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Effect of low-temperature neutron irradiation on the properties of titanium beryllide

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ABSTRACT

Beryllium-based intermetallic compounds, such as $Be_{12}Ti$, are increasingly being considered as a material capable of replacing pure beryllium in structural elements of fusion reactors. $Be_{12}Ti$ is considered as a neutron breeder material, a structural part of the Helium Cooled Pebble Bed of the DEMO reactor. It is expected that the replacement of beryllium by $Be_{12}Ti$ will make it possible to reduce the capture of tritium in the blanket without a significant decrease in the neutronic characteristics. Unlike beryllium, beryllides have relatively recently begun to be considered for use in nuclear and thermonuclear facilities, so the radiation resistance of these compounds remains little studied. This paper presents the experimental results on effect of low temperature neutron irradiation to properties of titanium beryllide samples manufactured by industrial technology in the Ulba Metallurgical Plant (UMP, Kazakhstan). The manufactured samples before and after irradiation were analyzed by scanning electron microscopy (SEM), X-Ray diffraction (XRD), hydrostatic weighing method, dimension method and microhardness measurement by Vickers method.

1. Introduction

Titanium beryllide is a candidate material for the neutron breeder of the DEMO fusion reactor [1–4]. Titanium beryllide have a higher melting point, react less with water, and swell less under irradiation, being more suitable for use in fusion reactors [5–7].

When used in a fusion reactor, titanium beryllide will be irradiated by neutrons, which will affect its properties. And as far as we know, the initial properties of the material depend on the production technology. So, there are several different technologies of titanium beryllide production: arc-melting, hot isostatic pressing, rotating electrode method, vacuum hot pressing, spark plasma sintering [8–15]. Some of these technologies do not allow producing titanium beryllide on the industrial scale. JSC "UMP" had developed its own technology for industrial production of titanium beryllide [16,17]. Experimental data on the effect of neutron irradiation on the properties of titanium beryllide are poor [18] in contrast to metallic beryllium [19–25]. This motivated us to start working on the characterization of titanium beryllide using samples produced at JSC "UMP".

Kazakhstan has a lot of experience related to the development of nuclear and thermonuclear technologies in many areas of science: from radioecological studies at the former Semipalatinsk Test Site to reactor experiments of promising thermonuclear reactors materials [26–33].

The experiments performed today on the irradiation of titanium beryllide have shown that neutron irradiation of titanium beryllide at temperatures above 500 °C leads to swelling of no more than 10 % (volumetric). It is noted that at irradiation temperatures below 500 °C the swelling is about 3 % (volumetric). The density of beryllide hardly changes after irradiation. XRD analysis showed the presence of three phases in irradiated titanium beryllide: $Be_{12}Ti$, $Be_{10}Ti$, Be. It is observed that the presence of $Be_{12}Ti$ phase increases with increasing of irradiation temperature.

The present work shows experimental results of post reactor studies of titanium beryllide irradiated at low temperature to an integral neutron fluence of $3.9*10^{21}$ cm^{-2}. The effect of neutron irradiation on the properties of titanium beryllide is shown.

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Table 1
Characteristics of the studied samples.

Parameter	Value
Sample Type	Titanium Beryllide
Chemical formula of the sample	$Be_{12}Ti$
Sample density, g/cm^3	2.30 ± 0.01
Sample shape	Plate
Overall dimensions of the sample, mm	8x8.5x1.5
Sample weight, g	0.24

2. Methods and materials

The investigated samples are plates made of beryllium intermetallic compound. The main characteristics of the samples considered are given in Table 1, and the appearance of samples is shown in Fig. 1. The five samples were irradiated in the sealed capsules cooled by primary circuit water of the WWR-K reactor. Irradiation was in thermal fission spectrum and in the central position of the WWR-K reactor [34,35]. Irradiation parameters of the capsule with samples in detail is given in Table 2.

Diffractometric measurements were performed on a universal complex D8 ADVANCE (Bruker, Germany), Bragg-Brentano «θ-θ» imaging scheme. Imaging was performed with rotation of the sample holder at an angular velocity of 60 rpm using the Adjust control program. Phase analysis was performed in the EVA program. The ICDD: International Center of Diffraction Data (PDF2 powder database) was used for phase identification.

The surface morphology of samples was investigated by optical microscopy on Axio Observer optical microscope and by SEM on HITACHI TM4000 microscope. EDS (Brukker) X-ray spectroscopy used for elemental analysis.

Microhardness by Vickers method was measured on a PMT-3M microhardness tester.

XRD analysis, microstructure and microhardness after irradiation were performed on unpolished and polished samples. Polishing process consisted in polishing with abrasive paper, then the surface was polished with SOI (State Optical Institute) paste with a fraction of 15 μm, then diamond paste with a fraction of 1 μm was used and at the final stage the surface of the sample was polished with a silicon-based suspension with a fraction of 0.25 μm.

Density was measured by immersion in ethanol (Archimedes' principle) using the OHAUS AX224 scales. The measurement accuracy of the scales is 0.0001 g. The dimensions of the samples were measured using a micrometer with an accuracy of 0.01 mm.

3. Results

Measurements of the dimensions, mass and density of the samples were carried out five times for five samples. The density of the samples decreased by 5 % after irradiation, from (2.30 ± 0.01) g/cm^3 before irradiation and (2.18 ± 0.01) g/cm^3 after irradiation. Swelling of the titanium beryllide samples was not observed. The dimensional changes occurred within the measurement error. Tables 3–5 show the average values for the five measurements.

The shown dimensional values in Table 4 correspond to the average values from five measurements. The geometry of the samples has an irregular parallelepipedic shape, i.e. both sides of the length and width of the samples have some slope (non-parallel). This introduces

Table 2
Irradiation parameters.

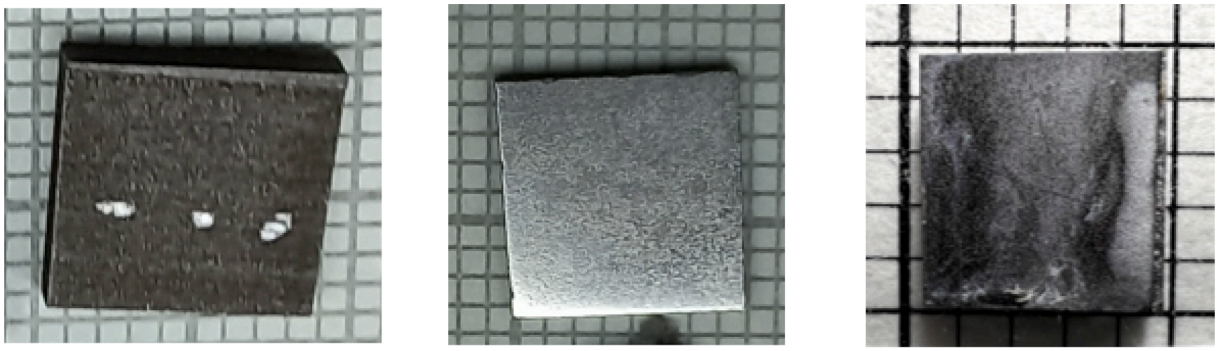
Parameter	Value
Irradiation duration, EFPD (effective full power days)	168
Accumulated thermal neutron fluence (E < 0.465 eV), cm^{-2}	8.2x10^{20}
Accumulated fast neutron fluence (E > 0.1 MeV), cm^{-2}	3.5x10^{20}
Temperature of irradiation, °C	80 ± 15
Irradiation environment	argon gas
Production helium, appm	134
Production tritium, appm	2
Damage dose, dpa	0.56

Table 3
Average measured data of samples density.

Sample	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Unirradiated	2.260 ± 0.005	2.256 ± 0.005	2.274 ± 0.005	2.256 ± 0.005	2.287 ± 0.005
Irradiated	2.230 ± 0.005	2.228 ± 0.005	2.231 ± 0.005	2.231 ± 0.005	2.227 ± 0.005

Table 4
Average measured data of samples dimensions.

Sample		Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Unirradiated	L, cm	0.869	0.885	0.880	0.871	0.874
	W, cm	0.837	0.830	0.824	0.848	0.822
	H, cm	0.161	0.155	0.157	0.156	0.156
	V, cm^3	0.1170	0.1139	0.1138	0.1152	0.1121
Irradiated	L, cm	0.902	0.893	0.891	0.898	0.872
	W, cm	0.858	0.853	0.850	0.848	0.828
	H, cm	0.163	0.164	0.167	0.163	0.167
	V, cm^3	0.1263	0.1251	0.1263	0.1239	0.1204



a b c

Fig. 1. Appearance of sample: a - before grinding (marked), b - after grinding, c - after irradiation.

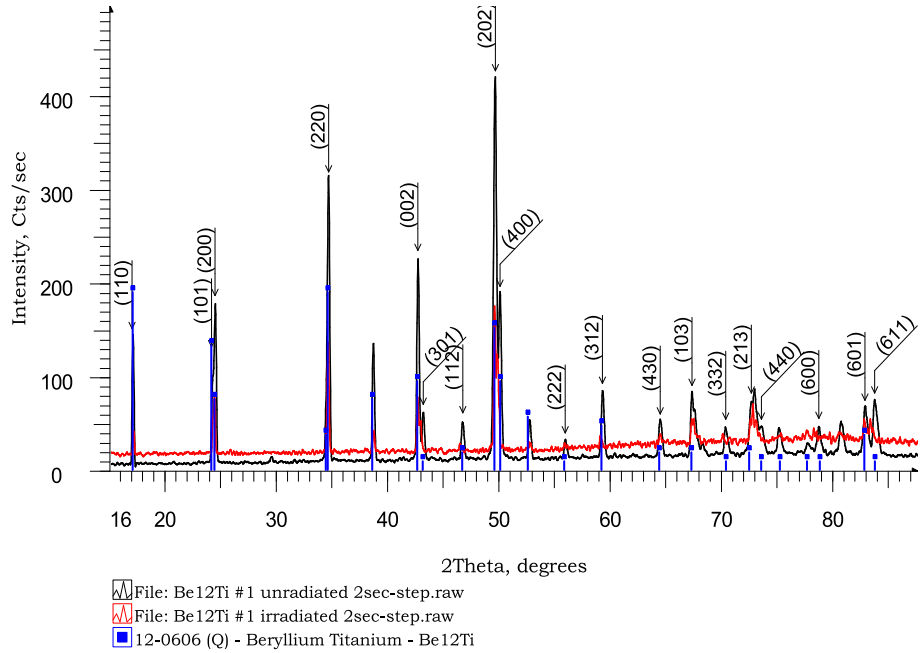


Fig. 2. X-ray pattern image of unirradiated $Be_{12}Ti$.

additional error in determining the volume of the samples.

The average value of swelling determined from the measured values of the density of the samples by the immersion method, according to formula (5) from [36], was S = 1.67 %. Whereas the average value of swelling determined from the measured values of sample sizes, according to formula (6) from [38], was S = 8.03 %. The authors believe that the more accurate value of swelling is the one measured by immersion method because of the irregular shape of the samples. Swelling of titanium beryllide is related to the production of radiogenic helium on beryllium, which at low temperature has diffusive mobility, due to which gas atoms do not migrate and cause distortions of the crystal lattice dimensions [37].

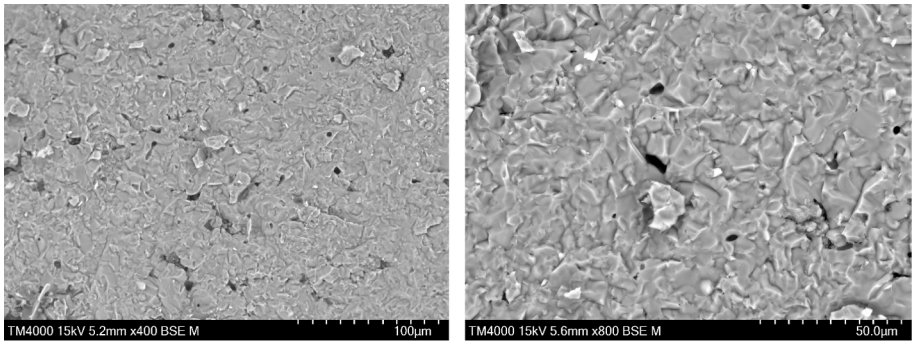
According to the data given in [36–39], the swelling of beryllium ranges from 0.1 % to 4.3 % and depends on the dose and irradiation temperature. In the listed works the extreme fluence of neutrons was achieved.

XRD analysis showed that titanium beryllide samples before irradiation and after irradiation are monophasic, the parameters of tetragonal volume-centered crystal lattice of the sample: a=(7.2818 ± 0.0078) Å, c= (4.2439 ± 0.0064) Å. However, another peak appears between reflexes (002) and (301), the peak from plane (103) exhibits a doublet structure and (213) is a triplet. The crystallite sizes estimated from Scherrer's formula for the unirradiated sample are 42 nm and 60 nm for the irradiated sample.

The peak (110) is well pronounced in the unirradiated sample and loses 70 % of its intensity in the irradiated sample. The next detectable doublet (101) and (200) have inverted intensities relative to the card data, which is most likely due to the influence of lattice deformation during mechanical processing of the samples and is brightest in the unirradiated sample. Reflex (220) has only a slight asymmetry on the left wing from reflex (211), which does not separate from (220) even for the unirradiated sample, but is shifted toward smaller angles for the irradiated sample. The peaks (002) and (301) in the vicinity of angles 42–43°, as well as (202) and (400) at angles 49–50° and (601) and (611) at 83–84° for the irradiated sample are shifted toward each other relative to the positions in the unirradiated sample and relative to the card positions, as can be easily seen from the blue bar diagram labels (Fig. 2).

Before the microstructural studies, the sample was mechanically destroyed, the investigated section was irradiated with a microscope electron beam, and the structure of the material was determined. The effect of reactor irradiation was investigated on the surface of microsheets of the initial and irradiated samples.

Fig. 3(a, b) shows microphotographs of the chipped surface. The microstructure of the sample is dense, formed by well-formed grains. The crystals are heterogeneous in size and shape, ranging in size from 5



a b

Fig. 3. Microphotographs of the chipped surface at different magnifications.

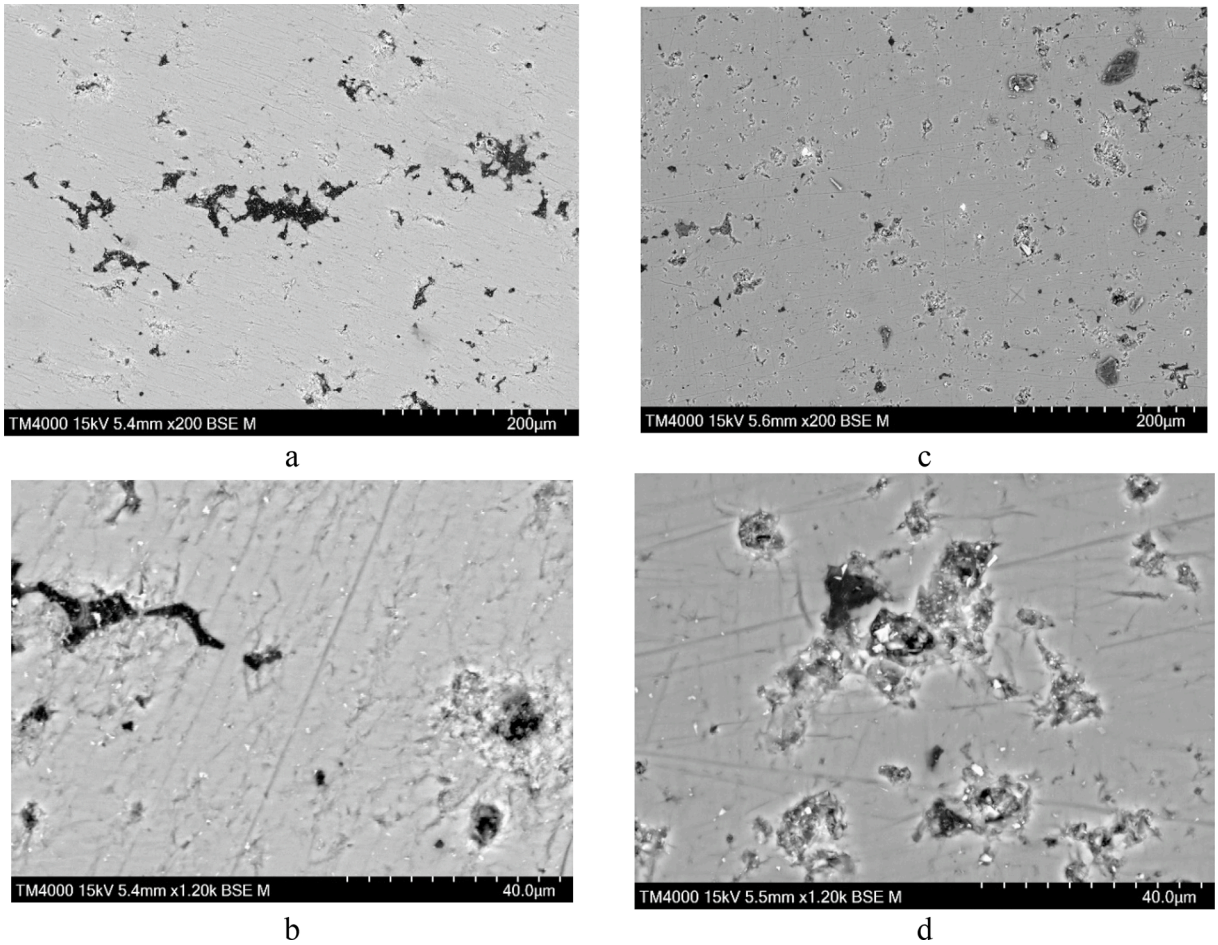


Fig. 4. Microphotographs of the polished surface at different magnifications: a, b - before irradiation, c, d - after irradiation.

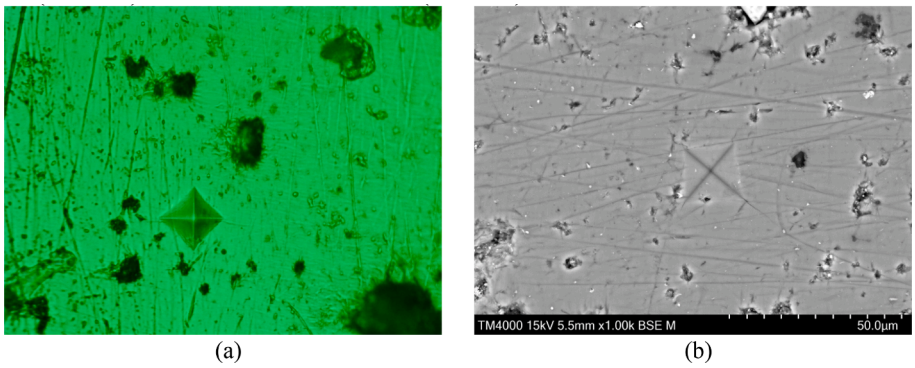


Fig. 5. Microhardness measurement location: (a) – optical image; (b) SEM image.

to 15 µm. The grains are rather uniformly distributed among themselves in the sample volume, well contact with each other, forming aggregates (Fig. 3a). The structure contains traces of binding materials in the form of oxides (light inclusions up to 2 µm in size, Fig. 3b). The material is characterized by the presence of intra-grain porosity. Pores of rounded shape, 0.5–5 µm in size (Fig. 3b), are observed in single quantities, not more than one pore in one grain. Intergranular pores occur many times less, have larger size and arbitrary shape.

Electron-microscopic investigation of the microslip also shows a uniform structure of the material (Fig. 4). The dark areas of arbitrary shape in Fig. 4a are the cavities formed when the sintered aggregates were scratched out during mechanical grinding. Their average length is up to 200 µm with a width of 20–30 µm. Also, traces of mechanical processing (scratches) and boundaries of sintered $Be_{12}Ti$ powder granules are observed on the surface (Fig. 4b). Pores on the microslip are less noticeable, but their size and number correlate with the data of the study of the chipped surface. Residual materials in the form of oxides are also present on the surface of the microslip.

After neutron irradiation, the size and number of aggregates precipitated during mechanical grinding decreases more than 2 times (Fig. 4c, d). The boundaries of sintered $Be_{12}Ti$ powder granules are not observed in the irradiated material. The changes of porosity parameters on the microslip are numerically difficult to estimate, but the tendency to decrease their sizes is noticeable. The listed changes in the shape and

size of structural components of the material can be evidence of the radiation swelling process.

The microhardness of titanium beryllide samples was measured on five samples before irradiation and after irradiation (Fig. 5). The average value of microhardness before irradiation was (2330 ± 70)HV and after irradiation was (1218 ± 32)HV.

4. Conclusion

As a result of the conducted studies on the effect of low-temperature irradiation up to integral neutron fluence $3.9*10^{21}$ cm^{-2} on the properties of titanium beryllide, the following conclusions can be drawn:

- The density of titanium beryllide samples decreased from 2.27 to 2.23 g/cm^3.
- Swelling is 1.7 % was observed.
- XRD analysis showed that the samples of titanium beryllide before irradiation and after irradiation are monophasic, the parameters of tetragonal objemocentered crystal lattice of the sample: a=(7.2818 ± 0.0078) Å, c= (4.2439 ± 0.0064) Å. The crystallite sizes estimated by Scherrer's formula for the unirradiated sample were 42 nm, and for the irradiated sample − 60 nm.
- There is an evolution of the microstructure of the samples.
- The microhardness of the samples after irradiation changed from (2330 ± 70)HV to (1218 ± 32)HV.

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CRediT authorship contribution statement

A. Shaimerdenov: . **A. Akhanov:** . **Sh. Gizatulin:** . **A. Nessipbay:** Investigation. **B. Shakirov:** Investigation. **S. Askerbekov:** . **T. Kulsartov:** . **I. Kenzhina:** Investigation. **A. Larionov:** Investigation. **S. Akayev:** . **S. Udartsev:** .

Declaration of competing interest

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Data availability

Data will be made available on request.

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