Nuclear Materials and Energy 35 (2023) 101444

Contents lists available at ScienceDirect

Nuclear Materials and Energy

journal homepage: www.elsevier.com/locate/nme

# Beryllium intermetallics: Industrial experience on development and manufacture

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ARTICLE INFO

*Keywords:*
Beryllides
Beryllium
Vacuum hot pressing
Neutron multiplier materials
High-temperature materials, DEMO

ABSTRACT

Ulba Metallurgical Plant JSC has been leading several research and development programs aimed at producing beryllides for both structural and fusion applications. The main focus has been on the development of tantalum beryllide as a high-temperature material, and chromium and titanium beryllides as neutron multipliers for EU DEMO. Utilizing vacuum hot pressing, billets of tantalum, chromium, and titanium beryllides were successfully produced, with their key properties such as density, phase composition, and microstructure extensively analyzed. These results show promising potential for expanding the use of beryllides and developing new compositions, highlighting the continued importance of research in this area.

## Introduction

Beryllides represent a unique class of materials with an intriguing combination of properties, yet they remain one of the least studied groups of materials [1,2]. This can be attributed in part to the scarcity of beryllium worldwide, and the specialized conditions required to handle this element. Consequently, only a limited number of companies in the world are capable of manufacturing intermetallic compounds of beryllium, known as beryllides. The majority of research on the development and characterization of beryllides was conducted by Brush Beryllium Co. between the 1960s and 1980s, which is now known as Materion Corp. In the 2000s, the Ulba Metallurgical Plant JSC (UMP JSC) collaborated with Japan to develop beryllides, and several large ingots were successfully manufactured through zone melting. Renewed interest in beryllides emerged in the aerospace industry in the 2010s, due to their exceptional properties including high melting point, low density, high corrosion resistance, and high thermal conductivity. In the 2020s, UMP JSC partnered with the Karlsruhe Institute of Technology (KIT), Germany, to produce a series of beryllide blocks for fusion applications [3]. The successful production of these blocks led to the replacement of beryllium pebbles with solid beryllide blocks in the helium-cooled pebble bed (HCPB) blanket design of the European DEMO fusion reactor [4].

This article provides an overview of the research and development of intermetallic compounds of beryllium with tantalum ($Ta_2Be_{17}$), chromium ($CrBe_{12}$), and titanium ($TiBe_{12}$) at UMP JSC. These beryllides exhibit promising potential for application in various industries such as nuclear and fusion power engineering, aerospace, and instrumentation. However, despite their potential, the process of obtaining billets and articles from beryllides has not yet advanced to the stage of stable industrial production.

## Materials and experimental techniques

Pure beryllium, tantalum, titanium, and chromium powders were utilized as the starting materials for synthesizing the corresponding beryllides. The initial powder mixtures were prepared in stoichiometric proportions with a slight excess of beryllium. The resulting beryllides were then ground into powders and processed using vacuum hot pressing (VHP) technology, which has been developed and optimized by UMP JSC. The VHP process is conducted in graphite molds at a temperature range of 0.7–0.8 times the melting temperature of the corresponding beryllide. Further specific details of the VHP process are proprietary and cannot be disclosed.

Following VHP, the surface of the beryllides was cut using electrical discharge machining (EDM). The phase composition of the synthesized

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https://doi.org/10.1016/j.nme.2023.101444
Received 6 April 2023; Received in revised form 4 May 2023; Accepted 8 May 2023
Available online 12 May 2023

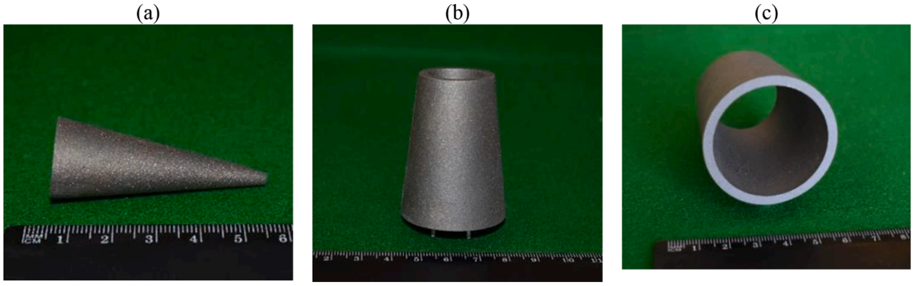


**Fig. 1.** Appearance of tantalum beryllide $Ta_2Be_{17}$ products: a) cone of Ø25 mm × 50 mm; b) hollow cone of Ø27–37 mm with a wall thickness of 3 mm; c) hollow cone of Ø35–45 mm with a wall thickness of 5 mm.

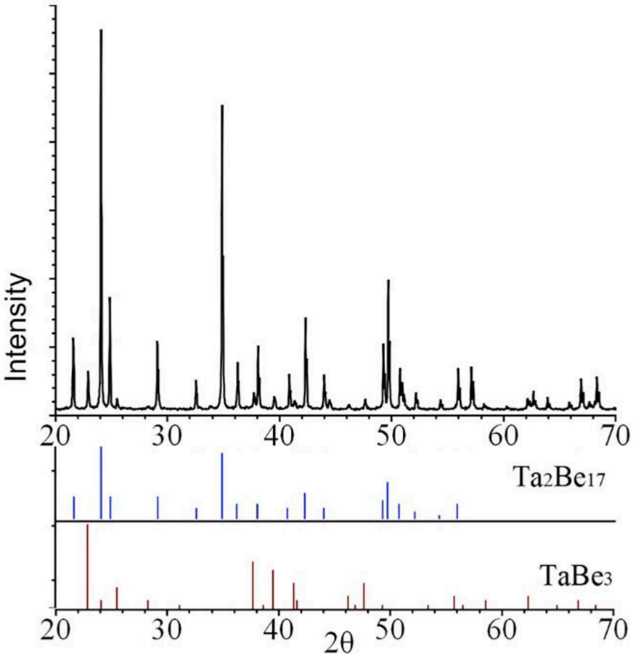


**Fig. 2.** X-ray diffraction pattern of a $Ta_2Be_{17}$ sample. Below the diffraction pattern are the peaks for $Ta_2Be_{17}$ and $TaBe_3$ used for the analysis.

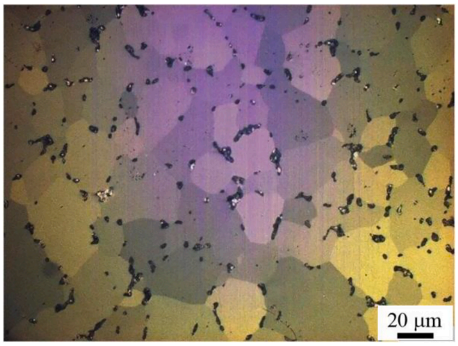


**Fig. 3.** Microstructure of $Ta_2Be_{17}$ produced by VHP. Optical microscopy in polarized light.

beryllides was determined using X-ray diffractometry. The microstructure of the densified material was evaluated using optical metallography with polarized light, as well as scanning electron microscopy. Energy-dispersive spectroscopy (EDS) was performed to determine the chemical composition of particles in chromium and titanium beryllides. EDS maps were generated for oxygen, nitrogen, and the respective metal (chromium or titanium). The beryllium content in the material could not be determined using EDS due to the absorption of its characteristic radiation by the material itself.

## Results and discussion

### *Development of technology for obtaining billets and products from tantalum beryllide $Ta_2Be_{17}$*

Tantalum beryllide $Ta_2Be_{17}$ is highly regarded as a high-temperature material due to its exceptional creep and corrosion resistance, as well as its high melting point of 1980 °C [2,5,6]. This material was extensively studied in the 1960s–1980s as a potential material for reentry and hypersonic transportation [5,6]. In light of renewed interest in high-temperature beryllides, UMP JSC embarked on the development of a manufacturing process for $Ta_2Be_{17}$ products.

However, the high melting temperature of this material posed a challenge in obtaining defect-free, solid, single-phase billets with high density close to the theoretical density (TD) of 5.05 g/cm$^3$ [2,7]. R&D efforts included the optimization of initial powder synthesis parameters for beryllium and tantalum, as well as the exploration of various technological methods and modes for billet manufacturing. Both sintering and vacuum hot pressing were utilized, requiring temperatures above 1300 °C to achieve minimal porosity in the billets.

After several attempts, the billets with a diameter of Ø90 mm and a height of up to 90 mm were successfully manufactured. The resulting billets attained a maximum density of 4.85 g/cm$^3$, or 96% of TD. Fig. 1 showcases some of the final products machined from these billets, including test prototypes for the aerospace industry. For example, hollow cones with various diameters and wall thicknesses can serve as nose cones for new hypersonic vehicles.

Fig. 2 shows the results of the X-ray diffraction (XRD) analysis performed on a tantalum beryllide sample. The XRD pattern revealed that the material is predominantly composed of the desired $Ta_2Be_{17}$ phase, but small peaks of the lower beryllide $TaBe_3$ were also detected. Based on quantitative analysis of peak heights, the volume fraction of $TaBe_3$ was estimated to be 5–7%. The microstructural analysis of tantalum beryllide has revealed the presence of polygonal grains with an average size of approximately 25 μm, as depicted in Fig. 3. Notably, the grain

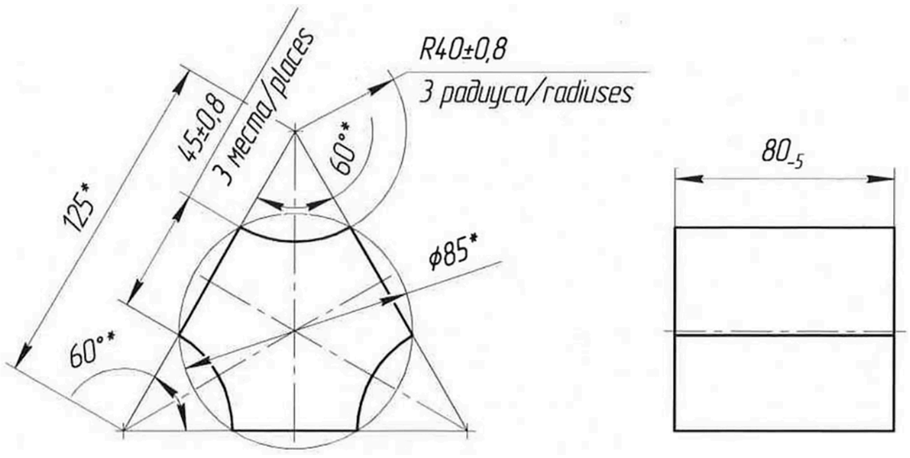


**Fig. 4.** Drawing of a block of complex shape – a prototype neutron multiplier according to the alternative design of HCPB.

**Fig. 5.** Manufacture of blocks from chromium beryllide $CrBe_{12}$: (a) billet after vacuum hot pressing; (b) final full size neutron multiplier block after machining.

boundaries and triple junctions exhibit the occurrence of pores and beryllium oxide particles, which is a common feature observed in materials based on beryllium that are produced via the powder metallurgy route, as per previous research [2,8–10]. Since pores and particles cannot be distinguished, their combined volume fraction was determined to be about 4%. The optical metallography images did not allow to distinguish the grains of phases $Ta_2Be_{17}$ and $TaBe_3$. However, the characterization of tantalum beryllide samples is still ongoing, and further investigations are necessary to enhance the resulting blanks' size and density. Hence, additional research and development efforts are required to improve the material's properties for potential high-temperature applications.

## *Production of billets and blocks from chromium beryllide $CrBe_{12}$*

Chromium beryllide $CrBe_{12}$ is a lesser studied material among beryllides, primarily due to its relatively low melting point of 1337 °C [2,11]. However, this material shows promise as a high tritium breeding ratio material in the blanket of future fusion reactor EU DEMO [12]. In cooperation with KIT, UMP JSC undertook the development of chromium beryllides for use as solid neutron multiplier materials. The objective was to manufacture solid, complex shape blocks of $CrBe_{12}$, as shown in Fig. 4, and evaluate its potential as an alternative to titanium beryllide ($TiBe_{12}$) as a neutron multiplier for fusion technology, as well as its use as a neutron moderator and reflector in nuclear technology.

The production of chromium beryllide encountered minimal difficulties. Using VHP, several billets with dimensions of Ø90 mm × 90 mm

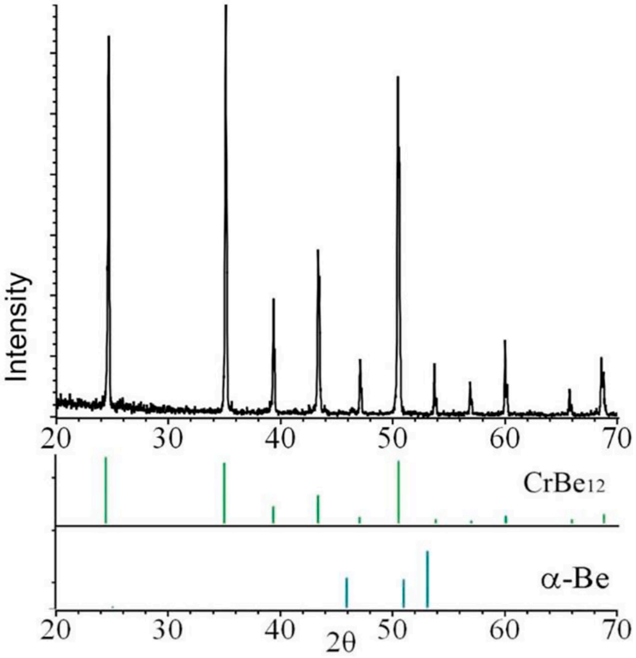


**Fig. 6.** X-ray diffraction pattern of the $CrBe_{12}$ billet. Below the diffraction pattern are the peaks for $CrBe_{12}$ and Be used for the analysis.

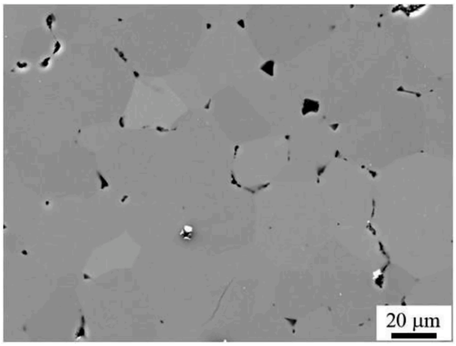


**Fig. 7.** Microstructure of material from $CrBe_{12}$ billet. SEM.

and a density of 2.42 g/cm3 (99.3% of TD, 2.44 g/cm$^3$ [2,11]) were successfully produced, as illustrated in Fig. 5a. Subsequently, the complex shape block of $CrBe_{12}$, as shown in Fig. 5b, was manufactured in accordance with the drawing. To date, a total of 6 blocks of chromium beryllide have been successfully produced, out of which 2 are full-size while the remaining 4 have dimensions of approximately Ø50 mm × 50 mm.

Fig. 6 presents the results of XRD analysis performed on a sample of chromium beryllide cut from the final block of the neutron multiplier. The XRD pattern confirms that the material has a structure corresponding to the target binary beryllide $CrBe_{12}$. No other phases, such as beryllium or beryllium oxide, were detected in an amount sufficient to be discernible by XRD analysis. Microstructural studies of the chromium beryllide revealed an average grain size of approximately 40 μm, as shown in Fig. 7. Similar to the observations made with tantalum beryllide, the microstructure of chromium beryllide also displayed the presence of beryllium oxide particles and occasionally pores along the grain boundaries and at triple junctions. Their joint volume fraction is estimated as 2–4%. According to EDS analysis (Fig. 8), beryllium oxide particles can be distinguished by the absence of chromium and the presence of oxygen, as well as a small amount of nitrogen, whereas only the absence or low content of chromium is observed on the pores.

### *Production of billets and articles from titanium beryllide $TiBe_{12}$*

Titanium beryllide $TiBe_{12}$ has been recognized as a high-temperature material due to its remarkably low density of 2.288 g/cm$^3$ and high melting point of 1600 °C, making it a promising material for high-temperature applications since the 1960s and 1980s [2,5–7]. Recent interest in it was revived as a potential neutron multiplier material for fusion reactors [13–15], which offers a tritium breeding ratio of 1.08 for pebbles [12] and 1.20 for blocks [4]. In collaboration with KIT, the UMP JSC embarked on developing a method to produce solid hexagonal titanium beryllide blocks, as neutron multiplier blocks for the EU DEMO HCPB blanket, depicted in Fig. 9. A more detailed account of the collaboration is available in [3]. Eighteen billets of titanium beryllide were successfully fabricated, with dimensions up to Ø200 mm × 200 mm, as illustrated in Fig. 10a. The density of the billets ranged from 2.17 to 2.20 g/cm$^3$ (97.3–98.7% of TD). The final hexagonal block with a central hole is displayed in Fig. 10b,c. It is worth noting that the successful fabrication of titanium beryllide blocks with such a large size represents a significant milestone, as to the best of our knowledge, such blocks have not been produced before. Moreover, there is a strong potential to further increase the block sizes by at least three-fold, which could pave the way for the development of larger-scale and more efficient applications of this material in high-temperature and nuclear-related fields.

X-ray diffraction analysis revealed the exclusive presence of the intended $TiBe_{12}$ phase in the material (as depicted in Fig. 11). Due to their low volume fraction, beryllium and beryllium oxide present in the microstructure could not be detected using XRD. The grain size of titanium beryllide was notably smaller, measuring approximately 8 μm (as demonstrated in Fig. 12). Along the grain boundaries, some porosity was observed, and particles of beryllium oxide and free beryllium phase were also occasionally found between and within the grains. The microstructure appears to contain a large number of particles, as it has been etched to make the grain boundaries more visible. EDS analysis (Fig. 13) revealed that the grain boundaries look lighter due to the presence of relief and pores. Beryllium oxide particles were found at the boundaries, particularly at triple junctions, and these particles sometimes exhibit also a higher concentration of nitrogen (Fig. 13c).

As part of the thermal cycling program for titanium beryllide, one of the blocks was sectioned lengthwise and across to produce 12 fragments of varying heights: 100 mm, 50 mm, and 33 mm (as shown in Fig. 14). In the second stage of thermal cycling, the block dimensions were specified and 6 fragments were produced with 3 mm gaps between them, as shown in Fig. 15a. In addition, a solid hexagonal block with a diameter of 150 mm and a height of 40 mm was produced as a witness sample, as shown in Fig. 15b. A detailed description of the thermal cycling

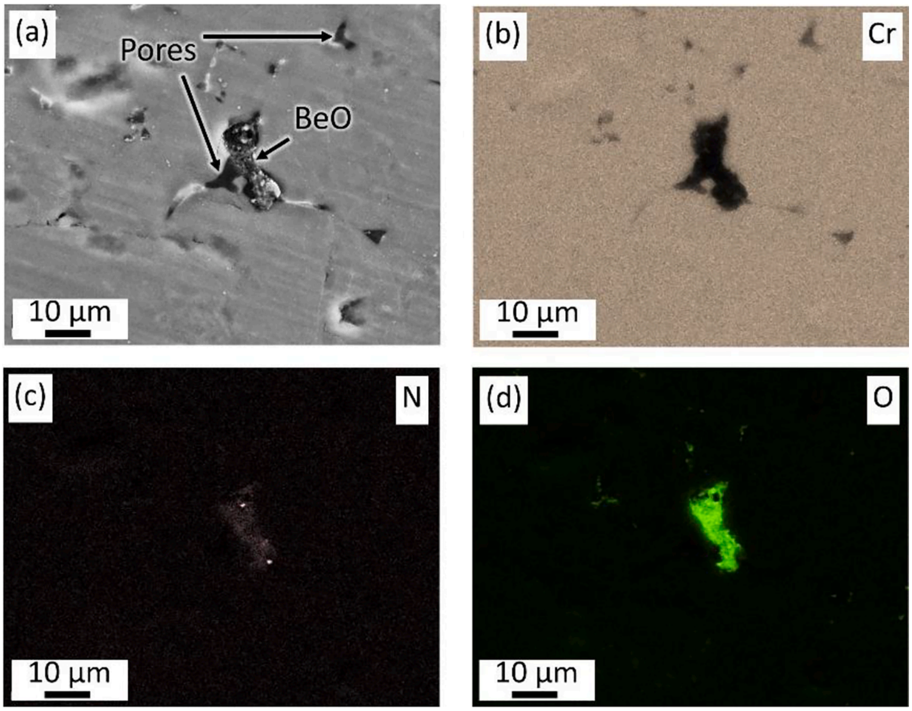


**Fig. 8.** (a) SEM microstructure and (b-d) corresponding EDS maps for a particle and pores in $CrBe_{12}$: (b) chromium, (c) nitrogen, (d) oxygen.

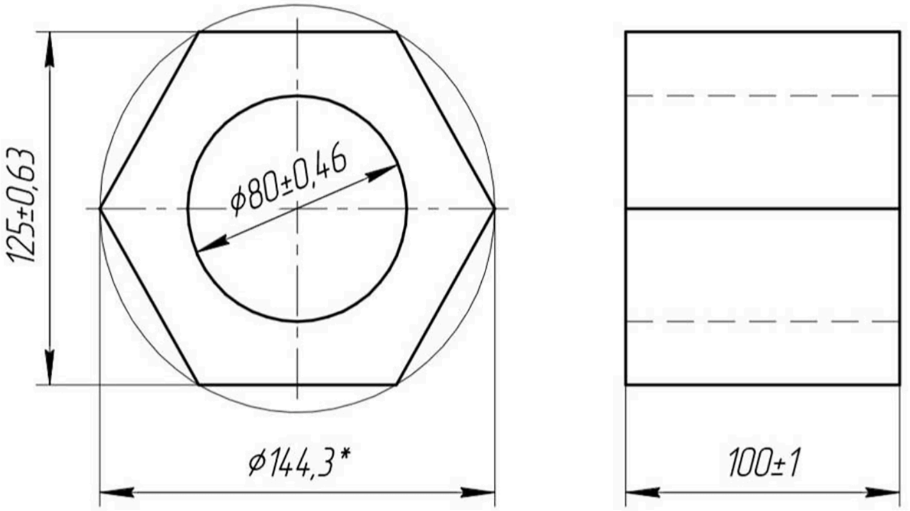


**Fig. 9.** Drawing of a hexagonal block – a prototype neutron multiplier for HCPB.

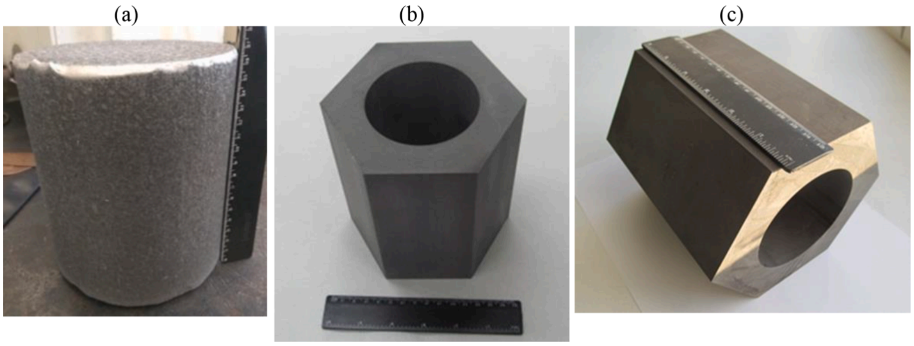


**Fig. 10.** Manufacture of blocks from titanium beryllide $TiBe_{12}$: (a) billet after vacuum hot pressing; (b,c) final hexagonal block with center hole after machining.

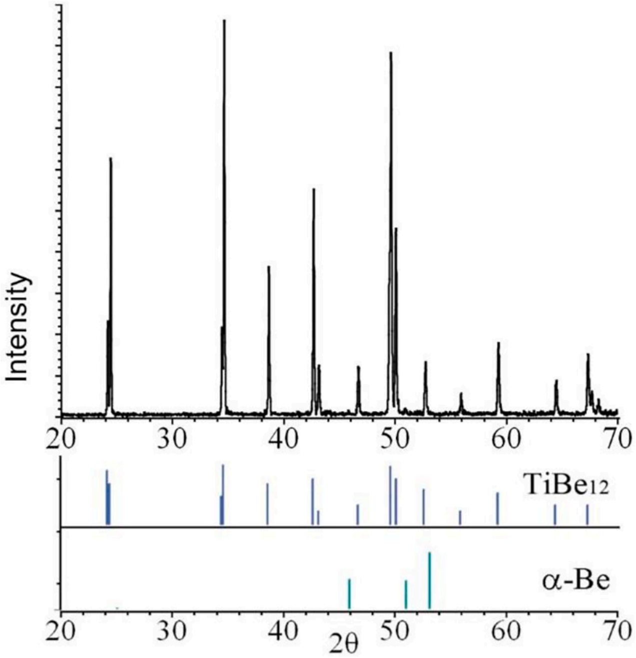


**Fig. 11.** X-ray diffraction pattern of the $TiBe_{12}$ billet. Below the diffraction pattern are the peaks for $TiBe_{12}$ and Be used for the analysis.

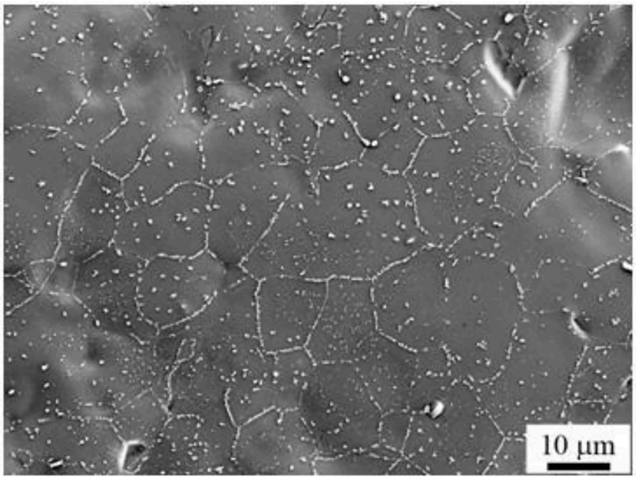


**Fig. 12.** Microstructure of $TiBe_{12}$ billet. SEM of the etched surface.

experiment will be provided in a separate publication.

UMP JSC has expanded the scope of its titanium beryllide manufacturing to include test samples for the automotive and aerospace industries. Among the produced samples is a six-bladed impeller with a size of Ø50 mm × 75 mm, as shown in Fig. 16. These prototypes will undergo corrosion testing under various pressures in contact with water and water vapor to evaluate their suitability for use in harsh environments.

## Conclusion

Recently, significant progress has been made in the development and

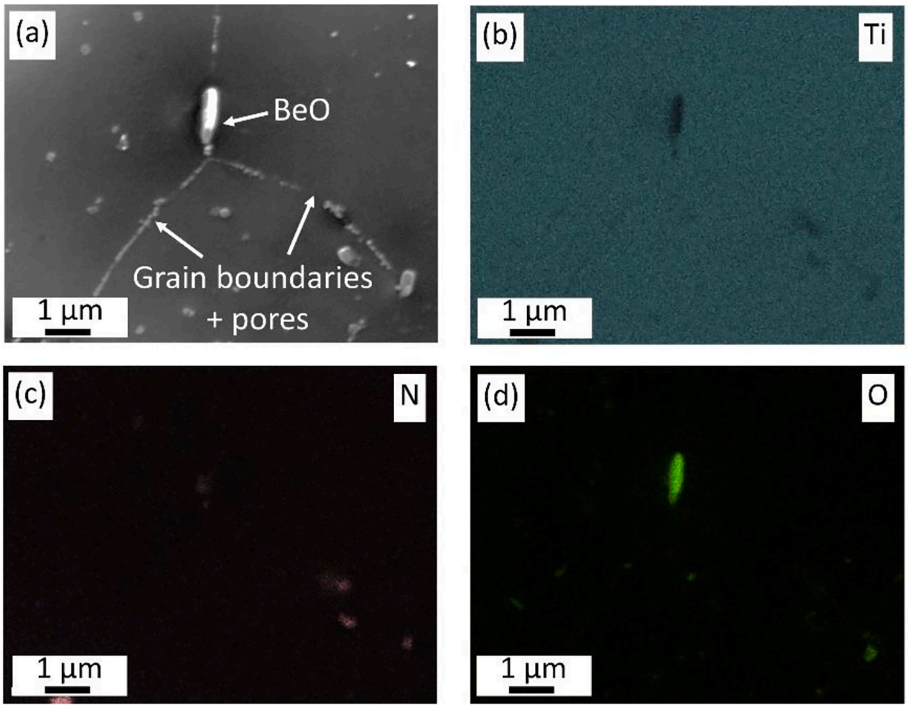


**Fig. 13.** (a) SEM microstructure and (b-d) corresponding EDS maps for a particle and pores in $TiBe_{12}$: (b) titanium, (c) nitrogen, (d) oxygen.

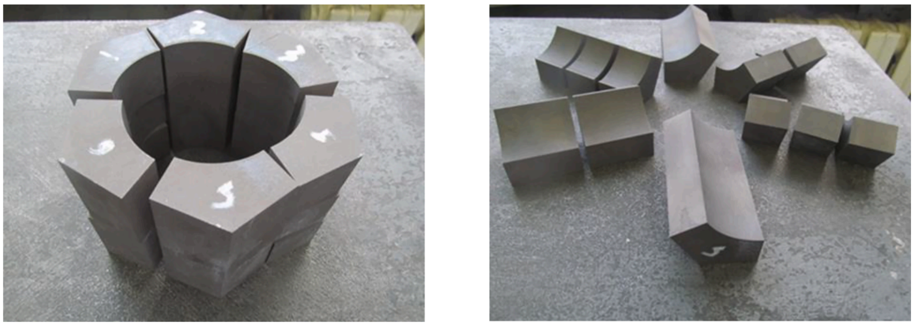
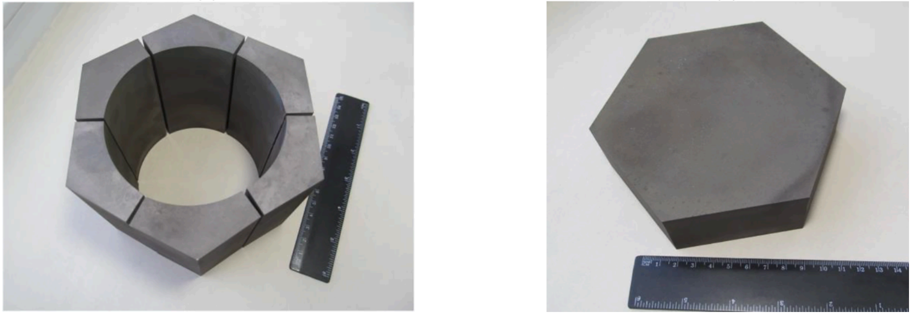


**Fig. 14.** Hexagonal titanium beryllide $TiBe_{12}$ block fragmented in longitudinal and transversal sections for thermal cycling.



(a) (b)

**Fig. 15.** (a) Updated design titanium beryllide $TiBe_{12}$ block fragmented block in longitudinal section and (b) solid witness block manufactured from the same billet.

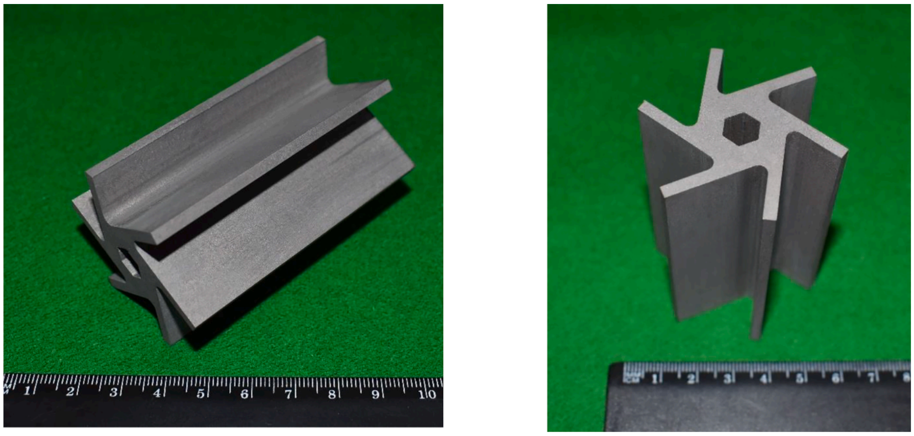


**Fig. 16.** Six-bladed impeller from titanium beryllide $TiBe_{12}$.

implementation of technologies for the production of tantalum, chromium and titanium beryllides. The tantalum beryllide can be produced in sizes up to Ø90 mm × 90 mm, while the chromium and titanium beryllides can be manufactured up to Ø200 mm × 200 mm. The obtained titanium and chromium beryllides demonstrate a single-phase structure of the target corresponding beryllides with minimal traces of beryllium and beryllium oxide. The density of the fabricated beryllide billets exceeds 96%, indicating high-quality production. Ongoing efforts are focused on expanding the applications of beryllides and developing new compositions.

## CRediT authorship contribution statement

**Ramil Gaisin:** Writing – original draft, Writing – review & editing, Investigation. **Yevgeniy Frants:** Writing – original draft, Funding acquisition, Supervision. **Maxim Kolmakov:** Investigation. **Boris Zorin:** Investigation. **Manarbek Kylyshkanov:** Funding acquisition. **Mikhail Podoinikov:** Supervision. **Sergey Udartsev:** Writing – original draft, Writing – review & editing, Visualization, Investigation. **Anatoly Vechkutov:** Investigation, Visualization. **Vladimir Chakin:** Investigation. **Pavel Vladimirov:** Supervision, Funding acquisition.

## 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.

## Data availability

Data will be made available on request.

### *Acknowledgements*

The authors would like to extend their sincere gratitude to the technical staff at UMP JSC who contributed to the successful execution of the experiments. Additionally, the authors from KIT express their thanks to Dr. Klaus Seemann for his valuable contributions in conducting the X-ray diffraction studies.

This work has been partially 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.

We acknowledge support by the KIT-Publication Fund of the Karlsruhe Institute of Technology.

## References

[1] R. Tewari, N.K. Sarkar, D. Harish, B. Vishwanadh, G.K. Dey, S. Banerjee, Chapter 9 - Intermetallics and alloys for high temperature applications, in: A. Tyagi, S. Banerjee (Eds.), Materials Under Extreme Conditions, Elsevier, Amsterdam, 2017, pp. 293–335.

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

[3] R. Gaisin, V. Chakin, P. Vladimirov, F.A. Hernández, S. Udartsev, A. Vechkutov, M. Kolmakov, Industrial-scale manufacturing experience of titanium beryllide block for DEMO blanket application, Fusion Eng. Des. 161 (2020), 111862, https://doi.org/10.1016/j.fusengdes.2020.111862.

[4] F.A. Hernández, P. Pereslavtsev, G. Zhou, B. Kiss, Q. Kang, H. Neuberger, V. Chakin, R. Gaisin, P. Vladimirov, L.V. Boccaccini, G.A. Spagnuolo, S. D'Amico, I. Moscato, Advancements in the helium-cooled pebble bed breeding blanket for the EU DEMO: holistic design approach and lessons learned, Fusion Sci. Technol. 75 (2019) 352–364, https://doi.org/10.1080/15361055.2019.1607695.

[5] J. Booker, R.M. Paine, A.J. Stonehouse, Investigation of intermetallic compounds for very high temperature applications: WADD Tecnical report TR 60-889, The Brush Beryllium Cleveland OH, 1961.

[6] A.J. Stonehouse, R.M. Paine, W.W. Beaver, Mechanical Properties of Intermetallic Compounds: Mechanical Properties of Intermetallic Compounds, 1960.

[7] L.A. Jacobson, R.J. Hanrahan Jr., J.L. Smith, Beryllides, in: Intermetallic Compounds - Principles and Practice, 2002, pp. 37–51.

[8] R. Gaisin, V. Kuksenko, M. Duerrschnabel, V. Chakin, A. Goraieb, P. Vladimirov, Effect of HIP at 1000–1200 °C on microstructure and properties of extruded Be-Ti composites, Nucl. Mater. Energy 30 (2022), 101128, https://doi.org/10.1016/j.nme.2022.101128.

[9] R. Gaisin, V. Chakin, R. Rolli, J. Hoffmann, H. Leiste, T. Bergfeldt, U. Jäntsch, M. Klimenkov, J. Lorenz, A. Goraieb, P. Vladimirov, A. Möslang, Synthesis of Be12Ti compound via arc melting or hot isostatic pressing, J. Alloy. Compd. 818 (2020), 152919, https://doi.org/10.1016/j.jallcom.2019.152919.

[10] R. Gaisin, V. Chakin, M. Duerrschnabel, R. Rolli, T. Weingaertner, A. Goraieb, P. Vladimirov, Effect of HIP at 800 and 900 °C on microstructure and properties of extruded Be-Ti composites, Nucl. Mater. Energy 24 (2020), 100771, https://doi.org/10.1016/j.nme.2020.100771.

[11] G.V. Samsonov, I.M. Vinitskii, Handbook of Refractory Compounds (translated from the Russian by Kenneth Shaw), IFI/Plenum Press, New York, 1980.

[12] F.A. Hernández, P. Pereslavtsev, First principles review of options for tritium breeder and neutron multiplier materials for breeding blankets in fusion reactors, Fusion Eng. Des. 137 (2018) 243–256, https://doi.org/10.1016/j.fusengdes.2018.09.014.

[13] M. Uchida, E. Ishitsuka, H. Kawamura, Thermal conductivity of neutron irradiated Be12Ti, Fusion Eng. Des. 69 (2003) 499–503, https://doi.org/10.1016/S0920-3796(03)00395-8.

[14] C.K. Dorn, W.J. Haws, E.E. Vidal, A review of physical and mechanical properties of titanium beryllides with specific modern application of TiBe12, Fusion Eng. Des. 84 (2009) 319–322, https://doi.org/10.1016/j.fusengdes.2008.11.009.

[15] H. Kawamura, H. Takahashi, N. Yoshida, V. Shestakov, Y. Ito, M. Uchida, H. Yamada, M. Nakamichi, E. Ishitsuka, Application of beryllium intermetallic compounds to neutron multiplier of fusion blanket, Fusion Eng. Des. 61–62 (2002) 391–397, https://doi.org/10.1016/S0920-3796(02)00106-0.