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DEVELOPMENT AND CHARACTERIZATION OF ADVANCED NEUTRON MULTIPLIER MATERIALS

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Neutron multiplier materials are essential for self-sufficient tritium production and are closing the fuel cycle of fusion reactors. Until now, a concept of pebble bed consisting of interchanging layers of beryllium and lithium ceramic pebbles was considered for the Helium-Cooled Pebble Bed (HCPB) tritium-breeding module of the first experimental fusion reactor ITER as well as for the next demonstration fusion reactor DEMO. However, this concept depends on the availability of large amounts of pure beryllium pebbles and is also limited by its material properties like for example the tritium accumulation under irradiation.

The results of tritium retention and analytical microstructural studies of beryllium pebbles obtained within the framework of the HIDOBE irradiation campaign suggest that a significant fraction of generated tritium (up to 100% below 500°C) is trapped within helium bubbles. Being negligible in the ITER tritium-breeding module (TBM), the total accumulated tritium inventory imposes severe safety issues and exceeds acceptable limits for the DEMO blanket. Therefore, advanced neutron multiplier materials such as beryllides have to be well characterized for their applicability in the HCPB blanket of DEMO and beyond. The usage of an advanced material with lower volumetric swelling, lower tritium retention, increased irradiation and chemical resistance as well as with higher melting temperature allows to switch from the pebble bed concept to a solid hexagonal block-based one. For the fabrication of titanium beryllide samples both the semi-industrial fabrication route utilizing the hot extrusion of rods and the industrial approach using vacuum hot pressing of a Be-Ti powder mixture were explored. In this contribution, we discuss reasons for the transition from pure beryllium to beryllides, respective changes of the HCPB blanket design and a successful demonstration of the feasibility of a beryllide block fabrication by an industrial method.

Keywords: beryllium; titanium beryllides; neutron irradiation

Highlights:

- Transition from pure beryllium to beryllides as neutron multiplier material enables new "fuel-breeder pin" HCPB blanket design
- Solid blocks of $TiBe_{12}$ are considered in the new HCPB reference design
- Feasibility of industrial fabrication of solid beryllide blocks is successfully demonstrated
- Titanium beryllide block survives 50 heating-cooling cycles despite anticipated intrinsic brittleness of intermetallic compounds

Introduction

The previous design concept of helium-cooled pebble bed (HCPB) blanket made use of alternating layered pebble beds of lithium and beryllium. Lithium enriched by the ^{6}Li isotope is intended for tritium breeding, while beryllium is used for neutron multiplication. This combination allows achieving a sufficiently high tritium breeding ratio necessary for closing the fuel cycle of fusion reactors. The major advantages of the concept based on solid breeding and neutron multiplier materials can be summarized as follows. A solid tritium-breeding blanket is the most compact one, while beryllium has the largest cross-section for neutron multiplication [1]. This concept avoids the use of liquid metal and, hence, should not deal with related liquid metal corrosion and does not require any coatings for its mitigation. There is no potential risk of coolant (either liquid metal or water) leakage which may result in exothermal reactions with surrounding structural or functional materials. In addition, the operation temperature of structural materials in the HCPB blanket concept is above 400°C, i.e. the temperature below which all structural materials are subject to irradiation embrittlement.

An obvious bottleneck of this HCPB concept is the necessity of fabrication of a very large amount of beryllium pebbles. It should be mentioned that beryllium pebbles with a diameter of 1 mm produced by the rotating electrode method are still the reference material for the ITER Tritium Breeding Module (TBM). The ITER TBM will require about 700 kg of beryllium pebbles [2]. This amount can still be timely delivered by upscaling the existing fabrication technology. However, the production of 300 tons of beryllium pebbles envisaged for the demonstration fusion reaction DEMO is not feasible by this fabrication method. Another potential problem is excessive tritium retention in beryllium. Tritium is generated as a byproduct of neutron multiplication reactions within the beryllium bulk material. If accumulated inside Be-pebbles, tritium poses a serious safety risk and hinders radioactive waste utilization after the end-of-life of the blanket. These considerations enforced us to seek for another solution and to reconsider the pebble bed concept.

The concept of pebble beds was suggested in the early 1990s by Mario Dalle Donna [3] and was motivated by the following considerations. (i) Pebbles with a diameter of 1 mm were selected to reduce the diffusion path of tritium to escape from pebbles thus reducing tritium residence time within pebbles, and, hence, the overall tritium inventory. (ii) The usage of pebbles instead of beryllium solid blocks decreases internal stresses induced by swelling under neutron irradiation and thermal expansion, thus, preventing pebble fracture. (iii) Purge gas with hydrogen addition being intended for efficient tritium removal can be pumped between the pebbles.

As an alternative to pure beryllium, some intermetallic compounds on beryllium basis were also considered as advanced neutron multiplier materials since the beginning of the new century [4–7]. These compounds possess higher melting point and demonstrate lower chemical reactivity with oxygen and water [8,9], better compatibility with structural materials [10,11], as well as have higher stability under irradiation and lower tritium retention [10,12]. Earlier these materials were mainly fabricated in the form of tablets by using plasma syntering or as rods by hot extrusion methods [13,14] or less commonly by other methods [15–17], which all have their limitations with respect to fabrication on the industrial scale.

In our opinion, wider practical application of these materials was hindered up to now by two major reasons. Firstly, intermetallic alloys are generally brittle at room temperature due to a insufficient

number of independent slip systems, which can be traced back to the complex structure of the underlying crystal lattice (see, e.g., [18]). Secondly, the technology for their fabrication on an industrial scale was largely missing.

After reconsidering the arguments leading to the selection of the pebble bed concept in the past, it became clear that if material free of these disadvantages could be found, the concept of pebble bed might bereevaluated.

In this paper, we demonstrate the feasibility of production of alternative solid neutron multiplier material on an industrial scale, reconsidering the pebble bed concept in favor of solid blocks from the new material and describe respective changes of the DEMO HCPB blanket design.

Experimental methods

Materials

Fabrication of beryllium pebbles

The reference beryllium pebbles with a diameter of about 1 mm were fabricated by the NGK Insulators Ltd., Japan using the rotating electrode method [19]. More details on the fabrication method can be found in section 0.

These pebbles were irradiated in the High Flux Reactor (HFR), Petten, the Netherlands within the HIDOBE-02 irradiation campaign [2] at temperatures 375-675°C until an accumulation of 6000 appm He.

Microstructure and properties of the pebbles were investigated in detail before [2,20,21] and after irradiation [22–25].

Fabrication of beryllides by hot extrusion

Pure Be and Ti powders were blended together, sealed into capsules, and extruded at 650°C with a reduction ratio of 8:1 at the Extrusion Research and Development Center, Technical University of Berlin. Extrusion details have been previously described in [13]. The chemical composition of the extruded composite is as follows in wt.%: Be-29.11Ti-0.219O-0.102Fe-0.077C-0.037Al-0.036Mg-0.022Si-0.0018N-0.0018Ca-0.00193U [15]. After hot extrusion, sections of Be-Ti rods having Ø40mm×12mm were sealed into capsules and subjected to the hot isostatic pressing (HIP) at temperatures of 800 and 900°C under an argon pressure of 102 MPa for 4 h in the “Edmund Buehler HIP 2000” at the Karlsruhe Beryllium Handling Facility (KBHF). The samples were heated and cooled at a rate of about 10–20 K/min.

After HIP, Be-Ti samples were removed from the capsules and cut with a diamond saw along and across the extrusion direction. Sample surfaces were polished either mechanically or electrolytically after sequential mechanical grinding. The samples were investigated using scanning electron microscopy (SEM) on a Zeiss Merlin microscope.

Fabrication of beryllides by vacuum hot pressing

Many of beryllium products are fabricated by powder metallurgical route. Elemental source powders are mixed together and then compacted to a solid sample by using one of the available consolidation

methods: sintering, hot extrusion, hot isostatic or vacuum hot pressing. In this work we tested the hot isostatic pressing augmented with the vacuum hot-pressing method.

Beryllium, the PTB56 grade manufactured by Ulba Metallurgical Plant (UMP), Kazakhstan, and Ti (RP-Ti grade OM-1, JSC Polema) powders were taken as source materials. Beryllium powder consists of 98.93Be, 0.78O, 0.11Fe, 0.025Si, 0.019Mg, 0.019Al, 0.018Ni, 0.012Mn (wt.%). The maximum content of other elements is limited as follows (wt.%): 0.005Pb, 0.01Cu, 0.05C, 0.001F. A strong advantage of PTB-56 Be powder is its very low uranium content of less than 0.4 ppm. Impurities in titanium powder are 0.35H, 0.2Fe, 0.2Ni, 0.1Si, 0.08N, 0.05C (wt.%).

Be and Ti powders were blended together in a ratio of 70/30 respectively and were compacted to cylindrical composite samples of Ø30 mm × 50 mm using cold isostatic pressing (CIP). Be-Ti composite samples were annealed in vacuum in the temperature range of 650–1275°C for an hour. After annealing and cooling, the phase composition, dimensions, and density were studied.

Titanium beryllide powder was fabricated by hot isostatic pressing (HIP). The Be-Ti composites after CIP were sealed into steel capsules. After degassing, capsules were annealed at 1150°C for 5 hours in an argon atmosphere with a pressure of 132 MPa. After dissolving the capsule shell in acids, the beryllide pieces were ground to powder.

At the last stage of sintering, vacuum hot pressing (VHP) was performed. VHP parameters are a trade secret of UMP and cannot be yet disclosed. After VHP, a hole with a diameter of 2 mm was drilled using a waterjet. In total, three workpieces of Ø150 mm×170 mm were manufactured. The flat surfaces of the hexagon, as well as the internal hole of the required diameter and samples for research, were cut using an electrical discharge machining (EDM).

More details on the fabrication method can be found in [26].

TEM sample preparation

For the preparation of the transmission electron microscopy (TEM) samples, irradiated pebbles were embedded into epoxy resin and mechanically polished to obtain a metallographic cross section suitable for optical microscopy. After optical inspection a targeted TEM lamellae preparation from the region of interest was performed using a Zeiss Auriga Focused Ion Beam (FIB) system. TEM analyses were carried out in a Thermofisher Talos F200X (scanning) transmission electron microscope, equipped with a Super-X detector for energy dispersive X-ray spectroscopy (EDS), a Gatan Enfinium spectrometer for electron energy-loss spectrometry (EELS) and a Scanning Device (STEM) including a high angle annular dark-field ring detector (HAADF). The electron transparent region has typically a size of 4–8 µm. TEM imaging and most analytical mapping were performed using a FEI Tecnai F20 electron microscope. Acquisition of electron energy-loss spectrum images of single helium bubbles was performed in STEM mode with a spatial resolution of ~1 nm.

Results

Beryllium as NMM

Beryllium with its exceptional neutron moderation and multiplication properties is long considered as a primary candidate for solid neutron multiplier material (NMM) for fission and fusion facilities [1,2,27]. It was chosen as NMM for the ITER TBM as a material with the highest neutron

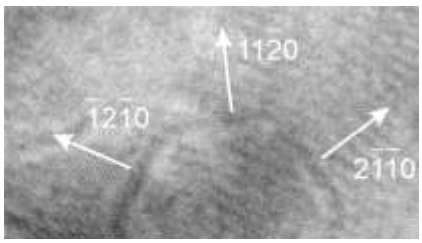
multiplication ability compared to other non-fissile materials. Apart from its advantages, it has also some shortcomings such as high chemical reactivity with steam and air as well as relatively high volumetric swelling under neutron irradiation at temperatures above 650°C [28]. To avoid cracking of beryllium blocks under stresses induced by swelling and allow quick release of generated tritium, it was decided in the 90s to use beryllium in a form of pebbles – small spheres with a diameter of 1 mm. Although bringing several advantages, this decision enforced specific fabrication process with intrinsically low product yield. It is assumed that Be-pebbles can be fabricated by a rotating electrode process (REP) consisting in melting of a rotating beryllium rod by electric arc [19]. In this process, droplets of molten beryllium are thrown off the end of the rod solidifying on the fly in an inert gas atmosphere. The yield of spherical pebbles with an acceptable size of 1±0.1 mm is about 50%. Such a fabrication process might be suitable for the production of several hundred kilograms of beryllium pebbles for the ITER TBM. However, due to its low scalability and yield, this method is obviously not appropriate for fabrication of several hundred tons of Be-pebbles necessary for the future DEMO fusion reactor.

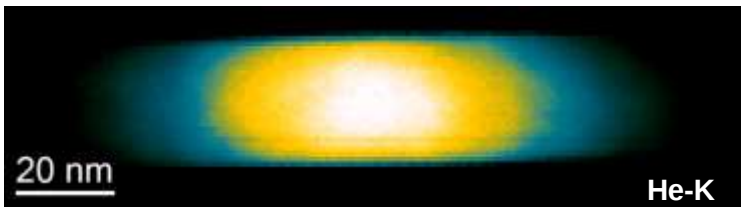
As far as fabrication of pebbles by REP is quite expensive and cannot be extended to an industrial scale, several alternative fabrication methods were exploited and pebbles produced by these methods were characterized at KIT [27,29]. However, the outcomes were not very encouraging, since the alternative pebbles were more porous, contained more impurities as well as hidden cracks in comparison to the reference REP pebbles.

A deep understanding of the reference Be-pebble behavior was gained from the post irradiation examinations of the high dose beryllium irradiation campaign HIDOBE [2,22,24,27]. Under neutron irradiation, helium and tritium are generated in beryllium due to nuclear transmutations. At temperatures relevant for the HCPB blanket operation, helium facilitates formation of bubbles dominating the microstructural changes (see Figure 1a) [25]. Using advanced characterization methods, the KIT scientists were able to show for the first time that tritium is trapped directly within helium bubbles predominantly sticking to the bubble walls (see Figure 1b) [30]. This effect is also predicted by our *ab initio* simulations (C. Stihl unpublished) and is tentatively attributed to the formation of beryllium hydride.

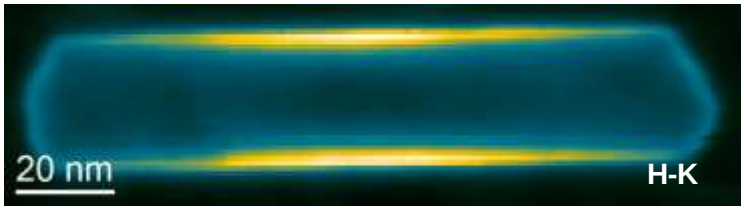
These first of a kind microstructural studies together with the state-of-the-art modelling revealed a mechanism of tritium trapping in beryllium and explain its strong binding to helium bubbles.

Tritium release experiments performed at NRG and KIT have shown that the tritium inventory notably reduces with the increase of irradiation temperature [2,27]. However, the operating temperature of a beryllium pebble bed (PB) cannot exceed 650°C due to excessive volumetric swelling of beryllium [31,32]. Based on the observed irradiation temperature dependence of tritium retention, one can conclude that despite of the significant reduction of tritium inventory in the center of the PB operated at 600°C, tritium cannot escape from the bubbles at periphery regions having lower temperatures.





(a)



(b)

Figure 1: (a) Helium bubbles in irradiated beryllium as viewed along [0001] axis; (b) EELS measurement of the helium intensity (upper panel) and the tritium intensity distribution (lower panel) inside a single bubble viewed along [$2\bar{1}\bar{1}0$] axis.

This excessive tritium retention together with the impossibility of an affordable fabrication technology for manufacturing hundreds of tons of beryllium pebbles represent a critical hindrance for any solid breeding blanket using beryllium and forced us to look for other innovative neutron multiplier materials.

Beryllides as advanced NMM

Since 2004, the KIT team has started the development of advanced NMM based on intermetallic beryllium alloys. Various methods of production have been tested in cooperation with the KIT spin-off, Karlsruhe Beryllium Handling Facility and TU Berlin: melting, hipping, and hot extrusion. Melting turned out to be an inadequate fabrication method due to the formation of other phases, cracks, and shrinkage cavities [15,33]. Whereas the powder metallurgy route with powder consolidation by the hot isostatic pressing (HIP) has led to the first success [34]. The hot extrusion method has been developed in the frame of the EU-JA cooperation program running within “Broader Approach to realization of Fusion Power”. This work was aimed at the production of beryllide rods suitable for the fabrication of pebbles from beryllides by REP [35]. Such pebbles are foreseen in the Japanese concept of the water-cooled solid breeder (WCSB) design [36]. Unfortunately, already the first extrusion tests at TU Berlin have revealed that beryllides are too hard to be extruded homogeneously.

Therefore, an innovative idea was proposed to reduce the extrusion temperature thus avoiding the formation of the beryllide phase with its exceptional hardness during extrusion. Instead, a two-phase composite still retaining some ductility of the source metals, but strong enough to survive a high-speed rotation during pebble fabrication, was successfully produced and then used for the pebble fabrication at QST, Rokkasho, Japan. Formation of the desired beryllide phase can be accomplished during pebble fabrication and consequent homogenization annealing if required.

Although the extrusion method might be more suitable for industrial fabrication than sintering used in Japan, its scalability is still limited. The extrusion method may be appropriate for the production of several tons of beryllide pebbles required for the Japanese ITER TBM but seems to be too restrictive for the fabrication of several hundred tons necessary for the DEMO reactor. Therefore, we continued the search for an industrially scalable fabrication process for beryllium alloys. As it will be shown in section 0, the powder metallurgy route has led us to success.

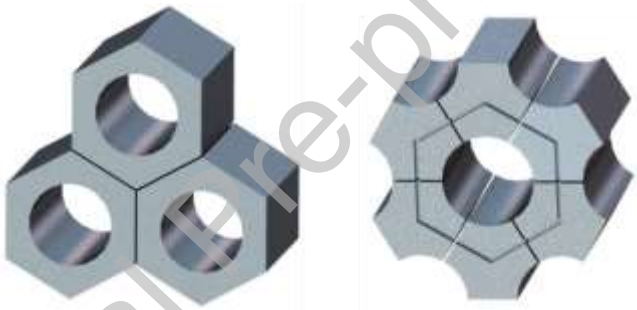
Transition from pure beryllium to innovative intermetallic alloys enabled the redefinition of the previous breeding blanket design by using solid blocks instead of pebbles at much higher temperatures. This decision (1) solves the critical safety issue with tritium retention, (2) significantly reduces irradiation swelling, (3) greatly reduces interaction with air and steam, enhancing intrinsic

safety, (4) enables scalable fabrication routes for the timely and affordable industrial production of NMM, and (5) allows in future the increase of the overall fusion reactor thermal efficiency by using higher coolant temperatures.

Innovative HCPB tritium-breeding blanket design

The development of the superior neutron multiplier material has been reflected in the enhanced blanket design. Major conceptual change has been proposed for the HCPB concept in 2017-2018, which resulted in the current "fuel-breeder pin" as the new reference design of the candidate driver blanket for the EU DEMO [37–39]. In this concept, hexagonal prismatic blocks from titanium beryllide are used as NMM and lithium ceramic pebbles as tritium-breeding material. The central channel of the pin is filled with lithium ceramic pebbles and is purged with a gas containing the addition of hydrogen for facilitating tritium release. This innovative design allows very dense space filling with $TiBe_{12}$ blocks thus achieving a high tritium-breeding ratio (TBR).

According to the new design, beryllides will be used in the form of massive hexagonal blocks (see Figure 2a) or more complex shape prisms (see Figure 2b), which might be more resistant to the transient thermal stresses. The latter shape also allows more efficient use of material – up to 60% vs 40% for the former shape, although in both cases the rests will be reused in the fabrication process.



(a) (b)

Figure 2: Two types of beryllide blocks for neutron multiplication in the enhanced HCPB design: (a) hexagonal prism (Ø144 mm × 150 mm) with a hole of Ø80 mm, (b) alternative complex shape prism (Ø85 mm × 90 mm)

Modern state of the art computer aided design coupled with neutron transport calculations as well as with thermohydraulical and thermomechanical calculations allows the application of a holistic design approach permitting integration of various reactor systems and direct evaluation of the material operation conditions like operation temperature, neutron damage, helium and tritium generation rates, etc. [15].

Temperature distribution within a typical hexagonal block from $TiBe_{12}$ obtained as a result of coupled neutronic and thermohydraulical calculations (see Figure 3) confirms that the maximum temperature of a beryllide block is below 950°C, while that of the stainless-steel tube will not exceed 550°C.

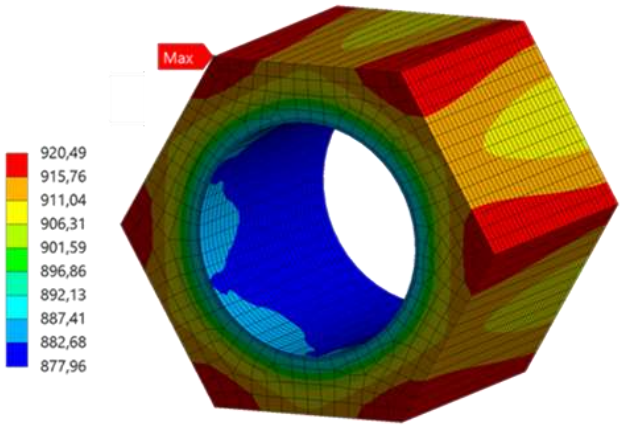


Figure 3: Temperature distribution within hexagonal $TiBe_{12}$ block at the end of a plasma pulse

As far as beryllides are generally known for their intrinsic brittleness, their resistance against fracture under periodical thermal stresses induced by a pulse operation of the DEMO reactor as well as a reliable control of the block temperature under such conditions had to be demonstrated. First results towards a successful accomplishment of this challenge are described in section 0.

Development of industrial-scale fabrication technology of beryllide blocks

An important step in accessing of a feasibility of the fuel-breeder pin design was a development of an industrial scale fabrication technology of hexagonal blocks from titanium beryllide. This step was undertaken by KIT in close cooperation with the Ulba Metallurgical Plant, Kazakhstan. Based on the long-term experience in the development of beryllides, KIT provided the necessary geometrical and material specifications as well as the ranges for the most important technological parameters. From the very beginning, the powder metallurgical route was identified as the most advanced way for industrial production of beryllium-containing products.

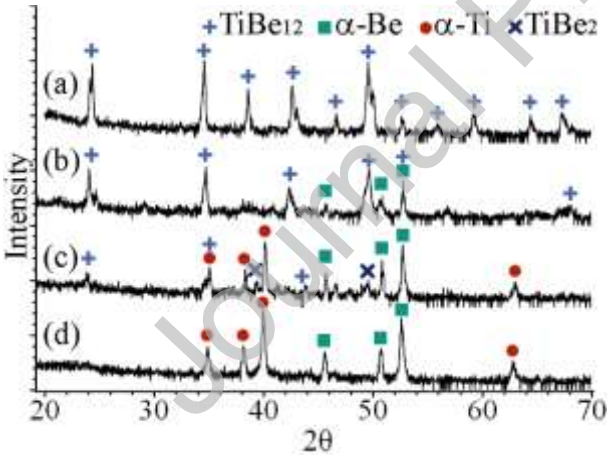


Figure 4: X-ray diffraction patterns showing (a) a formation of single phase $TiBe_{12}$ at 1100-1275°C, and a mixture of beryllides and pure Be and Ti phases at (b) 1000°C, (c) 800°C, (d) 650°C

Commercial purity powders of Be and Ti were blended together and compacted to cylindrical composites using cold isostatic pressing. These samples were annealed at temperatures 650–1275°C and analyzed with respect to their phase composition. Corresponding X-ray diffraction curves reveal the formation of a single $TiBe_{12}$ phase starting from 1100°C (see Figure 4). Accordingly, the hot isostatic pressing (HIP) treatment at 1150°C for 5 hours under an argon pressure of 132 MPa was used to manufacture titanium beryllide powder.

The pieces of the HIP-compacted beryllide were ground to powder and consolidated again using vacuum hot pressing. The latter method has proved to produce blocks without cracks in contrast to HIP. Since beryllides are very hard and brittle materials, electrical discharge machining and waterjet were used to finish the outer hexagonal shape and to cut the inner hole.

Three blocks in total were produced up to now demonstrating a good replicability of this novel technology. Similar technologies were used in the 1960s on laboratory scale for fast screening of suitability of various beryllides for high temperature applications [40]. However, up to our knowledge, the feasibility of an industrial fabrication of samples from beryllides of such size, complex shape, and surface finishing has not been demonstrated up to now. This approach paves the way for the fabrication of a full-scale prototypical mock-up and its qualification and functional testing under conditions foreseen in the HCPB blanket of the DEMO fusion reactor.

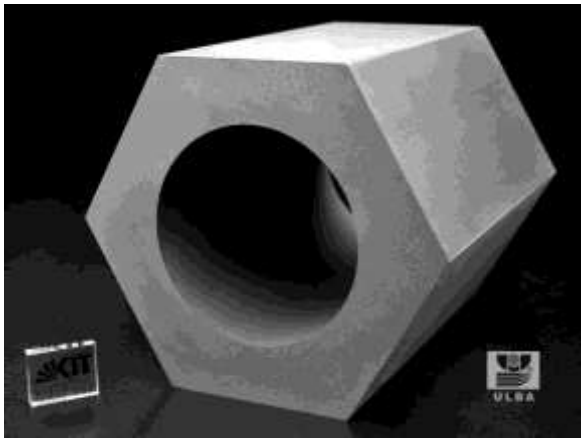


Figure 5: Hexagonal $TiBe_{12}$ block after final machining

We can conclude that for the first time, $TiBe_{12}$ blocks with dimensions ∅144×150 mm^2 and density reaching 98.8% of the theoretical value were produced using industrial equipment (see Figure 5*).*

Being intermetallic compounds, beryllides suffer from intrinsic brittleness. Successful fabrication of the real-scale blocks opened an opportunity to test the resistance of $TiBe_{12}$ to thermo-cycling being typical for the pulse operation conditions in the DEMO fusion reactor. An accelerated thermal cycle representing DEMO relevant ramp-ups and downs is depicted in Figure 6. The block temperature is cycled between 200 and 900°C with heating and cooling periods of 60 seconds and a holding time of 45 seconds. Preliminary tests on cylindrical samples using induction heating and natural convection on air were performed at the UMP.

A test with 50 cycles confirmed mechanical and chemical stability of titanium beryllide under these conditions: no cracks or traces of oxidation were found. In addition, a hexagonal mock-up was successfully thermocycled under similar conditions (see Figure 7). More advanced and long-lasting testing is on the way. Such real-size mock-up tests present a major progress in the development of advanced NMMs permitting a fast increase of the technology readiness level for the complete HCPB tritium-breeding blanket.

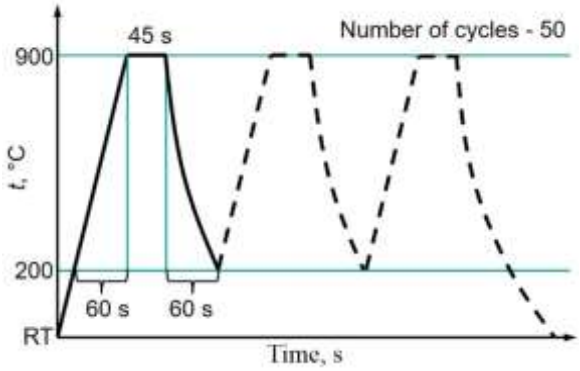


Figure 6: Change of temperature of $TiBe_{12}$ block with time under conditions similar to pulsed operation of the DEMO fusion reactor

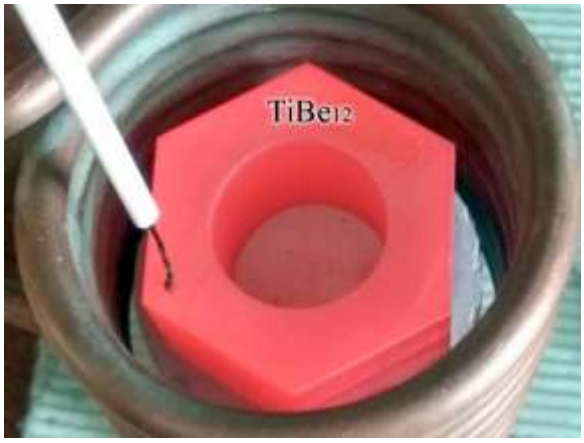


Figure 7: Induction heating of a $TiBe_{12}$ hexagonal mock-up up to 1100 °C as preparation step for advanced thermocycling

Conclusions and outlook

Based on the long-term development of neutron multiplier materials for the solid tritium-breeding blanket at KIT [27], an industrial-scale fabrication technology for massive titanium beryllide blocks was successfully developed in cooperation between KIT and Ulba Metallurgical Plant. Preliminary thermocycling tests confirm the stability of this material against cracking and oxidation. These developments greatly support the innovative tritium-breeding blanket "fuel-breeder pin" Helium-Cooled Pebble Bed (HCPB) concept utilizing hexagonal prismatic blocks of titanium beryllide as neutron multiplier material (NMM) and lithium ceramic pebbles for tritium breeding [1,37]. These achievements will pave the new avenue for a real-size mock-up fabrication route as well as for mock-up testing under operational conditions foreseen inside the ITER TBM and the fuel-breeder pin blanket of the DEMO fusion reactor.

Acknowledgments

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Availability of the data

The raw data required to reproduce these findings except technological parameters for $TiBe_{12}$ fabrication are available on request from the corresponding author. The technological parameters for $TiBe_{12}$ fabrication are a trade secret of the Ulba Metallurgical Plant, Ust-Kamenogorsk, Kazakhstan, and cannot be disclosed.

Declaration of Competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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