, recently, there has been a growing interest in the usage of beryllide block instead of beryllide pebble. However, the evaluation of the mechanical properties of beryllide has not been sufficiently performed. Thus, the aim of this study is to investigate the tensile properties of beryllide.

A mixed powder of Be-7.7at.% Ti, which is the stoichiometric value of Be$_{12}$Ti, was subjected to a single-phase treatment at 1200°C for 24 hours in an argon atmosphere. Also, Be$_{12}$Ti powder which has been crushed using a planetary ball mill at 300 rpm for 5 hours was prepared for comparison of the effect of refinement. For the fabricating samples, plasma sintering (KE-PAS II, manufactured by Kaken) was conducted using beryllide single-phase powder. Tensile tests were performed on various temperatures with sub-sized tensile specimens, which fabricated by electric discharge machining.

Under the above-mentioned sintering conditions, Be$_{12}$Ti single-phase beryllide was obtained, and the sintering density of Be$_{12}$Ti was almost 100% above 1000°C. Detail of tensile properties of beryllides will be reported.

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Introduction

- Developing advanced neutron multipliers
  - Beryllium (Be) used to be a candidate for neutron multipliers
    - Swelling by neutron irradiation
    - Oxidation resistance at high temperatures
    - Hydrogen generation by water vapor
    - Deterioration of mechanical properties and thermal conductivity due to irradiation
  - Beryllium intermetallic compound \((\text{Be}_{12}\text{Ti}, \text{Be}_{12}\text{V})\)

- Design

- Objective
  
  To evaluate the mechanical properties of beryllide fabricated by the plasma sintering method for establishment of the material database for DEMO design.
Experimental

- Test Material
  - Be-7.7at.%Ti isothermal heat treatment (Ar ATM 1200°C 24hr)
  - Plasma sintering
  - Ball milling (BM) 300rpm, 5hr

- Particle analysis
  - CAMSIZER X2

- Sintering condition
  - KE-PASII, kaken.co.
    - Sintered temp.: 900, 1000, 1050°C, 20 min
    - Sintered pressure: 15, 17 kN

- Hardness
  - load: 300 gf
  - Cal. For toughness: IF method, load: 5kgf

Results of XRD and size analysis

- The diffractogram suggests that no peaks were observed for any impurities, indicating that the Be$_{12}$Ti intermetallic compound was successfully synthesized.
- The average size of powder was approximately 16.5 μm
Effect of sintering temperature on density and hardness

- As the sintering temperature increases from 900 to 1050 °C, the sintering density also increases and reaches a theoretical density of 2.26 g/cm³.
- Beryllide has a low density because the low sintering temperature of 900 °C, which corresponds to 60% of the melting point (1500 °C), for Be₁₂Ti resulted in insufficient consolidation of powders.
- The plot of Vickers microhardness for Be₁₂Ti seems to be consistent with the sintering density as a function of temperature.

Effect of Sintering Temper. on Be₁₂Ti Microstructure

- Due to insufficient sintering temperature (Fig. 4(a)), consolidation between particles was not sufficient during plasma sintering, and many pores were formed on the surface.
- As the sintering temperature increased to 1050 °C, the volume fraction of pores decreased.
The fracture toughness of plasma-sintered Be$_{12}$Ti sintered at 1050 °C and 54.1 MPa was found to be comparable to that of Be$_{12}$Ti.

The fracture toughness of plasma-sintered Be$_{12}$Ti was 2.16, and 2.00 MPa·m$^{0.5}$, respectively, based on the calculation formula, indicating that it is brittle compared to Be at 9.0 MPa·m$^{0.5}$.

Summary

The mechanical properties of Be$_{12}$Ti fabricated by plasma sintering were investigated to provide important properties data for the material database for beryllide blocks as advanced neutron multipliers.

From the experimental results, the following conclusions can be drawn:

- Block-type single-phased Be$_{12}$Ti was successfully fabricated by homogenization treatment and plasma sintering.

- As the sintering temperature increased, the sintering density and microhardness increased up to 1000 °C and then saturated.

- The Vickers microhardness was congruent with the sintering density. The microhardness of Be$_{12}$Ti sintered at 1050 °C was 1154 Hv.

- To determine the fracture toughness of beryllide sintered at 1050 °C, the IF method was used, and the results showed that the fracture toughness values were 2.16, and 2.00 MPa·m$^{0.5}$ according to Anstis, and Tanaka equations, respectively. There was no significant difference compared with HIP Be$_{12}$Ti by KIT.
SESSION 5

Modeling & experimental validation
Investigation of radiation damage effects in beryllium: updates on recent results obtained on proton, neutron and He-ions irradiated samples

Slava Kuksenko

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

Beryllium is an essential material for a wide variety of application such as operating and future nuclear facilities including material testing nuclear fission reactors, fusion energy experimental and future commercial reactors, target component materials in currently running and near-future multi-megawatt particle accelerator sources. Therefore, beryllium and beryllium-based alloys are under extensive investigation by nuclear facility communities.

The paper gives an overview of the recent results obtained in the UK Atomic Energy Authority on beryllium samples, particularly during collaborative studies with the Karlsruhe Institute of Technology (Germany), the University of Horsfield (UK) and the international Radiation Damage In Accelerator Target Environments (RaDIATE) collaboration and include:

- micromechanical test results obtained on high energy proton irradiated and helium implanted samples including novel TiBe12 and CrBe12 alloys;
- atom probe tomography mapping of the fine-scale distribution of impurities and transmutants in beryllium after neutron irradiation at different DEMO relevant conditions during the HIDOBE-2 campaign in the HFR reactor;
- updates on the in-situ and ex-situ TEM studies of He implanted beryllium, TiBe12 and CrBe12 alloys.

The paper will also give an overview of the future plans.

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

Slava Kuksenko
UK Atomic Energy Authority

This work was supported by the Fermi Research Alliance, LLC under Contract No. DE-AC02-07CH11359 with the United States Department of Energy, and by the UK Science and Technology Facilities Council. This work has been part funded by the RCUK Energy Programme [grant number EP/T012250/1].

Outline
- Hardening and embrittlement under the high energy proton irradiation at 50ºC (in the NuMI beamline);
- Hardening and embrittlement under helium implantation at 50ºC and 200ºC;
- Effects of neutron irradiation at different DEMO relevant conditions during HIDOBE-2 campaign in the HFR
- Thermal shock effects in beryllium
- Investigation of correlation between neutronic performance of beryllium and its microstructure and chemical composition
- Erosion and tritium retention in JET plasma facing beryllium
- Combined Effects of Light Gas and Damage Accumulation in Beryllium (poster of J. Sharp at SOFT)
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

Beryllium is of particular importance for nuclear application:
- First wall material in JET experiment and the currently constructed ITER
- Neutron multiplier candidate material for tritium breeding blanket of the future DEMO and STEP fusion reactors
- Reflector and moderation in research reactors and spallation neutron sources
- Beam windows and target components material in neutrino sources

<table>
<thead>
<tr>
<th>Reactor/Source</th>
<th>He Production in Beryllium, appm/dpa</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMA-3 high-flux reactor (Russia)</td>
<td>130</td>
</tr>
<tr>
<td>BOR-60 reactor (Russia)</td>
<td>280</td>
</tr>
<tr>
<td>HFR, HIBALL-03 irradiation campaign (Petten, Netherlands)</td>
<td>160</td>
</tr>
<tr>
<td>Beryllium reflector in the ISIS neutron source (RAL, UK)</td>
<td>130 - 220</td>
</tr>
<tr>
<td>DEMO fusion reactor</td>
<td>700</td>
</tr>
<tr>
<td>Neutrino target component (FNAL, USA)</td>
<td>4000</td>
</tr>
</tbody>
</table>

Proton irradiated beam window: (NuMI)
- 120GeV proton beam
  - $1.57 \times 10^{21}$ protons during its lifetime
  - Up to 0.5 dpa and 2000 appm of He
  - $T = 50^\circ C$
120GeV proton beam
- $1.57 \times 10^{21}$ protons during its lifetime
- Up to 0.5 dpa and 2000 appm of He
- $T = 50^\circ C$

Proton irradiated beam window: (NuMI)

Sample preparation and light microscopy done at KIT

Non-irradiated beryllium – mainly transgranular cleavage

Grain-boundary fracture may be caused by strengthening of the matrix or “weakening” of GBs

Transition from transgranular to grain boundary/mixed mode fracture
Micro-cantilevers fracture tests, as-received vs irradiated state

- 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
• 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?
He implantation in Be through Al degrader (50°C and 200°C)

- radiation damage appeared as “black dots” (less than 10 nm) arranged in lines parallel to the surface of samples
- the damaged layers of the samples exposed at 50 and 200°C were very similar and no difference was noticed between two investigated beryllium grades (FIB damage?)
- no cavities or He bubbles at both temperatures, including grain boundary areas.

Micro-cantilevers fracture tests, He implantation vs transmutation under proton irradiation

- Fracture load of both GB and cleavage cantilevers increased after irradiation and ion implantation. No GB softening is observed
- Work of fracture is lower for cantilevers pre-notched for the basal cleavage, but the difference between two types of cantilevers is after irradiation
• Low purity grade is harder in as-received state and after implantations
• Irradiation induced hardening after 50°C implantation is about 75% higher than after 200°C.
• Less pure grade (S-200) exhibited higher hardening (especially for “soft” grains)

He implantation vs transmutation under proton irradiation at 50°C

• Same He content introduces similar hardening
• Same displacement damage – less hardening after implantation
• Comparison dpa and He concentrations infuses (>0.1dpa) on hardening suggest that He concentration dominates
Hardening of He implanted samples:

Dispersed barrier hardening model:

As a first approximation:

\[
\Delta \sigma_y = aM\mu b\sqrt{N} + d\quad \text{(} M = 4.3, \mu = 133\text{GPa}, b = 0.23\text{nm}, N \approx 10^{22} \text{m}^{-3} d \approx 5\text{nm})
\]

\[
\Delta H = 3 \Delta \sigma_y
\]

<table>
<thead>
<tr>
<th>Implantation at 50°C</th>
<th>Implantation at 200°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measured av. hardening</td>
<td>3.3 GPa</td>
</tr>
<tr>
<td>Black dots hardening, 0.4</td>
<td>From 0.85 to 1.7 GPa depending on</td>
</tr>
<tr>
<td>Non-&quot;black-dots&quot; hardening</td>
<td>From 1.5 to 2.6 GPa</td>
</tr>
</tbody>
</table>

May it be from solid solution hardening?

\[
\Delta \sigma_y = \frac{M}{760} \mu \cdot \delta^2 \cdot c_{\text{He}}^{1/2} \approx 33\text{MPa} \quad \text{for} \ 2000\text{appm of He in solid solution}
\]

So, He should be in invisible by TEM bubbles

More details in Kuksenko et al. JNM 555 (2021)

Conclusions (1st part)

1. Radiation induces significant hardening and fracture mechanism change of beryllium even at 0.1 dpa
2. Irradiation at 200°C leads to much lower hardening
3. Less pure grade (S-200) exhibited higher hardening (especially for "soft" grains)
4. Both displacement damage and He atoms cause hardening. We have indications that He content has a dominant effect.
5. He implantation led to increase of fracture strength of pre-notched microcantilevers and decrease of work to fracture, with severe drop for 50°C implantation and high energy proton irradiation
6. Calculations suggest that the radius of bubbles should be \( \approx 0.8\ldots1.5\text{ nm} \) with \( \text{Nd} > 1023\text{m}^{-3} \) and they should be weak obstacles \( \alpha \leq 0.2 \)

Difference between 50 and 200°C implantation may originate from different sizes or different vacancy/He ratio in bubbles
Irradiation at HIDOBE-02 (collaboration with KIT)

<table>
<thead>
<tr>
<th>Name</th>
<th>T irr, °C</th>
<th>dpa</th>
<th>He, appm</th>
<th>T, appm</th>
<th>Experiments</th>
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<tbody>
<tr>
<td>6bPC2</td>
<td>600</td>
<td>34</td>
<td>5557</td>
<td>600</td>
<td>SEM, EDX, EBSD, APT, Raman</td>
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<tr>
<td>6mPC1</td>
<td>480</td>
<td>28.2</td>
<td>4788</td>
<td>508</td>
<td>SEM, EDX, EBSD, APT</td>
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<tr>
<td>5mPC1</td>
<td>387</td>
<td>19.5</td>
<td>3638</td>
<td>369</td>
<td>SEM, EDX, EBSD</td>
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<tr>
<td>49sPC</td>
<td>370</td>
<td>20.8</td>
<td>3632</td>
<td>367</td>
<td>SEM</td>
</tr>
<tr>
<td>8sPC</td>
<td>560</td>
<td>34.4</td>
<td>5524</td>
<td>596</td>
<td>SEM</td>
</tr>
<tr>
<td>7sPC</td>
<td>650</td>
<td>37.2</td>
<td>5925</td>
<td>644</td>
<td>SEM</td>
</tr>
<tr>
<td>1473-10</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>SEM, EDX, EBSD, Raman</td>
</tr>
</tbody>
</table>

- 6 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.”

EDX and APT chemical maps: as-received state

- Precipitates have white contrast on the SEM image and located at high- (>10°) and low-angle (<10°) GB-s as well as inside the grains;
- Majority of the white contrast particles are enriched with Al, Si, Mg and Ti. Some precipitates enriched by Fe and, rarely, by O and Cr.
The EDX chemical elements distribution in the pebbles irradiated at 378°C, 480°C was very similar to the one observed in the unirradiated pebbles.

In the pebbles irradiated at 600°C more than a hundred micrometres wide channels with reduced impurity content and almost free of precipitates were observed.

Nanometric-scale investigation of distribution of tritium transmutant

New mass spectrum peaks appear after neutron irradiation at "3", "6" and "12" Da should be related to tritium and/or lithium.
APT chemical maps: irradiation at 387°C (work in progress)

After irradiation at 387°C, matrix composition is similar to the one before irradiation.
- New mass-spectrum peaks (3 Da, 6 Da and 12 Da) have been observed.
- These new transmutants (presumably Li and T) are observed only near the BeO and Fe-Al-Si precipitates. And absent in the matrix.

APT chemical maps: irradiation at 600°C (work in progress)

The sample has many He bubbles that makes APT sample preparation and analysis very difficult.
- After irradiation at 600°C, matrix composition is similar to the one before irradiation.
- New mass-spectrum peaks (3 Da, 6 Da and 12 Da) have been observed.
- These new transmutants (presumably Li and T) are observed only near the BeO/Fe-Co-Si precipitate. And absent in the matrix.
The "new elements/isotopes" are non-homogeneously distributed (480°C irradiation).

Comparison with TEM suggests that the new element(-s) segregate to helium bubbles (hexagon coins shaped).
Conclusions (preliminary)

- New mass spectrum peaks appear after neutron irradiation at "3", "6" and "12" Da should be related to tritium.
- The "new elements/isotopes" are non-homogeneously distributed.
- The transmutants (presumably Li and T) are observed only near the BeO and Fe-Al-Si precipitates in the HIDOBE samples irradiated at 387 and 600°C.
- The plate-like shape suggests that in the HIDOBE samples irradiated at 480°C the new element(s) segregate to helium bubbles.
- Matrix composition of n-irradiated HIDOBE samples is similar to the one before irradiation. There is not transmutants detected in the matrix.

Acknowledgements:
MRF and health physics teams

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Thank you!
First principles simulation of resistivity recovery in irradiated beryllium

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Future fusion devices like ITER or DEMO require closed fuel cycles. These vitally depend on neutron multiplying materials as part of a breeding blanket module like beryllium pebbles in the European Helium-Cooled Pebble-Bed. During operation the beryllium pebbles will accumulate point defects, tritium, and helium due to inevitable exposure to highly energetic neutron irradiation as emitted by the fusion plasma. A detailed knowledge of the characteristics of point defects is decisive for reliable simulations of microstructure evolution in irradiated beryllium. Such models are a prerequisite for predicting tritium inventory during operation as well as after the blanket’s end of life since tritium retention and release is the paramount safety concern.

A well-established experimental approach to assess the dynamics of relevant atomic defects consists in measuring electrical resistivity recovery (ERR) after irradiation during annealing. Within this approach, temperatures corresponding to electrical recovery steps are correlated with activation energies which are associated with different type of reactions between defects.

In this work, results of our ongoing efforts to model and understand the ERR of beryllium are presented. To that end, we introduce a rate equation-based approach to model ERR spectra (see picture below) utilizing density functional theory results as input. Within this approach, electrical resistivity recovery models comprising the volume of spontaneous recombination of monovacancies and self-interstitial atoms in beryllium as well as various additional defects are considered. As a result, an intricate interplay between different defect dynamics is uncovered, suggesting a clear route for further research to obtain systematically improved electrical resistivity recovery models.

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First principles simulation of resistivity recovery in irradiated beryllium

Christopher Stihl

Outline
- Resistivity Recovery Experiments
- Resistivity Recovery Modelling
  - Relevant Be properties
  - Model derivation
  - Various models
- Conclusions & outlook
Resistivity recovery experiment(s by example)

- Sample preparation
  - Cool to 4.2 K
  - Measure resistivity
  - Irradiate
  - Measure resistivity
- Measurements
  - Do annealing cycle
    - Heat
    - Hold
    - Cool
  - Measure resistivity
  - Repeat at higher T
- Process spectrum

Model-relevant Be properties (from DFT)

- HCP structure
- AB-stacked layers
- Mono vacancies
  - Immobile (0.8 eV)
- Basal interstitials
  - Mobile (0.12 eV)
- Spontaneous recombination hull
  - Oblate spheroid
  - With (very) fast recombination
Deriving a hull-only recovery model ...

... as an initial value problem (IVP):
- recombination from hull
- various small barriers $\Delta E < 0.12 \, \text{eV}$
- $c_0 \approx 1\%$ per hull defect pair
- ordinary differential equations (ODEs)

\[
\frac{d}{dt} c \left( \begin{array}{c} \text{hull} \\ \end{array} \right) = -c \left( \begin{array}{c} \text{hull} \\ \end{array} \right) \cdot \nu \cdot e^{-\frac{\Delta E}{k_B T(t)}}
\]

\[
\frac{d}{dt} c \left( \begin{array}{c} \text{various small barriers} \end{array} \right) = + \sum c \left( \begin{array}{c} \text{various small barriers} \end{array} \right) \cdot \nu \cdot e^{-\frac{\Delta E}{k_B T(t)}} \propto \text{recovery signal}
\]

Solving the hull-only recovery model’s IVP

- ODEs

\[
\frac{d}{dt} c \left( \begin{array}{c} \text{hull} \\ \end{array} \right) = -c \left( \begin{array}{c} \text{hull} \\ \end{array} \right) \cdot \nu \cdot e^{-\frac{\Delta E}{k_B T(t)}}
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\]

- $T(t)$ from experiment
- initial values of $\approx 1\%$ per defect
Solving the hull-only recovery model’s IVP

- ODEs
  \[ \frac{d}{dt} c(t) = -c(t) \cdot \nu \cdot e^{\frac{-\Delta E}{k_B T(t)}} \]
  \[ \frac{d}{dt} \sum c(t) = \sum c(t) \cdot \nu \cdot e^{\frac{-\Delta E}{k_B T(t)}} \]

- \( T(t) \) from experiment
- Initial values of \approx 1\% per defect
- Symmetric hull and recovery evolution
- Sloped step patterns from annealing cycles

\[ \sum c(t) \]

\[ T(t) \]

\[ \text{time (s)} \]

\[ \text{concentration (\%)} \]

Solving the hull-only recovery model’s IVP

- ODEs
  \[ \frac{d}{dt} c(t) = -c(t) \cdot \nu \cdot e^{\frac{-\Delta E}{k_B T(t)}} \]
  \[ \frac{d}{dt} \sum c(t) = \sum c(t) \cdot \nu \cdot e^{\frac{-\Delta E}{k_B T(t)}} \]

- \( T(t) \) from experiment
- Initial values of \approx 1\% per defect
- Symmetric hull and recovery evolution
- Sloped step patterns from annealing cycles
- +/- peaks show in/decreasing concentration
- Lowest-energy hull pairs decay early
- Initial values are important free parameter
Defect mobility-enhanced recovery model …

... by adding:
- free interstitials & vacancies
- \( \Delta E \approx 0.12 \, eV \)

new corresponding ODEs

\[
\frac{d}{dt} c(\text{Interstitials}) = \sum c(\text{Interstitials}) \cdot \nu \cdot e^{\frac{-\Delta E}{k_B T(t)}} - c(\text{Interstitials}) \cdot c(\text{Vacancies}) \cdot \nu \cdot e^{\frac{-\Delta E}{k_B T(t)}}
\]

\[
\frac{d}{dt} c(\text{Vacancies}) = \sum c(\text{Interstitials}) \cdot \nu \cdot e^{\frac{-\Delta E}{k_B T(t)}} - c(\text{Interstitials}) \cdot c(\text{Vacancies}) \cdot \nu \cdot e^{\frac{-\Delta E}{k_B T(t)}}
\]

additional terms in existing ODEs

---

Defect mobility-enhanced recovery spectrum

**hull-only model**

**defect mobility-enhanced model**

C. Stihl et al., First Principles Simulation of Resistivity Recovery in Irradiated Beryllium
Impurity-enhanced recovery models …

... from adding (de)trapping at impurities

\[ \frac{d}{dt} c = + c \left( \frac{v}{k_B T} \right) e^{-\Delta E/k_B T} \]

Interstitials (de)trapping at Fe adds ODEs

\[ \frac{d}{dt} c = - c \left( \frac{v}{k_B T} \right) e^{-0.12 eV/k_B T} + c \left( \frac{v}{k_B T} \right) e^{-\Delta E/k_B T} \]

\[ \frac{d}{dt} c = - c \left( \frac{v}{k_B T} \right) e^{-\Delta E/k_B T} + c \left( \frac{v}{k_B T} \right) e^{-0.12 eV/k_B T} \]

Analogous vacancy (de)trapping ODEs

Additional terms in existing ODEs

Fe impurity-enhanced recovery spectrum

defect mobility-enhanced model

Impurity-enhanced model
**Fe impurity-enhanced recovery spectra**

<table>
<thead>
<tr>
<th>impurity-enhanced model</th>
<th>impurity-enhanced, vac-starved model</th>
</tr>
</thead>
<tbody>
<tr>
<td>recovered</td>
<td>free Fe</td>
</tr>
<tr>
<td>free ints</td>
<td>Fe-bound ints2</td>
</tr>
<tr>
<td>free vats</td>
<td>Fe-bound ints1+3</td>
</tr>
<tr>
<td>hull</td>
<td>Fe-bound vats</td>
</tr>
<tr>
<td>experiment</td>
<td></td>
</tr>
</tbody>
</table>

**Conclusions & outlook**

- first peaks due to closely correlated pairs
- reasonable defect concentrations sufficient
- gaps likely due to non-exhaustive set of pairs
- diffusion onset decisive for later spectrum
- last peaks due to mobile vacancies
- here from vats weakly bound to Fe
- interdependent dynamics of different traps
  - e.g. different traps at Fe (and additional ones …)
- intermediate peaks **likely not** due to impurities
  - concentrations too low
  - might still contribute some peaks
- additional Be-only defects to be considered
- model can be extended to calorimetry signals
Radiation induced formation gas bubbles in beryllium after neutron irradiation up to 6000 appm helium production

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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. 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 14 MeV 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 45 MW 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 the uniform shape. The diameters of the bubbles increase from a few nanometers for 410°C to more than hundred nanometers for 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. Also the Si and Fe segregation on the voids was detected [2].

EDX mapping shows that the precipitates inside the grains and on the GBs have increased iron and aluminum content, indicating the formation of an Fe-Al-Be phase.

In the material irradiated at 440°C, most of the precipitates also have Fe-Al-Be composition, while several other single- and multiphase precipitates were found. The Fe-Al-Be phase is observed as 10-15 nm precipitates within the grains and as 200 nm particles bound to a gas bubble at the GB. The present study shows detailed microstructural changes induced by neutron irradiation in beryllium.


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

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Content

- Introduction
- Microstructural examination of neutron irradiated beryllium at IAM-AWP (HIDOBE I and HIDOBE II)
- Detection of He and $^3$H inside bubbles
- Distribution of impurity atoms in irradiated Be
- Conclusions
Beryllium application in fusion technology

- 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.

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

- $^{9}$Be + n → $^2$He + 2n ($E_{n}>2.7$ MeV)
- $^9$Be + n → $^7$Li + $^3$He ($E_{n}>10.7$ MeV)
- $^7$Li + n → $^4$He + $^3$H + n
- $^9$Be + n → $^6$He + $^3$H ($E_{n}>0.6$ MeV)
- $^6$He → $^6$Li + b– + ν
- $^6$Li + n → $^4$He + $^3$H

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.

Neutron irradiation programs 2005 - 2016

**High Dose Beryllium irradiation program (HIDOBE-I) (2005-2007)**
High-Flux Reactor (HFR), Petten, Netherlands
Dose 18 dpa, 3000 appm $^4$He, 300 appm $^3$H
Irradiation temperatures: 410°C, 480°C, 590°C, 700°C

**High Dose Beryllium irradiation program (HIDOBE-II) (2005-2011)**
High-Flux Reactor (HFR), Petten, Netherlands
38 dpa displacement per atom 5900 appm $^4$He, 640 appm $^3$H
Irradiation temperatures: 370°C, 440°C, 540°C, 650°C
**HIDOBE-I – TEM sample preparation**

Irradiated beryllium pebbles

Crushing tool for preparation of Be powder

After grinding of Be pebble small pieces were deposited on the copper grid covered by carbon film.

TEM images of small powder particles

**Be fiber 27µm*0.5µm**

**HIDOBE-I post irradiation examination**

Bubbles – hexagonal shape

Bubbles – rectangular shape

C-axis

Prismatic-disc shape of bubbles in beryllium
HIDOBE-I - post irradiation examination

Precipitates 480°C

The FeAlMnCr precipitates located in a fiber of 350 nm thickness.


HIDOBE-I post irradiation examination

Precipitates contain FeAlMnCrTi beryllides

HIDOBE-II

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

Microstructural examination:
2016-2021

Transmission Electron Microscope
Talos F200X G2 / 200 kV FEG

TEM lamellae were prepared from irradiated tungsten using focused ion beam (FEI SCIOS) in the FML at KIT.

Specimen preparation using Focussed Ion Beam

Removal of larger amounts of surrounding material using Ga ion beam.
Deposition of prepared lamellae on the Cu greed.
Preparation of thin transparent window for TEM analysis.
The thickness varied from 120 to 350nm mean path in beryllium ≈0.6-2.1λ.
A 500nm lamella is ready for TEM analysis.
Microstructure of irradiated beryllium

- Bubbles show hexagonal prismatic-disc shape in the transmission electron microscope (TEM)

![Diagram showing c-axis and prismatic-disc shape of bubbles in beryllium]

View on basal face: (1120)

View on prismatic face: (0001)

Tomography in TEM

66° total tilt (from -32° to +34°)

23 images

Step ~3°
Bubbles in beryllium irradiated at different temperatures

Images of the bubbles in the basal and prismatic orientations

- 440°C
- 520°C
- 650°C

Bubbles in beryllium irradiated at 370°C

Tilt of 60° between these images

A uniform bubble shape

10nm
Quantitative evaluation

Effect of radiation dose on the microstructure of irradiated beryllium

**Bubble diameter**

- **HIDOBE II**
- **HIDOBE I**

~20% increase

**Swelling**

- **HIDOBE II**
- **HIDOBE I**

~25% increase

---

Analytical study of bubbles in irradiated beryllium

Low energy EELS spectra

TEM dark field image

- **3H2 signal**
- **He signal**

Formation of beryllium hydride \( \text{BeH}_2 \) layers on internal walls

He detection inside bubbles

Calculation of the gas pressure inside the individual bubbles:

\[
\text{pressure} = \frac{I_x}{d \cdot \sigma_0} \cdot \frac{1}{n_x}
\]

- \( n_x \) is the number density of the gas
- \( I_x \) and \( f_x \) are integrated intensities from the spectra
- \( \sigma_0 \) is the partial inelastic cross-section for He/H
- \( d \) is the bubble thickness in the direction of electron beam.

\( n_{\text{He}} = 4.2 \pm 1.0 \) at/nm\(^3\)

\( n_{\text{H}_2} = 3.5 \pm 0.8 \) at/nm\(^3\)
Analytical study of bubbles in irradiated beryllium: Dr. N. Zimber

EELS spectrum inside of the bubble (red) and a spectrum outside of the bubble (blue)

Dr. Michael Klimenkov
Institute of Applied Materials – Applied Materials Physics (IAM-AWP)

M. Klimenkov et al., Radiation Induced Formation of Gas Bubbles in Be After n-Irradiation
HIDOBE II publications

- M. Klimenkov, P. Vladimirov, J. Hoffmann, N. Zimber, A. Möslang, V. Kuksenko “First simultaneous detection of helium and tritium inside bubbles in beryllium” Micron 127 (2019) 102754
- N. Zimber, P. Vladimirov, “The role of grain boundaries and denuded zones for tritium retention in high-dose neutron irradiated beryllium” Journal of Nuclear Materials 568 (2022) 153855

Distribution of impurity elements

presence of precipitates at 370°C and 440°C

A loop decorated by a segregation of Fe-Al-Be phase pinned by a complex phase precipitate (a) observed at 643 K (370 °C).

Various phases in EDX map are colored as follows:
- AlFe
- MnSi
- CrTi
- UFe
Summary

- Successful microstructural examination of neutron irradiated beryllium at IAM-AWP over the past 10 years (6 publications in peer reviewed journal).
- Detection of He and H₂ in bubbles by TEM in neutron irradiated Be was reported for the first time.
- Recent study were focused on the characterization of gas bubbles located inside grains and at the GBs as well as on the study of spatial distribution and composition of secondary phase precipitates.
- **Recent results (HIDOBE II):**
  - Helium bubbles were found inside grains at all irradiation temperatures, which is consistent with previous literature. Their shape is spherical at 370°C and changes to a flat hexagonal prism at higher irradiation temperatures. The bubble size increases strongly with irradiation temperature. The apparent swelling estimated from the TEM data reaches ~9% at 650°C
  - Precipitation of Fe-Al-Be phase within grains were detected at low irradiation temperatures (370°C and 440°C)
  - EELS spectroscopy was used for detection and analysis of He and H₂ gases trapped inside flat hexagonal bubbles formed on the basal planes of beryllium under neutron irradiation.
  - The number densities of both gases inside the bubbles were calculated using atomic scattering cross-section and the intensity of the zero-loss peak. The values He=(4.2±1) at/nm³ and nH₂=(3.5±1.2) molecules/nm³ were determined for a bubble with a diameter of 160nm.
Effect of impurities on microstructural evolution under irradiation in beryllium

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Impurities are known to affect mechanical properties of beryllium, but their effect on development of irradiation induced microstructure is still unknown. In this contribution we are making further attempt to reveal behavior of impurities in neutron irradiated beryllium pebbles by using both analytical transmission electron microscope (TEM) and first principles computer simulations.

TEM studies have revealed Al-Fe-Be precipitates, complex multiple phase precipitates, homogeneous segregations of elements to grain boundaries as well as abundant precipitation along dislocations. All precipitates are richly decorated with helium bubbles which are smaller in size than typical bubbles inside grains. Precipitate-free and helium-bubble-free zones were observed along grain boundaries.

Using density functional theory approach, we have calculated interaction of typical solutes found in beryllium, namely, Al, Fe, Cr, Mg and Si with vacancies, interstitials and free surfaces which can simulate a surface of helium bubbles. Interesting correlation has been revealed: an impurity which has attractive binding with a vacancy has also positive affinity to free surface. In particular, Al, Mg and Si are strongly bound with vacancies and also attracted by the free surfaces. This result is supported by the EDX measurements, (see Fig. above) which reveal decoration of He bubbles with Al, Si and Mg, while Fe is homogeneously distributed. Those impurities which repulse vacancies are attracted by self-interstitials, however, no correlation with the formation volume of respective substitutonal atoms was found in this case.

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Effect of impurities on microstructural evolution under irradiation in beryllium

P. Vladimirov, D. Bachurin, C. Stihl, T. Le Crane and N. Zimber

Outline

- Introduction
  - Beryllium as neutron multiplier
  - Typical impurities in Be
  - Behavior under irradiation
- Calculation methods
- Results
  - DFT results
    - Solutes and their dipole tensors
    - Interaction with vacancies and interstitials
    - Interaction with surfaces
  - Comparison with experimental results
- Discussion/Conclusions
Beryllium as neutron multiplier in ITER TBM

- Lithium and beryllium are needed to increase TBR and close fuel cycle
- Beryllium will be used as 1-mm pebbles filling space within BU around Li-ceramic layers
- Formation of gas filled bubbles (swelling) is critical for tritium accumulation

BU = Breeder Unit
Consists of Li-ceramics and Be interchanging pebble beds

Impurities in Be

- Why impurities are important:
  - Mechanical properties (hot shortness)
  - Activation under irradiation (e.g., U)
  - Affect microstructure development under irradiation

- Major metallic impurities are:
  - Mg, Al, Si
  - Cr, Fe

- Major non-metals:
  - C, O

- Impurities produced by neutron-induced nuclear transmutations:
  - He, Li

Impurities in as received Be

Impurities in as received Be

Above 850°C:
- Al und Fe in solid solution
- Below 650°C precipitate as AlFeBe₄

650–850°C → AlFeBe₄

Fe

Al + FeBe₁₁ + Be

> 850°C

Beryllium microstructure under irradiation

Loops & precipitates  Loops, bubbles & precipitates  Gas-filled bubbles

Irradiation temperature

0°C  150°C  300°C  350°C  400°C  450°C  500°C  550°C  600°C

Loops, bubbles & precipitates

70 - 120°C

370°C

P. Vladimirov et al., Effect of Impurities on Microstructural Evolution under Irradiation in Beryllium
Beryllium microstructure under irradiation

Loops & precipitates  Loops, bubbles & precipitates  Gas-filled bubbles

0°C 150°C 300°C 350°C 400°C 450°C 500°C 550°C 600°C

0°C 150°C 300°C 350°C 400°C 450°C 500°C 550°C 600°C

Loops, bubbles & precipitates

370°C 387°C

Irradiation temperature

70 - 120°C

387°C

370°C

70 - 120°C

387°C

70 - 120°C
Beryllium microstructure under irradiation

**Loops & precipitates**

**Loops, bubbles & precipitates**

**Gas-filled bubbles**

Irradiation temperature

- 0°C
- 150°C
- 300°C
- 350°C
- 400°C
- 450°C
- 500°C
- 550°C
- 600°C

- 70 - 120°C
- 370°C
- 387°C
- 487°C
- 600°C

**Notes:**

- C-axis: [0001], [11¯20]
- Gas-filled bubbles
- Loops, bubbles & precipitates
- Irradiation temperature range: 0°C to 600°C

---

P. Vladimirov et al., Effect of Impurities on Microstructural Evolution under Irradiation in Beryllium
SESSION 5  MODELING & EXPERIMENTAL VALIDATION

Beryllium microstructure under irradiation

Loops & precipitates  Loops, bubbles & precipitates  Gas-filled bubbles

What is the role of impurities for microstructure evolution? How solute atoms interact with He-bubbles?

Irradiation temperature

0°C  150°C  300°C  330°C  360°C  400°C  450°C  500°C  550°C  600°C

Loops, bubbles & precipitates

Gas-filled bubbles

600°C

Simulation methods

Density Functional Theory

VASP 5.3 or 6.2.0

Generalized Gradient Approximation (GGA)

Pseudopotentials:

Plain Augmented Waves (PAW), xc=PBE_54

Li:  2s1  Al:  3s2 3p1
Be:  2s2  Si:  3s2 3p2
Mg:  3s2  Cr:  4s1 3d5
Fe:  3d6 4s2

Gamma centered Monkhorst-Pack k-point grid
13 × 13 × 13 or KSPACING = 0.12016

Energy cutoff \( \text{ENCUT} \geq 450 \text{ eV} \)

Simulation cell sizes for solute-vacancy interaction:

- \( 4 \times 4 \times 3 \) – 96 atoms
- \( 5 \times 5 \times 3 \) – 150 atoms
- \( 6 \times 6 \times 3 \) – 216 atoms

Simulation cell sizes for slabs:

- \((0001) 4\times4\times12\) – 384 atoms
- \((1\bar{1}00) 3\times4\times17\) – 168 atoms

Migration barrier calculations:

- NEB
- Dimer
Solute-vacancy interaction

- Insufficient size of simulation cell along c-axis.

Interaction of solutes with SIAs in Be

- Self-interstitial configuration with the lowest energy (ground state) is basal octahedral (BO) position.
- Interaction with solute atom changes this ground state configuration dramatically in some cases.
Interaction of solutes with SIAs

SIAs are bound to Cr and Fe in non-basal configurations, but are repelled from Al, Mg and Si.

Largest repulsion and lowest binding are observed for basal configurations.

For both Cr and Fe, basal configuration is loosely bound => conversion from non-basal to basal configuration might be an important step for SIA detrapping.
Interlayer distance after slab relaxation

Impurities near Be (0001) surface

He is slightly repelled by surface, but favorable inside vacuum
Impurities near Be (0001) surface

- **He** is slightly repelled by surface, but favorable inside vacuum
- **Li** is also slightly repelled, but favorable at the surface
- **Mg** is attracted by surface
Impurities near Be $(0001)$ surface

- $\text{He}$ is slightly repelled by surface, but favorable inside vacuum
- $\text{Li}$ is also slightly repelled, but favorable at the surface
- $\text{Mg}$ is attracted by surface
- $\text{Al}$ is similar to $\text{Mg}$
- $\text{Si}$ is the most favorable impurity at the surface
Impurities near Be (0001) surface

- **He** is slightly repelled by surface, but favorable inside vacuum
- **Li** is also slightly repelled, but favorable at the surface
- **Mg** is attracted by surface
- **Al** is similar to Mg
- **Si** is the most favorable impurity at the surface
- **Cr** is attracted, but the most favorable position is in the first subsurface layer
- **Fe** is unfavorable at the surface
Impurities near Be \((1\bar{1}00)\) surface

- He goes inside bubble from layers \#0 & 1
- Li is also slightly repelled, but favorable at the surface
Impurities near Be (1100) surface

- **He** goes inside bubble from layers #0 & 1
- **Li** is also slightly repelled, but favorable at the surface
- **Mg** goes to the surface from layers #0 & 1

**Diagram:**
- Energy gain vs. slab depth
- Layers: 0, 1, 2, 3, 4, 5, 6, 7

**Graph:**
- X-axis: Slab depth, Å
- Y-axis: Energy gain, eV

**Legend:**
- Mg
- Al (similar to Mg)
Impurities near Be (1100) surface

- He goes inside bubble from layers #0 & 1
- Li is also slightly repelled, but favorable at the surface
- Mg goes to the surface from layers #0 & 1
- Al is similar to Mg
- Si is the second favorable impurity at the surface after Mg
- Cr is slightly unfavorable at the surface, but favorable at subsurface layers #2 & 3
Impurities near Be (1100) surface

- He goes inside bubble from layers #0 & 1
- Li is also slightly repelled, but favorable at the surface
- Mg goes to the surface from layers #0 & 1
- Al is similar to Mg
- Si is the second favorable impurity at the surface after Mg
- Cr is slightly unfavorable at the surface, but favorable at subsurface layers #2 & 3
- Fe is unfavorable at the surface, but slightly favorable at subsurface layer #3

Interaction of impurities with surfaces

- He
- Li
- Mg
- Al
- Si
- Cr
- Fe

Energy gain, eV

-3.00
-2.25
-1.50
-0.75
0.00
0.75
1.50

Layer #: 0, 1, 2, 3, 4, 5, 6, 7

Slab depth, Å

He Li Mg Al Si Cr Fe

(0001)

(1-100)
Comparision with experiment

\[ T_{irr} = 600^\circ C \]

- Al
- Fe
- Mg
- Si
- HAADF

Net intensity vs. position (nm)
Comparision with experiment

- Fe is homogeneously distributed over beryllium matrix
- Al, Si and Mg cover surfaces of helium bubbles

\[ T_{\text{irr}} = 600^\circ\text{C} \]

Conclusions

- DFT calculations suggest that there is a correlation between interaction of solutes with vacancies, surfaces and self-interstitials; those which have binding with vacancies are also attracted by free surfaces and repelled by self-interstitials.
- Mg, Al, Si have positive binding with vacancies and free surfaces
- Cr and Fe have no binding with vacancies, also have no binding or even slightly repelled by surfaces, but have attraction to SIAs

- Consequences for diffusion mechanisms:
  - Fe and Cr migrate against vacancy gradient
  - Si and Al migrate along vacancy gradient as Solute-Vac complex (e.g. to bubbles or dislocations)

- These findings were confirmed experimentally using TEM equipped with EDX:
  - formation of Mg, Al, Si enriched zones around gas bubbles in beryllium under irradiation
  - formation of gas bubbles on FeAlBe4 precipitates as a consequence of Vac-Al complex diffusion

- Obtained results will be used to evaluate the effect of impurities on gas bubble growth, swelling and tritium retention inside helium bubbles.
Ab initio study of hydrogen behavior in titanium beryllides

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An interest in titanium beryllides as candidate materials for advanced neutron multiplier for the Helium Cooled Pebble Bed breeding blanket of European DEMO fusion reactor is related to their lower tritium retention, lower swelling and higher oxidation resistance in comparison with pure beryllium. The latter was initially suggested as neutron multiplier in the International Thermonuclear Experimental Reactor (ITER) and for the above reasons has a number of limitations compared to beryllides.

One of the most important questions is how much weaker tritium, which is formed as a result of the interaction of high-energy neutrons with the pebbles is bound in titanium beryllides in contrast to pure beryllium. Such an interaction awakens formation of helium bubbles and degradation of the material properties. One of the main promising methods for studying the behavior of hydrogen in titanium beryllides is first-principles modeling technique based on density functional theory.

The present work is devoted to ab initio study of hydrogen (isotope effects were neglected and hydrogen was considered instead of tritium) behavior in three titanium beryllides (Be2Ti, Be17Ti2, Be12Ti). All of them have different crystal structure and contain a different number of crystallographically non-equivalent interstitial hydrogen sites.

Both the hydrogen solution energy in defect-free lattice and binding energy with a vacancy are important characteristics in terms of tritium dissolution, retention and release. Static ab initio calculations demonstrate that hydrogen solution energy in all interstitial non-equivalent sites is noticeably lower as compared with pure beryllium suggesting an easier dissolution of hydrogen atoms in titanium beryllides. Computation of binding energy of single hydrogen atom with all non-equivalent monovacancies reveals that hydrogen might be trapped by a vacancy without being inside it. The obtained results sheds light on the understanding of earlier tritium release in different titanium beryllides during thermo-desorption experiments and expand our knowledge of their properties.

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Ab initio study of hydrogen behavior in titanium beryllides

Dmitry Bachurin, Christopher Stihl, Pavel Vladimirov

Outline
1. Motivation and goals
2. Computational methodology
3. Structure and lattice parameters
4. Hydrogen in interstitial positions
5. Diffusion of hydrogen in Be₁₂Ti
6. Hydrogen in vacancies
7. Conclusions
**Beryllium in fusion reactor**

- Be is considered as **plasma facing material** and as effective **neutron multiplier** for tritium breeding blanket (HCPB)
- Hydrogen isotopes and impurities are implanted into Be first wall tiles
- He and T are produced in Be under neutron irradiation

**Be: 680 m² or 12.5 t**

**TBM ITER – 2 t**

**DEMO – 300 t**

**ITER Torus cross-section**

K. Ioki et al. Nucl. Fusion 41(3) 2001 265-275

---

**Motivation and goals**

- **Motivation**: Intermetallic beryllium compounds as Be$_{12}$Ti, Be$_{12}$V and Be$_{12}$Zr are considered as possible candidates for fusion applications, namely as neutron multiplier for the DEMO breeder blanket to be used instead of pure beryllium.

- **Global Goal**: Elucidation of the origin of the superior properties of beryllides affecting the hydrogen release at lower temperatures by performing comparison of its properties with pure Be.

- **Goal**: Elucidation of the origin of the superior properties of titanium beryllides (Be$_2$Ti, Be$_{17}$Ti$_2$, Be$_{12}$Ti) and its effect on the hydrogen isotope retention rate

- **Approach**: Ab initio methods

Why do we choose titanium beryllides?

**Beryllium vs. Titanium beryllides**

- high neutron multiplication efficiency
- lower neutron multiplication efficiency
- lower melting point
- higher melting point
- lack of oxidation resistance
- higher oxidation resistance
- higher tritium retention
- lower tritium retention
- higher swelling
- lower swelling
- bad compatibility with the structural material
- good compatibility with the structural materials

---

**Microstructure of Be-Ti composite after extrusion and hot isostatic pressing at 900°C**

The structure along with the main phase of Be$_{12}$Ti contains inclusions of other phases: Be$_2$Ti, Be$_{17}$Ti$_2$, pure Ti and Be

- Be$_2$Ti in the form of a thin layer surrounding the pure Ti phase;
- Be$_{17}$Ti$_2$ in the form of small particles located at the prior Ti phase locations;
- pure Ti and Be phases, which do not dissolve completely

Computational methodology

- Static ab-initio calculations (using VASP)
- Projector augmented wave potential (PAW)
- Generalized gradient approximation (GGA)

- No volume and shape relaxation
- No restrictions on relaxation of atoms

Hydrogen solution energy

\[ E_s = E_{\text{total}}^{\text{Be+H}} - E_{\text{total}}^{\text{Be}} - E_{\text{ref}}^{\text{H}} \]

are the total energies of the simulation cells with and without hydrogen

is the energy of hydrogen atom in \( H_2 \) molecule

Hydrogen binding energy

\[ E_b = E(H + V) - E(V) + E(H_1) - E_{\text{bulk}} \]

are the total energies of the simulation cells with vacancy

and one hydrogen atom and only with a vacancy

\( E(H_1) \) is the energy of hydrogen atom in interstitial position

\( E_{\text{bulk}} \) is the energy of the bulk

Structure and lattice parameters

<table>
<thead>
<tr>
<th>Compound</th>
<th>Lattice Parameters</th>
<th>Be atoms</th>
<th>Ti atoms</th>
<th>Cryst. non-eq. atomic sites</th>
</tr>
</thead>
<tbody>
<tr>
<td>Be(_2)Ti</td>
<td>cubic</td>
<td>32</td>
<td>16</td>
<td>2 (1Be+1Ti)</td>
</tr>
<tr>
<td>Be(_{17})Ti(_2)</td>
<td>hexagonal</td>
<td>34</td>
<td>4</td>
<td>6 (4Be+2Ti)</td>
</tr>
<tr>
<td>Be(_{12})Ti</td>
<td>tetragonal</td>
<td>48</td>
<td>4</td>
<td>4 (3Be+1Ti)</td>
</tr>
</tbody>
</table>

15/09/22
Institute for Applied Materials - Applied Materials Physics

D. Bachurin et al., Ab Initio Study of Hydrogen Behavior in Titanium Beryllides
Hydrogen in interstitial positions

Hydrogen was placed in the center of all possible non-equivalent tetrahedra.

Octahedral (O)  
Basal Tetrahedral (BT)

Positive solution energy (endothermic)  
Negative solution energy (exothermic)

Easier dissolution of hydrogen atoms in the beryllides

Diffusion in Be₁₂Ti

7 energetically nonequivalent hydrogen interstitial sites (A-G) within Be₁₂Ti lattice

Hydrogen path through non-equivalent interstitial sites and 10 jumps between them

Diffusion in Be and Be$_{12}$Ti


Be


Be$_{12}$Ti

7 energetically nonequivalent H interstitial sites (A-G) within Be$_{12}$Ti lattice

The formation energies of Be vacancies in Be_{12}Ti and Be_{17}Ti_{2} are circa two times less than those of Ti vacancies (except for Be_{2}Ti).

The formation energies of Be vacancies in Be_{12}Ti and Be_{17}Ti_{2} are very close to each other.
Binding energy of hydrogen atom in vacancy

- Binding energy of H atom in Ti vacancy is circa 0.9 eV lower than that in Be.
- (Be₂Ti) Binding energies of H atom with Be and Ti vacancies are 0.36 and 0.46 eV, respectively.
- (Be₁₂Ti) Binding energies of H atom with Be vacancies are 0.76, 0.71 and 0.60 eV, with Ti vacancy is 0.34 eV.
- (Be₁₇Ti₂) Binding energies of H atom with Be vacancies are 0.34, 0.36, 0.41 and 0.56 eV, with Ti vacancies are 0.21 and 0.16 eV.
- Binding energy of H atom with Be vacancy is always higher than with Ti vacancy.

---

Binding energy of hydrogen atom in vacancy

- Binding energy of H atom with Be vacancy is significantly higher than that in Ti.
- Some hydrogen atoms located outside a vacancy are trapped by it.
- All hydrogen atoms with negative binding energy were made invisible.
Binding energy of hydrogen atom in vacancy

**Be$_2$Ti**

- Be vacancy
  - $0.11\text{ eV}$
  - $0.46\text{ eV}$
- Ti vacancy
  - $0.03\text{ eV}$
  - $0.12\text{ eV}$
  - $0.36\text{ eV}$
  - $0.25\text{ eV}$

All hydrogen atoms with negative binding energy were made invisible.

Binding energy of H atom with vacancy of Be$_2$Ti is at least circa 0.5 eV lower than that in pure Be.

Some hydrogen atoms located outside a vacancy are trapped by it.

---

**Be$_{17}$Ti$_2$**

- Be7 vacancy
  - $0.25-0.34\text{ eV}$
  - $0.16\text{ eV}$
- Be12 vacancy
  - $0.04\text{ eV}$
  - $0.16\text{ eV}$
- Be19 vacancy
  - $0.36\text{ eV}$
  - $0.09\text{ eV}$
- Be34 vacancy
  - $0.27-0.41\text{ eV}$
  - $0.44-0.56\text{ eV}$
- Ti0 vacancy
  - $0.02-0.21\text{ eV}$
  - $0.02-0.21\text{ eV}$
- Ti2 vacancy
  - $0.16\text{ eV}$

All hydrogen atoms with negative binding energy were made invisible.

Binding energy of H atom in a vacancy of Be$_{17}$Ti$_2$ is at least 0.4 eV lower than that in pure Be.

---

SESSION 5  MODELING & EXPERIMENTAL VALIDATION
Binding energy of hydrogen atom in vacancy

Be_{12}Ti

0.08 eV
0.75 eV
0.04 eV
0.21-0.63 eV
0.15-0.60 eV
0.14-0.34 eV
0.12 eV

Be6 vacancy
Be12 vacancy
Be22 vacancy
Ti0 vacancy

Binding energy of H atom in a vacancy of Be_{12}Ti is at least 0.4 eV lower than that in pure Be.

Binding energy of H atom inside the vacancies of titanium beryllides is always lower than outside them and is in the range of 0.04-0.16 eV.

Conclusions

- Hydrogen solution energy in interstitial positions of titanium beryllides are at least 0.2-0.7 eV lower than that for Be suggesting easier dissolution of hydrogen atoms in the studied beryllides.
- In titanium beryllides hydrogen atoms prefer to occupy the interstitial sites near Ti.
- Formation energy of Be vacancies is circa two times less than the corresponding energy for Ti vacancies in Be_{12}Ti and Be_{17}Ti_2. In Be_{2}Ti the formation energies of Be and Ti vacancies does not differ much from each other.
- Some hydrogen atoms might be trapped by a vacancy without being inside it.
- Hydrogen binding with a monovacancy is 0.4-0.5 eV lower in the studied titanium beryllides, suggesting easier release of hydrogen from beryllides as compared to pure Be.
- The latter conclusion is consistent with the experimental data on deuterium release from Be and Be_{12}Ti after deuterium implantation.
Nanoscale characterization of beryllide materials

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The most recent version of the Helium Cooled Pebble Bed (HCPB) foreseen for the European DEMO blanket considers solid blocks of titanium beryllide as neutron multiplicator material. The advantage of beryllide materials over pure beryllium is their higher operating temperature, higher corrosion resistance, lower swelling, and retention of tritium under neutron irradiation. Understanding the micro- and nanostructure especially after neutron irradiation is of crucial importance for the qualification process of the material.

The focus of this work will lie on the transmission electron microscopy (TEM) characterization of a titanium beryllide/beryllium composite material irradiated at two different temperatures during the HIDOBE neutron irradiation campaign. In particular, the structure and chemistry of the nanosized cavities in the pure beryllium region and the beryllide region was analyzed and is compared to each other. Apart from the cavities, structural defects were observed in the beryllide region that are not known from irradiated pure beryllium.

The presented results can be used for understanding and quantifying for example tritium retention in beryllide materials and to further optimize the material synthesis and the breeding blanked design in general.

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Beryllide materials foreseen as an effective neutron multiplier materials in future fusion reactors.

Questions needed to be answered:
- Understanding the evolving beryllide microstructure under neutron irradiation.
- Understanding of atomic scale mechanisms of tritium trapping and release is necessary for assessment of radioactive inventory.

High-dose irradiation of beryllium (HIDOBE)

- STEM analyses on two Be/Be$_{12}$Ti samples will be presented:
  - $T_{\text{irr}} = 664^\circ\text{C}$
  - $T_{\text{irr}} = 768^\circ\text{C}$
A high-brightness electron source produces a 100-300 keV electron beam with an energy spread of 0.3-1 eV.

Round magnetic lenses focus the beam to a spot size of between 0.05 and 1 nm, which is scanned across an electron-transparent sample.

Signals from scattered electrons and ionized atoms (simultaneously) recorded as the beam is scanned across the sample.

Chemical and bonding information can be obtained by measuring the energy lost by transmitted electrons (e.g., by EELS).

Electron microscopy: Methods & Machines

- Thermofisher Talos F200X
- Acceleration voltage up to 200 kV
- High brightness XFEG: \(1.8 \times 10^9\) A/cm² srad (@200kV)
- Super-X EDS system: 4 windowless SDD detectors in a symmetric design; energy resolution ≤136 eV for Mn-Kα and 10 kcps (output)
- Gatan Enfinium EEL spectrometer capable of acquiring up to 1000 spectra/s; energy resolution up to 0.7 eV
- STEM information limit: 1.2 Å (@ 200 kV)
- STEM point resolution: 1.6 Å (@ 200 kV)
Results of Be$_{12}$Ti sample irradiated at 664°C

- Bright-field imaging of the Be region shows bubbles.
- Selected area diffraction shows that Be is single crystalline.
- Contrast in the bright-field image of Be$_{12}$Ti looks homogeneous. Are there bubbles?
- SAED of Be$_{12}$Ti shows streaking in c-axis direction → stacking faults?
**Be\textsubscript{12}Ti T\textsubscript{irr} = 664°C bubble sizes**

- Bubbles in Be\textsubscript{12}Ti are log normally distributed and have an average size of about 14 nm.
- The bubble number density can be estimated using STEM-EELS (t ≈ 250 nm) → 6.86 \times 10^{21} \text{ m}^{-3}

**Be\textsubscript{12}Ti T\textsubscript{irr} = 664°C STEM imaging**

- Bubbles have a rounded cuboid shape with the round surface along [001] (not clear yet, APT?).
- Bubble surfaces are atomically sharp.
- Wavy contrast is due to distorted lattice along Be\textsubscript{12}Ti c-axis → result of neutron irradiation?
- Streaking in FFT result of (partial) shift of Ti atoms along Be\textsubscript{12}Ti c-axis.
**Be$_{12}$Ti $T_{\text{irr}}$ = 664°C STEM imaging**

- Presence of domain-like regions at Be$_{12}$Ti GB having different disorder inclination.
- "Domains" are separated by a ~5 nm thick domain wall.
- Stacked 30-40 nm sized bubbles at GB.
- "Domain walls" are regions of undisturbed Be$_{12}$Ti lattice.

---

**Be$_{12}$Ti $T_{\text{irr}}$ = 664°C STEM-EELS**

- Homogeneous element distribution.
- STEM-EELS quantification reveals phase composition of Be$_{12}$Ti:
  - Be: 93.8 ± 0.33 at% (nominal: 92.3 at%)
  - Ti: 6.2 ± 0.33 at% (nominal: 7.7 at%)
Results of Be$_{12}$Ti sample irradiated at 768°C

- Be and Be$_{12}$Ti are both crystalline at T$_{irr}$ = 768°C.
- Be/Be$_{12}$Ti interfaces are faceted.
- No visible bubbles in Be$_{12}$Ti at this magnification.
- Large bubbles in Be region and at the Be side of the Be/Be$_{12}$Ti interface.
**STEM-EELS Be$_{12}$Ti $T_{irr} = 768 ^\circ$C**

- Bubbles in [101] zone-axis (ZA) orientation have atomically sharp interfaces at (020).
- Bubble sizes comparable to $T_{irr} = 668 ^\circ$C sample.
- No visible atomic disturbance in [101] ZA orientation.
- Disturbed areas of 2-5 nm in height perpendicular to c-axis in [210] ZA visible.
- In [111] ZA two regions observed.
  - Region 1: Normal [111]-type HAADF contrast
  - In region 2 Ti atoms are shifted into the center of the (1-10) planes.

**STEM-EDX Be$_{12}$Ti $T_{irr} = 768 ^\circ$C**

- Bubble edges in Be$_{12}$Ti covered with a 1.5 nm thin Si layer.
- Be$_{12}$Ti less prone to oxidation compared to pure Be (→ open bubbles).
- No other foreign elements (Fe, U…) found as in pure Be.

*Pure Be: Various phases in EDX map (b) are colored as follows: Al-Fe-Be (red), Mg-Si (blue), Cr-Ti (green) and U-Fe (white).*
**STEM-EELS Be\textsubscript{12}Ti $T_{\text{irr}} = 768^\circ$C**

**Reminder:** bubble contents in pure Be:

- $^3$H is preferentially located on bubble c-axis surfaces.
- He is in the bubble center.

**Situation in Be\textsubscript{12}Ti less clear than in pure Be due to experimental limitations.**

Further reading:
4. Zimber & Vladimirov - The role of grain boundaries and denuded zones for tritium retention in high-dose neutron irradiated beryllium, *IJUCMAT* 541 (2022), 153855.
Summary

- A thorough TEM investigation of Be$_{12}$Ti neutron irradiated at 2 different temperatures was carried out.
- Bubbles generated by neutron irradiation are roughly 10x smaller in Be$_{12}$Ti than in pure Be.
- Extended structural defects (domain-like regions) observed in Be$_{12}$Ti besides bubbles for both irradiation temperatures.
- In both materials – Be and Be$_{12}$Ti – $^3$H is located at bubble surfaces and He is in the bubble center. In case of Be$_{12}$Ti monochromated, C$_S$-corrected (S)TEM experiments can yield better datasets.
- STEM-EDX shows the presence of 1.5 nm thin Si layers on bubble surfaces.
- STEM-EELS quantification reveals phase composition of Be$_{12}$Ti close to the nominal values.

Thank you for your attention!
SESSION 6

Mechanical properties & irradiation damage
Mechanical compression behaviors and microstructure change under He ion irradiation of single phase Be and binary Be$_{12}$Ti pebbles

Pingping Liu$^1$*, Qian Zhan$^1$, Wen Hu$^1$, Yumei Jia$^1$, Farong Wan$^1$

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The world urgently needs a carbon-free, safe, clean and limitless energy source. Fusion energy has the potential to meet this need. International thermonuclear fusion experimental reactor (ITER) and future commercial power demonstration reactor (DEMO) were designed and developed by scientists around the world. However, one key problem of "tritium fuel self-sustaining" needs to be solved firstly. Thus, tritium breeding blanket module (TBM) was developed. Beryllium (Be) pebbles with a diameter of 1 mm are planned to be used as a neutron multiplier in the helium-cooled ceramic breeder (HCCB) test TBM of ITER, which is also the primary option of the Chinese TBM program. Meanwhile, beryllium intermetallic compounds (beryllides) such as Be$_{12}$Ti are the most promising advanced neutron multipliers in advanced DEMO fusion reactors because of high melting point and high stability at high temperatures.

In this study, in the prospect of establishing a database of advanced neutron multipliers for fusion reactor, the preparation, mechanical properties test and irradiation damage of Be and beryllides pebbles were carried out. The process of producing pure Be and Be-Ti alloy pebbles can be roughly divided into two stages: fabrication of Be and beryllide electrode rods and granulation of Be and beryllide pebbles. The Be-Ti beryllides synthesized by hot isostatic pressing (HIP) with a pressure of 230 MPa at 1073 K for 2 h. The Be and Be$_{12}$Ti granulation process by rotating electrode process (REP). Then, surface analysis, mechanical compression and irradiation were performed to evaluate surface microstructure, mechanical properties and irradiation resistance.

Be pebbles with diameter of 1 mm and Be$_{12}$Ti pebbles with diameter of 0.7 mm were successfully fabricated by combining HIP and REP methods. According to the XRD results, the phase composition of the Be pebbles was identified as Be with little BeO. The phase composition of the as-granulated Be-7.7 at.%Ti pebbles was identified as Be$_{12}$Ti with little Be and BeO. The AFM and SEM results revealed that Be and Be$_{12}$Ti pebble were well shaped with small surface fluctuates. During mechanical compression tests, Be pebbles exhibited very good ductility (no fracture at 50% deformation), which is better than that of Be$_{12}$Ti pebbles. Rupture of Be$_{12}$Ti pebbles occurred at the deformation beyond 10%. After high dose helium ion irradiation, the surface blistering and internal damage structure were examined and analysed by SEM and TEM, which may provide experimental basis for good understanding of irradiation damage mechanism and design optimization of advanced neutron multipliers for the fusion reactor.
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Fabrication and surface irradiation damage of advanced neutron multiplier for fusion blankets

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School of Materials Science and Engineering, University of Science and Technology Beijing, China

2022.09.15

Outline

• Background: Fusion T-self & Neutron multiplier

• Fabrication of Be, Be$_{12}$Ti and Be$_{12}$W pebble

• Mechanical compression of neutron multiplier beryllium and beryllide pebble

• Surface irr. damage of Be, Be$_{12}$Ti and Be$_{12}$W pebble

• Summary
Background

*Part of the schematic diagram from Y.X. Wan., 2020.

### Background

#### Some parameters of Blanket of different fusion reactor

<table>
<thead>
<tr>
<th>Items</th>
<th>ITER</th>
<th>CFETR</th>
<th>DEMO Reactor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>150–350</td>
<td>&lt;650</td>
<td>600–900</td>
</tr>
<tr>
<td>He (appm)</td>
<td>~3000</td>
<td>~10 000</td>
<td>~20 000</td>
</tr>
<tr>
<td>Irr. damage (dpa)</td>
<td>10</td>
<td>~30</td>
<td>50</td>
</tr>
<tr>
<td>Be pebbles</td>
<td>√</td>
<td>?</td>
<td>×</td>
</tr>
</tbody>
</table>

**Neutron Multiplier of DEMO**

--High beryllium content  
--Low activation  
--High melting point

<table>
<thead>
<tr>
<th>Materials</th>
<th>Be</th>
<th>Be$_{12}$Ti</th>
<th>Be$_{12}$W</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting point</td>
<td>1280 °C</td>
<td>~1550 °C</td>
<td>~1750 °C</td>
</tr>
<tr>
<td>Be content(at.)</td>
<td>100%</td>
<td>92.3%</td>
<td>92.3%</td>
</tr>
</tbody>
</table>

### Background

**Initial Motivation:**

1. Fabrication of high-melting-point neutron multiplier pebbles.
2. Evaluation of irradiation resistance of the pebbles.

D-T fusion produces an extremely energetic (14.1-MeV) neutron, which is potentially useful for breeding more tritium but also creates challenges for the materials (Irradiation damage).
Outline

- Background: Fusion T-self & Neutron multiplier
- **Fabrication of Be, Be$_{12}$Ti and Be$_{12}$W pebble**
  - Mechanical compression of neutron multiplier beryllium and beryllide pebble
  - Surface irradiation damage of Be and Be$_{12}$Ti/W pebble
- Summary

**Fabrication of neutron multiplier pebbles**

Flowchart for the fabrication of neutron multiplier.
Fabrication scheme of the beryllium rod by hot isostatic pressing.

**Fabrication of neutron multiplier pebbles**

Rotating electrode process:

1. Rotating of beryllium electrode
2. Discharge between beryllium and W electrode
3. Solidification to spherical particles

*Part of the schematic diagram from K. Tsuchiya (JAEA), 2005.*
Fabrication of neutron multiplier pebbles

Be pebbles

Be-Ti pebbles

Be-W pebbles

d \approx 1 \text{ mm}
d \approx 0.7 \text{ mm}

Surface microstructure of Be and BeTi pebble produced by REP.
## Table 1. Chemical compositions of the Be, BeTi and BeW pebbles (wt.%).

<table>
<thead>
<tr>
<th>Samples</th>
<th>Al</th>
<th>Fe</th>
<th>Mg</th>
<th>O</th>
<th>W</th>
<th>Ti</th>
<th>Be</th>
</tr>
</thead>
<tbody>
<tr>
<td>Be pebble</td>
<td>0.02</td>
<td>0.088</td>
<td>&lt;0.001</td>
<td>0.986</td>
<td>—</td>
<td>—</td>
<td>98.1</td>
</tr>
<tr>
<td>BeTi pebble</td>
<td>0.06</td>
<td>0.19</td>
<td>&lt;0.010</td>
<td>0.081</td>
<td>—</td>
<td>28.27</td>
<td>71.27</td>
</tr>
<tr>
<td>BeW pebble</td>
<td>0.18</td>
<td>0.25</td>
<td>&lt;0.039</td>
<td>0.721</td>
<td>62.3</td>
<td>—</td>
<td>36.51</td>
</tr>
</tbody>
</table>

X-ray diffraction spectrum of the Be, BeTi and BeW pebbles.

Be, Be_{12}Ti and Be_{12}W pebbles were fabricated.


---

## Outline

- **Background:** Fusion T-self & Neutron multiplier
- **Fabrication of Be, Be_{12}Ti and Be_{12}W pebble**
- **Mechanical compression of neutron multiplier beryllium and beryllide pebble**
- **Surface irradiation damage of Be and Be_{12}Ti/W pebble**
- **Summary**
The scheme of loading of a pebble (Be or BeTi) during the compression test.

Load curves in compression tests of Be (a) and BeTi (b) pebbles with load speed of 0.2 mm/min.

(0.05 mm, 45 N), (0.1 mm, 88 N), (0.5 mm, 316 N) (0.035 mm, 76 N), (0.07 mm, 174 N)
Compression of Be and Be$_{12}$Ti pebbles


[*]https://en.wikipedia.org/wiki/Contact_mechanics

Compression of Be and Be$_{12}$Ti pebbles

(a) Compressive stress distribution (GPa)

(b) Compressive stress distribution (GPa)

Be Pebbles

Be$_{12}$Ti Pebbles

Load (N) vs. Displacement (mm)

Be Pebble

Experimental

Calculated

Be$_{12}$Ti Pebble

Experimental

Calculated

(c) Load (N) vs. $(a*d)*10^{-2}$ (mm$^2$)

$P = \frac{4}{3} \times E' \times (a \times d)$

(d) Pressure $p_0$ vs. Distance from the center $r$ (mm)

$p_{max}$ Be$_{12}$Ti pebble

$p_{max}$ Be pebble

$p_{max}$ BeTi pebble

$p_{max} = \frac{1}{\pi} \left( \frac{d p_0 E^2}{2} \right)^{1/3}$
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- Summary

He ion irradiation (30 keV, $10^{18}$ ion/cm$^2$)

Helium content profile in Be (a) and Be$_{12}$Ti (b) pebbles calculated by SRIM.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Be</th>
<th>Be$_{12}$Ti</th>
<th>Be$_{12}$W</th>
</tr>
</thead>
<tbody>
<tr>
<td>He ion peak</td>
<td>280 nm</td>
<td>290 nm</td>
<td>300 nm</td>
</tr>
<tr>
<td>He content</td>
<td>77.3 at.%</td>
<td>75.9 at.%</td>
<td>57.5 at.%</td>
</tr>
</tbody>
</table>
P. Liu et al., Mechanical Compression & Microstructure Change under He Irradiation

Surface of Be pebble after He ion irradiation

(a) Unirradiated Be pebble  
(b) Surface of Be pebble after He ion irradiation  
(c) Irradiated Be pebble  
(d) Surface Blisters

Surface of Be$_{12}$Ti pebble after He ion irradiation

(a) Unirradiated Be$\text{Ti}$ pebble  
(b) Surface of Be$_{12}$Ti pebble after He ion irradiation  
(c) Irradiated Be$\text{Ti}$ pebble  
(d) Surface Blisters
Surface of Be$_{12}$W pebble after He ion irradiation

(a) Unirradiated BeW
(b) Irradiated BeW

Blister mainly in the W-rich phase

Unirradiated BeW
Irradiated BeW

Surface swelling of Be, Be$_{12}$Ti and Be$_{12}$W pebble

(a) Blister of Be pebbles
\[ d_{av}=5.45 \times 10^{-6} \text{ m} \]
\[ \rho=1.54 \times 10^{10} / \text{m}^2 \]

(b) Blister of BeTi pebbles
\[ d_{av}=1.1 \times 10^{-6} \text{ m} \]
\[ \rho=8.62 \times 10^{10} / \text{m}^2 \]

(d) Second blister on Be-W alloy
\[ d_{av}=7.8 \times 10^{-6} \text{ m} \]
\[ \rho=1.28 \times 10^{13} / \text{m}^2 \]
Surface swelling of Be and Be$_{12}$Ti pebble

Surface swelling rate:
- Be: 35.91%
- Be-Ti: 8.19%
- Be-W: ~1.21%

Microstructure of Be before irradiation

The grain size of the metal fall in the range from several microns to several hundred microns.

“Clean”--No damage

Few impurity particles in the marked beryllium matrix were observed as shown in the morphology. However, some precipitates were observed near the grain boundary.
Large bubbles and dislocation loops of high density were induced by $1 \times 10^{18}$/cm$^2$ He ion irradiation.

Summary

1) Be, BeTi and BeW pebbles could be fabricated by REP.

2) Compression properties of Be and Be12Ti pebbles were evaluated by mechanical compression.
   ----Beryllium pebbles displayed high ductility (50% deformation without fracture), which is better than that of beryllide pebbles.

3) Be, BeTi and BeW were irradiated by high dose He-ion beam.
   ----The surface swelling-resistance of BeTi and BeW is better than beryllium.
Creep of beryllium pebbles after neutron irradiation to 6000 appm helium production

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Beryllium pebbles with 1 mm diameter are the reference neutron multiplier material in the Helium Cooled Pebble Bed (HCPB) blanket of ITER. High energy fusion neutrons cause swelling of the beryllium pebbles at the HCPB operation temperatures to 923 K. The radiation-induced swelling of beryllium as well as different thermal expansions of the beryllium pebbles and the structural material can cause the high thermal stresses in the pebble bed. Thermal creep of the pebbles should reduce the stresses because the relaxation. Neutron irradiation leads to degradation of mechanical properties, what expresses in the hardening and the embrittlement. This radiation effect hinders the effect of the relaxation.

In this study, creep properties of beryllium pebbles with 1 mm diameter produced by Rotating Electrode Method (REM) at NGK, Japan were studied. These beryllium pebbles were irradiated in the HFR, Petten, the Netherlands, at temperatures of 643, 723, 833, 923 K to 6000 appm helium production. The creep tests of individual pebbles were performed at temperatures which were equal to the irradiation temperatures by using of three different loadings per each temperature. For two lowest irradiation temperatures of 643 and 723 K, no creep effect was observed. The radiation hardening only occurs that manifests itself in significant reduction of the pebble deformation under loading. At higher irradiation temperatures of 833 and 923 K, the creep rates have significant values. The creep rates strongly depend on the testing temperatures and the loadings. At high irradiation temperatures the ability of beryllium pebbles to the significant deformation under applied loadings should provide the complete relaxation of the internal stresses in the beryllium pebbles.

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Creep of beryllium pebbles after neutron irradiation to 6000 appm helium production

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Outline

Materials, pebbles, creep testing at FML
HIDOBE-02: irradiation of unconstrained (free filled) Be pebbles and constrained Be pebble beds
Optical microstructure of irradiated Be pebbles
Creep behaviour of irradiated unconstrained and constrained Be pebbles
Conclusions
He-cooled Pebble Bed for ITER

Li₄SO₄ pebble bed
Be pebble bed

$^{9}$Be + n⁰ → 2n⁰ + $^{8}$Be
$^{6}$Li + n⁰ → $^{4}$He + $^{3}$H

2H + 3H → $^{4}$He + n⁰ + 17.5 MeV

Neutron multiplier material in HCPB concept
Beryllides (e.g. Be₁₂Ti)
lower swelling
lower $^3$H retention
higher oxidation resistance
higher strength

HIDOBE-02 experiment at HFR, Petten

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

V. Chakin et al., Creep of Beryllium Pebbles after Neutron Irradiation
HIDOBE-02 experiment at HFR, Petten

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

In irradiation conditions relevant for DEMO blanket, the Be and BeTi materials were studied to understand their microstructure evolution and tritium release/retention. The experiment was conducted from 2005 to 2011, involving 48 reactor cycles and 1247 Full Power Days, achieving ~30% of DEMO EOL Helium production.

Irradiation parameters of Be pebbles in HIDOBE-02 campaign

<table>
<thead>
<tr>
<th>Be pebble with Ø1 mm</th>
<th>( T_{\text{irr}}, K )</th>
<th>( F, \times 10^{26}, \text{m}^2, \text{E} &gt; 1 \text{MeV} )</th>
<th>D, dpa</th>
<th>(^4\text{He}, \text{appm} )</th>
<th>(^3\text{H}, \text{appm} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unconstrained (free filled)</td>
<td>644</td>
<td>1.06</td>
<td>21</td>
<td>3632</td>
<td>367</td>
</tr>
<tr>
<td>716</td>
<td>1.43</td>
<td>29</td>
<td>4751</td>
<td>502</td>
<td></td>
</tr>
<tr>
<td>832</td>
<td>1.68</td>
<td>34</td>
<td>5524</td>
<td>596</td>
<td></td>
</tr>
<tr>
<td>919</td>
<td>1.81</td>
<td>37</td>
<td>5925</td>
<td>644</td>
<td></td>
</tr>
<tr>
<td>Constrained</td>
<td>660</td>
<td>1.06</td>
<td>21</td>
<td>3632</td>
<td>367</td>
</tr>
<tr>
<td>754</td>
<td>1.43</td>
<td>29</td>
<td>4751</td>
<td>502</td>
<td></td>
</tr>
<tr>
<td>874</td>
<td>1.68</td>
<td>34</td>
<td>5524</td>
<td>596</td>
<td></td>
</tr>
<tr>
<td>958</td>
<td>1.81</td>
<td>37</td>
<td>5925</td>
<td>644</td>
<td></td>
</tr>
</tbody>
</table>
### Chemical composition of Be pebbles with Ø 1 mm

<table>
<thead>
<tr>
<th>Element</th>
<th>Content, wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Be</td>
<td>99.5</td>
</tr>
<tr>
<td>BeO</td>
<td>0.36</td>
</tr>
<tr>
<td>Fe</td>
<td>0.094</td>
</tr>
<tr>
<td>Al</td>
<td>0.048</td>
</tr>
<tr>
<td>Mg</td>
<td>0.024</td>
</tr>
<tr>
<td>Si</td>
<td>0.029</td>
</tr>
<tr>
<td>U</td>
<td>&lt;0.01</td>
</tr>
</tbody>
</table>

### Placement of Ø 1 mm Be pebbles in HIDOBE-02

Unconstrained Be pebbles are placed in capsule with Ø 3.2 mm. The pebbles were filled free in the capsule.

Constrained Be pebble beds are placed in capsule with Ø 14.9 mm. It was filled by pebbles and then, screwed on with screw cap with torque wrench. In particular, after contact of the cap with the pebbles, another forced turn of the cap along the thread was done. In this position, the cap was spot welded to the capsule. In this way, additional internal stresses were created in the pebbles.

This allows to simulate internal state of the pebbles swelled after irradiation to higher neutron dose than that in HIDOBE-02 experiment.
Pebble loading scheme in creep machine

1 – pebble
2 – bottom
3 – loading piston

$P$ – constant during creep test

$T_{\text{test}} = T_{\text{irr}}$ for each testing pebble

t = 80 h

Creep testing parameters and shortly about results

<table>
<thead>
<tr>
<th>State</th>
<th>$T_{\text{irr}}, K$</th>
<th>$T_{\text{test}}, K$</th>
<th>$P$, N</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unconstrained</td>
<td>644</td>
<td>643</td>
<td>100</td>
<td>no deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>110</td>
<td>cracks</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>120</td>
<td>cracks</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>150</td>
<td>cracks</td>
</tr>
<tr>
<td></td>
<td>716</td>
<td>723</td>
<td>100</td>
<td>no deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>110</td>
<td>cracks</td>
</tr>
<tr>
<td></td>
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<td>120</td>
<td>cracks</td>
</tr>
<tr>
<td></td>
<td>832</td>
<td>833</td>
<td>55</td>
<td>no deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>60</td>
<td>deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>70</td>
<td>deformation</td>
</tr>
<tr>
<td></td>
<td>919</td>
<td>923</td>
<td>15</td>
<td>no deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>20</td>
<td>no deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>25</td>
<td>no deformation</td>
</tr>
<tr>
<td>Constrained</td>
<td>660</td>
<td>643</td>
<td>35</td>
<td>deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>50</td>
<td>deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>70</td>
<td>deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>100</td>
<td>cracks</td>
</tr>
<tr>
<td></td>
<td>754</td>
<td>723</td>
<td>70</td>
<td>deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>100</td>
<td>deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>120</td>
<td>deformation</td>
</tr>
<tr>
<td></td>
<td>874</td>
<td>833</td>
<td>35</td>
<td>deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>50</td>
<td>deformation</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>70</td>
<td>cracks</td>
</tr>
<tr>
<td></td>
<td>958</td>
<td>923</td>
<td>-</td>
<td>not dismantled</td>
</tr>
</tbody>
</table>

“No deformation” means that the load is not enough to produce deformation or destruction of the pebble. “Cracks” means that cracks appeared as a result of loading. “Deformation” means that as a result of loading, the pebble deformed through creep. Therefore, only 10 diagrams (by red) from 23 diagrams of performed tests include really creep behavior therefore, they can be processed. 8 diagrams from 10 “red” diagrams (“deformation”) are constrained pebbles. This means that constrained pebbles have comparatively better creep behavior.
Examples of “no deformation” case after creep tests

- Unconstr., $T_{\text{test}} = 643 \text{ K}, P = 100 \text{ N}$
- Unconstr., $T_{\text{test}} = 833 \text{ K}, P = 80 \text{ N}$
- Unconstr., $T_{\text{test}} = 923 \text{ K}, P = 25 \text{ N}$

“No deformation” case was only for unconstrained Be pebbles (6 creep tests).
Reason for “no deformation” case is that was not enough load to deform or to break the tested pebble.

Examples of “cracks” case after creep tests

- Unconstr., $T_{\text{test}} = 643 \text{ K}, P = 120 \text{ N}$
- Unconstr., $T_{\text{test}} = 723 \text{ K}, P = 120 \text{ N}$
- Constr., $T_{\text{test}} = 643 \text{ K}, P = 100 \text{ N}$
- Constr., $T_{\text{test}} = 833 \text{ K}, P = 70 \text{ N}$

5 “cracks” cases are for unconstrained pebbles, 2 cases are for constrained pebbles.
Examples of “deformation” case

Unconstr., $T_{test} = 833 \text{ K}, P = 60 \text{ N}$

Unconstr., $T_{test} = 833 \text{ K}, P = 70 \text{ N}$

Constr., $T_{test} = 643 \text{ K}, P = 70 \text{ N}$

Constr., $T_{test} = 833 \text{ K}, P = 50 \text{ N}$

2 “deformation” cases are for unconstrained pebbles, 8 cases are for constrained pebbles.

Parameters of indentations on irradiated Be pebbles after creep testing

- $D$ is diameter of pebble
- $d$ is indentation diameter as a result of the creep process
- $h$ is distance between opposite indentations

$$\sigma = \frac{P}{S} = 4P/\pi d^2,$$ where $\sigma$ is internal stress in the pebble under loading $P$, $S$ is area of indentation with diameter of $d$.
There is a fundamental difference of microstructure evolution on irradiation temperature. No visible pores are after irradiation at two lowest $T_{irr}$ (644 and 716 K). Many big pores are after irradiation at two highest $T_{irr}$ (832 and 919 K).

Also, no pores but constrained pebbles have much more sub-grains and twins compared to unconstrained pebbles.
**Microstructure of constrained Be pebbles after irradiation at 874 K**

There are many big pores and developed sub-grain microstructure.

---

**Failure load on loading of irradiated Be pebbles at \( T_{\text{test}} = 723 \) and 833 K**

<table>
<thead>
<tr>
<th>( T_{\text{test}} )</th>
<th>( \times ) ( T_m )</th>
</tr>
</thead>
<tbody>
<tr>
<td>643 K</td>
<td>0.412 ( T_m )</td>
</tr>
<tr>
<td>723 K</td>
<td>0.463 ( T_m )</td>
</tr>
<tr>
<td>833 K</td>
<td>0.534 ( T_m )</td>
</tr>
<tr>
<td>923 K</td>
<td>0.592 ( T_m )</td>
</tr>
</tbody>
</table>

Creep starts at \( T_{\text{test}} > 0.5T_m \)

At 723 K is no creep: failure load of unconstrained pebbles is higher than that of constrained pebbles, and failure occurs at higher loading.

At 833 K is creep: failure load of unconstrained pebbles is lower than that for constrained pebbles, and failure occurs at lower loading.
Examples of creep curves for unconstrained and constrained irradiated Be pebbles

Both samples were tested at the same temperature of 833 K.
Shape of both curves is typical for creep tests.
Deformation for constrained pebble is significantly higher to unconstrained pebbles even though the loading was relatively less for constrained pebble (50 to 70 N).

Creep rate determination in second stage

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Creep rate determination in second stage

We carry out a linear approximation at second stage using experimental creep curve.

Steady-state strain-rate \( \dot{\varepsilon} = \frac{(l_2 - l_1)}{(t_2 - t_1) \times h} \),
where \( l_1 \) and \( l_2 \) are deformation at beginning and end of selected segment of creep curve,
\( t_1 \) and \( t_2 \) are time to reach beginning and end of selected deformation segment, \( h \) is distance
between opposite indentations on tested pebble.

Classical creep curve has three stages

We carry out a linear approximation at second stage using experimental creep curve.

Steady-state strain-rate \( \dot{\varepsilon} = \frac{(l_2 - l_1)}{(t_2 - t_1) \times h} \),
where \( l_1 \) and \( l_2 \) are deformation at beginning and end of selected segment of creep curve,
\( t_1 \) and \( t_2 \) are time to reach beginning and end of selected deformation segment, \( h \) is distance
between opposite indentations on tested pebble.
**Creep rate determination in second stage**

Classical creep curve has three stages

Experimental creep curve has only two incomplete stages

We carry out a linear approximation at second stage using experimental creep curve.

Steady-state strain-rate \( \dot{\varepsilon} = \frac{(l_2 - l_1)}{(t_2 - t_1) \times h} \),

where \( l_1 \) and \( l_2 \) are deformation at beginning and end of selected segment of creep curve,

\( t_1 \) and \( t_2 \) are time to reach beginning and end of selected deformation segment, \( h \) is distance between opposite indentations on tested pebble.

---

**Dependence of steady-state strain-rate \( \dot{\varepsilon} \) to stress \( \sigma \)**

V. Chakin et al., Creep of Beryllium Pebbles after Neutron Irradiation
Creep behavior of constrained pebbles is logical. Creep rate at lowest temperature 660 K is similar to that at 754 K but it has higher applied stress. Creep rate at highest temperature 874 K is higher than that at lower temperatures. Creep behavior of unconstrained pebbles is unclear because it was obtained a limited number of successful creep tests.
Institute for Applied Materials – Applied Materials Physics

Comparison of behavior of constrained pebble beds after irradiation at low- and high-temperatures

Constrained pebble bed, if it concerns level of internal stresses in the pebbles, means state of the pebble bed after high-dose irradiation because high internal stresses were created in the pebbles already before irradiation.

Low-temperature irradiation (<0.5T_m) – no pores, not high swelling, formation of dislocation sub-grain microstructure but no creep deformation therefore, defragmentation of pebbles occurs.

High-temperature irradiation (>0.5T_m) – many big pores and high swelling, but sub-grains and creep deformation are available therefore, stress relaxation exists resulting comparatively less pebble defragmentation comparing to low-temperature irradiation.

DC

T_{irr} = 660 K (0.423 T_m) – no creep
T_{irr} = 874 K (0.567 T_m) – by creep

Conclusions

Creep tests of Ø 1 mm Be pebbles irradiated to 6000 appm He production show difference of creep behavior of unconstrained (free filled) and constrained pebbles.

There are points:
➢ 10 from 23 performed tests include really creep behavior as “deformation” case. And 8 from 10 diagrams were obtained on constrained pebbles.
➢ There is transition to creep behavior between 723 K (0.463 T_m) and 833 K (0.534 T_m). Constrained pebbles at 833 K have comparatively higher failure load than that for unconstrained pebbles.
➢ Optical metallography shows very developed sub-gran microstructure in constrained pebbles. Unconstrained pebbles have practically no sub-grains.
➢ Concerning calculation of dependence of steady-state strain-rate to stress for constrained pebbles, it is logical behavior. For unconstrained pebbles, it was performed not enough successful creep tests (there were mainly “no deformation” or “cracks” cases).

This means that constrained pebbles have comparatively better creep behavior than unconstrained pebbles especially for irradiation at higher temperatures than 0.5T_m.

V. Chakin et al., Creep of Beryllium Pebbles after Neutron Irradiation
SESSION 6  MECHANICAL PROPERTIES & IRRADIATION DAMAGE

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

1999-2022 BeWS-4 BeWS-15
Thank you for your attention!

See you again on BeWS-16?
Thermo-mechanical behavior of titanium beryllide pebble beds at elevated temperatures

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The thermomechanical behavior of titanium beryllide pebble beds was investigated experimentally at temperatures between 200 and 500°C in helium atmosphere at atmospheric pressure. The pebbles consist of a mixture of TiBe₁₂ and Ti₂Be₁₇ titanate beryllide phases and a small residual amount of Be phase, denominated as Be-7.7Ti.

Like previous experiments at ambient temperature [1], the pebble beds were compressed uniaxially up to 4.5MPa and the effective thermal conductivity k was measured using the hot wire technique.

Compared to ambient temperature, the stress-strain curves do not differ significantly in investigated temperature range. Because the thermal conductivity of solid TiBe₁₂ is fairly constant in a wide temperature range [2], k increases moderately with increasing temperature because of the increasing thermal conductivity of helium.

Compared to beryllium pebble beds, the k of the Be-7.7Ti pebble beds increases again much lesser because of the significantly smaller thermal conductivity of the solid material and the mechanically harder behavior resulting in smaller contact surfaces.


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Thermo-mechanical behavior of titanium beryllide pebble beds at elevated temperatures

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Outline

Introduction
- Modelling parameters of the thermo-mechanical behaviour of pebble beds
- Previous investigations at ambient temperature

Experimental set-up

Results
- A) Uniaxial Stress-Strain dependence
- B) Effective thermal conductivity of pebble beds
- C) Thermal creep

Discussion/Conclusions
General Remarks

This paper is an extension of recent investigations at ambient temperature $T_a$ [1]. In all experiments, a simple experimental facility was used, therefore, the experimental results should be considered as screening exps.

The beryllium-titanate pebbles ($d=0.8-1.2\text{mm}$) were produced with the stoichiometric composition of Be and 7.7at% Ti, ideally resulting in single phase Be$_{12}$Ti pebbles. However, it was found that the used pebbles have also a Be$_{13}$Ti$_2$ and a Be phase, for details, see [1]. Therefore, the denomination Be-7.7Ti was chosen.

Main Parameters for the modelling of the thermo-mechanical behaviour of blanket pebble beds, pbs

BOL:
- stress-strain relationship $\sigma = f(\varepsilon, T, \ldots)$,
- pebble bed effective thermal conductivity $k_{\text{eff}} = f(\varepsilon, T, \ldots)$,
- thermal creep $\varepsilon_{\text{cr}}$.

EOL: Irradiation effects,
- Same parameters as above, see e.g. [2] analyses of Be-based materials from the ADOBE exps.

Previous investigations
Exps at ambient temperature, $T_a$, [1]


Experimental set-up

Uniaxial Compression Test facility combined with a Hot Wire, HW, set-up.

Set-up used for exps at Ta [1]. For details of the HW-system, see also [1].

Now: Use of the container with additional heating on cylindrical part and the heating system of HECOP facility [3]

Parameter range:
- max. pressure (uniaxial stress): 6MPa
- max. T: 500°C

Results: A) $\sigma$–$\epsilon$ relationship

- Besides the first stress increase curve, the subsequent stress release and following cycles are also blanket relevant.
- As already observed for Be pbs [3], the $\sigma$–$\epsilon$ dependence of 1st $\sigma$-increase curve is negligibly dependent on T as long as thermal creep is of no concern.
- The cycling curves curves indicate an elastic pebble material.
Results: B) Thermal conductivity of solid materials, $k_s$, and helium, $k_{he}$

Beryllide pebbles, are superior to Be pebbles in many aspects (irradiation, compatibility, oxidation...)

However, there is a significant drawback: the thermal conductivity of the solid materials, $k_s$, is significantly smaller than that of Be [2,4,5].

Because the present Be-7.7Ti pebbles consist of several phases, the exact $k_s$ value is not known. In the following, a constant value of $k_s = 40 \text{ W/(mK)}$ is assumed.

The thermal conductivity of helium increases with T.

---

B) Non-compressed pebbles, $k_{eff-0} = f(T)$

- Pebble bed thermal conductivities much smaller than solid material conductivities.
- $k_{eff-0}$ for Be-7.7Ti at 600°C about 10% lower than that of Be (although $k_s$-Be7.7 ≈ 0.5$k_s$-Be).
- However: for Be pebbles, $k_{eff}$ increases drastically with compression (uniaxial stress); how about Be-7.7Ti?
For Be pbs, \( k_{\text{eff}} \) increases drastically with \( \sigma \) in contrast to other pb materials. What is the reason for this? 
- ductility? 
- solid material thermal conductivity \( k_s \)?

Answer: both; assuming the same increase of contact surfaces as for Be pbs, the measured data for non-ductile materials are below the calculated curves.

In blankets, strain is the prime parameter. As observed for Be pbs [3], a fairly linear dependence is obtained.
B) Temperature dependence of $k_{eff}$ for first stress increase

With increasing temperature, the data are generally below the curve for Ta. The reason is that $k_{eff}-0$ increases with $T$ because of the increasing helium thermal conductivity.

Additionally, the contribution of contact surfaces decreases also with increasing gas thermal conductivity.

B) Temperature dependence of $k_{eff}$ during mech. cycling

For ductile pb materials, e.g. Be, $k_{eff}$ does not decrease significantly with stress decrease in a wide $\sigma$-range.

For Be-7.7Ti, this effect is not expressed, indicating again the elastic pebble behavior.
Thermal creep increases contact surfaces and with this increases effectively $k_{eff}$.

At 500°C, thermal creep is significant for Be pbs [6] and negligible for Be7.7Ti pbs. (Corresponding $k_{eff}$ results cannot be shown because of HW failure).

Discussions/Conclusions

- The thermo-mechanical behavior of beryllium titanate Be-7.7Ti pbs was investigated at temperatures up to 500°C.
- Compared to Be pbs, Be-7.7Ti pbs have a considerably smaller $k_{eff}$ during compression because of the smaller ductility and the smaller solid material conductivity.
- Thermal creep effects, causing also an increase of contact surfaces, are marginal for Be-7.7Ti in the investigated temperature range.
- $k_{eff}$ can be improved by using binary pbs instead of monosized pbs [7].
- Beryllide pbs can be superior to Be pbs, if mixed breeder and neutron multiplyer pbs are a viable option, requiring irradiation exps.
- Recent analyses of the HIDOBE irradiation programme showed much smaller swelling of Be5Ti and Be7Ti disks compared to Be disks, and smaller swelling rates for Be pbeles compared to Be disks. Again, irradiation exps with non-constrained/ constrained beryllide pbeles are required.
- First thermal creep results on irradiated Be pbeles were presented by [8].
APPENDIX A

Workshop photos
Figure 1: Group picture of the BeWS-15 participants (courtesy of J. Reimann). From left to right:
Figure 2: Group picture of the BeWS-15 participants without J. Reimann, who was taking this photo (courtesy of J. Reimann)
Figure 3: Workshop dinner in Hoepfner Schalander Hall
Figure 4: Awarding the Prof. Mario Dalle Donne Award to Aniceto Goraieb during the Workshop dinner in Hoepfner Schalander Hall
Figure 5: Awarding the Prof. Glen Longhurst Award to Christopher Dorn during the Workshop dinner in Hoepfner Schalander Hall
Figure 6. Guided tour of the Hoepfner Burghof Brewery
Figure 7: Group photo (from left to right): R. Gaisin, K. Zenkov, S. Udartsev, Ye. Frants, P. Vladimirov
Appendix A  WORKSHOP PHOTOS

Figure 8: Group photo (from left to right): P. Vladimirov, S. Kulsenko, R. Gasin
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<td>Milan Zmitko (online)</td>
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<td>Overview of R&amp;D activities on Neutron Multipliers in QST</td>
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<td>Creep of beryllium pebbles after neutron irradiation to 6000 appm helium production</td>
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7:00 PM      Conference Dinner at Hoepfner Burghof Restaurant
APPENDIX C

The list of participants
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