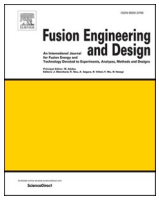
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Study of interaction of hydrogen isotopes with titanium beryllide ($Be_{12}Ti$)

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ABSTRACT

Titanium beryllide $Be_{12}Ti$ is not only a prospective material for the blankets of future fusion reactors as a neutron multiplier, but can also be used in other areas of the nuclear industry, such as reflectors in fission reactors, in rocket and space technology. This paper presents the results of experimental thermal desorption studies (TDS) of samples of monolithic titanium beryllide $Be_{12}Ti$ produced by "Ulba Metallurgical Plant" JSC (Kazakhstan). To conduct the thermal desorption spectroscopic studies, titanium beryllide $Be_{12}Ti$ samples were pre-saturated in deuterium medium at atmospheric pressure at temperatures of 873 K and 973 K. The TDS measurements of the deuterium release spectrum in the temperature range of 293 K-1473 K at linear heating rates of 10 and 20 K/min were conducted. Experimental data on the parameters of the interaction of deuterium with titanium beryllium $Be_{12}Ti$ have been established. Based on a preliminary analysis of the experimental data, a mechanism of deuterium release processes from the samples under linear heating is proposed.

1. Introduction

Beryllide is an intermetallide compound of beryllium with other metals such as zirconium, tantalum, titanium, nickel or cobalt. It is a solid metal-like material significantly different from these metals and has unique properties, especially in its resistance to oxidation.

Brush Wellman Inc. (USA), KIT (EU), Ulba Metallurgical Plant JSC (Kazakhstan) are the main manufacturers of beryllides. Although the manufacture technologies and properties of beryllides are well known, however, there is lack of experimental data on the behaviour of these composite materials under the influence of various operating factors in nuclear, thermonuclear and hydrogen energy. Beryllides can be used as a neutron breeder instead of metallic beryllium in fusion reactor blankets, in nuclear reactor reflectors, and also as materials for hydrogen storage in the hydrogen and energy industry.

Interest in the study of the interaction of hydrogen isotopes with beryllides is related to the accumulation of tritium and helium in the material, formed as a result of nuclear reactions during neutron irradiation and is driven by the need to understand the processes arising from such interactions. As a radioactive isotope, tritium poses an additional safety problem due to its possible release during operation, or a barrier to further beryllium recycling. Therefore, a detailed study of its accumulation and release is essential. In particular, it is important to find out which process controls the release of tritium: the binding of tritium by traps or diffusion from the bulk of the material.

A new information of the mechanisms and processes caused by the interaction of hydrogen isotopes with such materials will allow further prediction of the terms and operating conditions of beryllide-based products.

Under the conditions of neutron irradiation of pure beryllium, there is a degradation of its properties due to a significant gas swelling effect upon the impact of irradiation and high temperature. The use of composite materials such as beryllides allowed to significantly reduce the negative effects of irradiation while maintaining the operational features of products made of such beryllium-containing materials, and thereby increase their lifetime and expand operating parameters. amongst the intermetallic compounds in the Be-Ti system, $Be_{12}Ti$ stands out as the most suitable material for use in fusion and nuclear plants in

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terms of its key characteristics, according to research by Brush Wellman Inc. (USA) [1].

Compared to pure beryllium, titanium beryllide $Be_{12}Ti$ has a high melting point (1866 K), lower activation and higher corrosion resistance, according to [2,3]. Titanium beryllide, meanwhile, has a high enough beryllium content (69.3% wt.) for efficient neutron breeding. Moreover, an important property of titanium beryllide is its compatibility with structural materials. Thus, the authors of [2] experimentally found that the compatibility between $Be_{12}Ti$ and SS316 is an order of magnitude better than that of Be and SS316.

The swelling of titanium beryllide $Be_{12}Ti$ caused by neutron irradiation is much less than that of pure beryllium. Thus, in [4] it is shown that the value of swelling of $Be_{12}Ti$ titanium beryllide disk specimens after neutron irradiation at temperatures of 600 K, 673 K and 773 K and before obtaining a neutron fluence of 4×10^{20} n/cm^2 (E_n>1 MeV) is 0.8, 1.0 and 0.5%, respectively. In similar experiments with neutron irradiation of beryllium disk specimens the maximum swelling was 60% [5]. A review [6] compares the swelling characteristics of beryllium and titanium beryllide at high neutron fluences. The results of the study confirm that the Be-7Ti composition has much less swelling than beryllium Be. After irradiation at 703 K - 1023 K to a neutron fluence of $1.82 \cdot 10^{22}$ cm^{-2} (E>1 MeV), the swelling of Be is about 50%, whereas for Be-7at.% Ti it is 2.7%. This difference is explained by a more intensive process of bubble and pore formation in beryllium than in titanium beryllide.

As for interaction with hydrogen isotopes, it was shown in [7] that tritium release peaks from titanium beryllide samples irradiated at different temperatures were observed mainly in the temperature range 873 K – 973 K in contrast to beryllium, for which main tritium release peaks were observed at temperatures above 1173 K.

It is important to conduct comparative studies of titanium beryllide and pure beryllium in order to determine the effect of neutron irradiation on the mechanisms of tritium release from these samples.

Therefore, for example, post-irradiation TDS study of irradiated in the reactor at different temperatures Be-7Ti and Be samples showed that the lowest tritium retention in Be-7Ti samples was detected at the two highest irradiation temperatures of 940 K and 1040 K, where it was 1.0% and 0.5%, respectively. By comparison, the retention of tritium in Be samples at the same irradiation temperatures was 48% and 34%, respectively [8].

The release of tritium from irradiated titanium beryllide $Be_{12}Ti$ at high temperature depends on the release of tritium on the surface, and at low temperature depends on the diffusion of tritium into the material, with the small formation of an oxide (BeO) can affect the ability to release tritium [9].

The presented experiments are the initial stage of the study of beryllides manufactured by domestic technologies. The research is aimed to study the parameters of interaction of hydrogen isotopes with beryllides in order to establish the mechanisms of diffusion and hydrogen isotope release during degassing in a wide temperature range.

In addition to monolithic $Be_{12}Ti$ titanium beryllide, UMP JSC manufactures other poorly studied compositions (e.g., powders of $TiBe_2$, $ZrBe_2$, $TaBe_2$, $NbBe_2$ and $CrBe_2$). The crystal structure of some compounds, e.g. for $Be_{17}V_2$, $Be_{10}Ti$, $Be_{13}Ti_2$ and Be_5Ti_4 specimens, has not yet been clarified [3].

This paper presents experimental thermal desorption studies of titanium beryllide saturated in a deuterium medium at 100 kPa at two temperature values, 873 K and 973 K for 2 h. The spectra of gases in the chamber of the experimental setup were recorded during linear heating. As a result of the experimental data curation, the values of diffusion coefficients, diffusion activation energy and concentration of deuterium in titanium beryllide were obtained.

2. Experimental part

2.1. Materials, samples

Samples of titanium beryllide $Be_{12}Ti$, manufactured by Ulba Metallurgical Plant JSC, are presented for investigation. Titanium beryllide $Be_{12}Ti$ samples were in plate form, allowing to conduct their preparation for characterization for mechanical properties, structure by optical surface metallography, phase composition and others.

The size of the samples must be sufficient to achieve such a deuterium concentration at saturation so that the partial pressure of the released gases matches the sensitivity of the measuring system of the setup. Milled titanium beryllide samples were also investigated to study the influence of material geometry on the diffusion parameters of deuterium release.

The samples were square plates ~8 × 8 × 1.5 mm in size and weighing about 0.234 g (Fig. 1).

Selected titanium beryllide samples were saturated in deuterium at atmospheric pressure at 873 K, 973 K within 2 h before TDS experiments. Saturation of titanium beryllide samples with deuterium was carried out in an ad hoc setup (ampoule) with a pillow, filled with deuterium at atmospheric pressure at a sample temperature of 973 K within for 2 h. Thereafter, the samples were cooled in a deuterium medium to a temperature of 293 K and kept in a deuterium medium within for 12 h. After that, the samples were uploaded from the ampoule and sealed in polyethylene bags.

The MBe3 sample, saturated at 873 K, was milled (Fig. 2) and a part of the sample with a total mass of 0.0863 g was prepared for TDS experiments.

The MBe3 sample was milled to determine the features of the deuterium release mechanisms, associated with the limiting stage of deuterium desorption from titanium beryllide. For this purpose, it is planned to compare the desorption spectrum of deuterium from samples with significantly different linear dimensions, i.e. monolithic and the milled one samples. If the TDS spectrum show a deuterium peak in the milled sample, similar to the peak of a monolithic one, it could be concluded with confidence, that the deuterium release is mainly limited by its release from traps.

Preliminarily, the primary characterization of the test samples was carried out, similar to [10]. A monolithic titanium beryllide sample was used for characterization. The microstructure of the polished titanium beryllide sample $Be_{12}Ti$ is shown in Fig. 3. It is formed by grains with an average size of about 15 μm.

X-ray phase analysis showed that the crystal structure of $Be_{12}Ti$ has a tetragonal lattice with parameters of $a = b \approx 7{,}3$ Å and $c \approx 4{,}2$ Å.

X-ray diffraction pattern of titanium beryllide $Be_{12}Ti$ have shown in Fig. 4. There is a small amount of beryllium oxide phase BeO (Fig. 5), the remaining peaks mainly correspond to the $Be_{12}Ti$ phase, in addition, there are also particles of pure beryllium (Fig. 6)

Fig. 5 shows a SEM image of the $Be_{12}Ti$ surface, at a magnification of × 1200, showing beryllium oxide particles BeO, marked in white, with crystallite sizes of ~2 - 6 nm.

Fig. 6 shows an image of a crack on the surface of a $Be_{12}Ti$ sample, showing a black area, which are particles of Be.

The initial surface of the sample was formed in the EDM process of

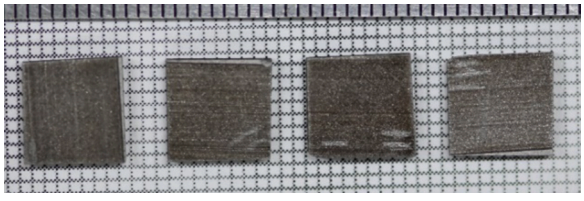


Fig. 1. Monolithic titanium beryllide samples $Be_{12}Ti$ after deuterium saturation.

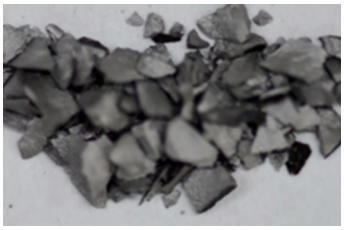


Fig. 2. Milled part of sample MBe3 (total weight 0.0863 g).

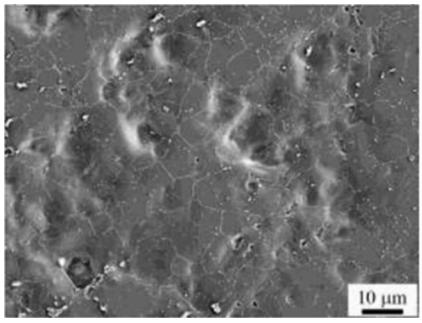


Fig. 3. Microstructure of titanium beryllide $Be_{12}Ti$.

the ingot. This surface is therefore highly inhomogeneous, with an arithmetic average roughness $R_a = 3$ µm. The surface profile is shown in Fig. 7.

Scanning electron microscopy allows a closer look at the elements of the initial surface structure of the sample after the EDM (Fig. 8), where strong inhomogeneity is observed at a magnification of × 500.

During the TDS experiments, the samples were saturated in a deuterium medium in this initial state.

2.2. TDS setup

To conduct thermal desorption experiments, an experimental setup was used (Fig. 9), which makes it possible to record the TDS spectra of gases in the measuring volume. Mass-spectrometric system of gas analysis of an experimental setup includes RGA-100 quadrupole mass spectrometer, which makes it possible to measure the partial pressures of gases with mass numbers from 2 to 100 a.m.u. The vacuum system is based on turbomolecular and magnetodischarge pumps, providing up to 10^{-5} Pa of residual pressure in the system.

The experimental setup is equipped with stationary heaters to heat the entire system to 420–453 K in continuous pumping mode to degas the vacuum system elements.

A molybdenum crucible with external heating is used to provide a programmable temperature mode for the sample.

The technical specifications of the experimental setup are shown in Table 1.

2.3. Methodology of experiments

The method of thermal desorption spectroscopy (TDS) is based on the study of the kinetics of gaseous diffusant release from the sample in its programmed heating mode. The experiment records either a decrease in the amount of diffusant in the sample (integral version) or an increase in its concentration in the carrier gas (or vacuum). The latter case

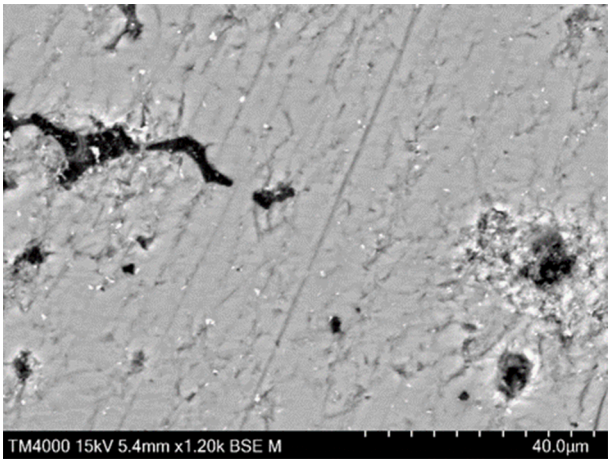


Fig. 5. SEM image of the $Be_{12}Ti$ surface, × 1200.

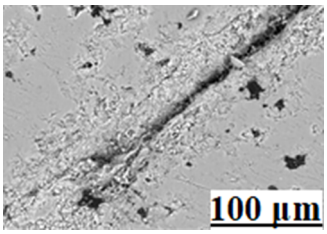


Fig. 6. SEM image of a crack on the $Be_{12}Ti$ surface, × 400.

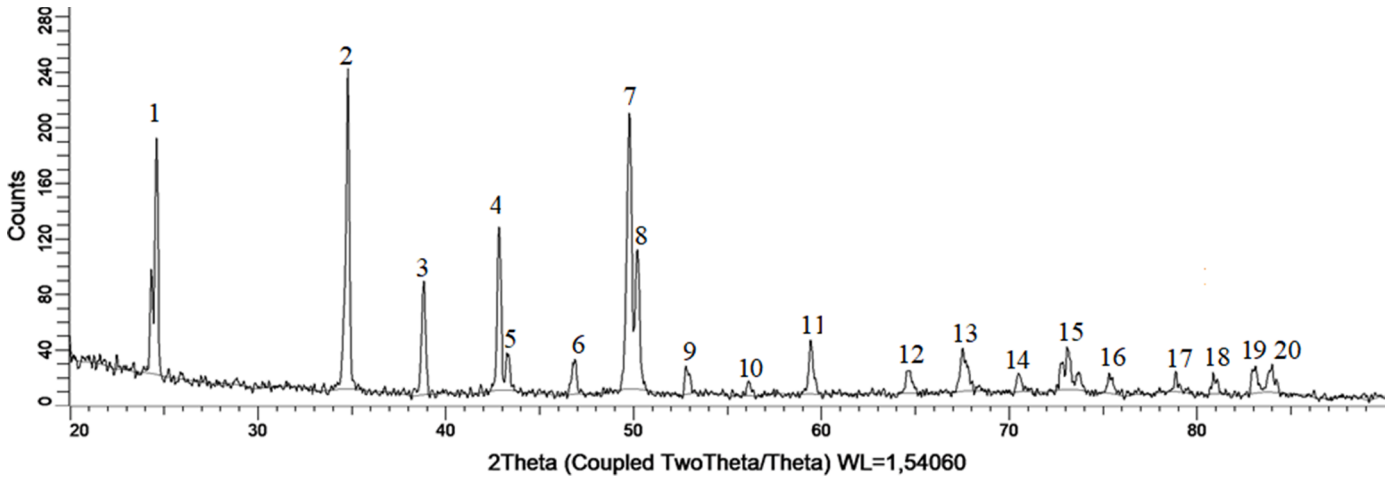


Fig. 4. X-ray diffraction pattern of the $Be_{12}Ti$.

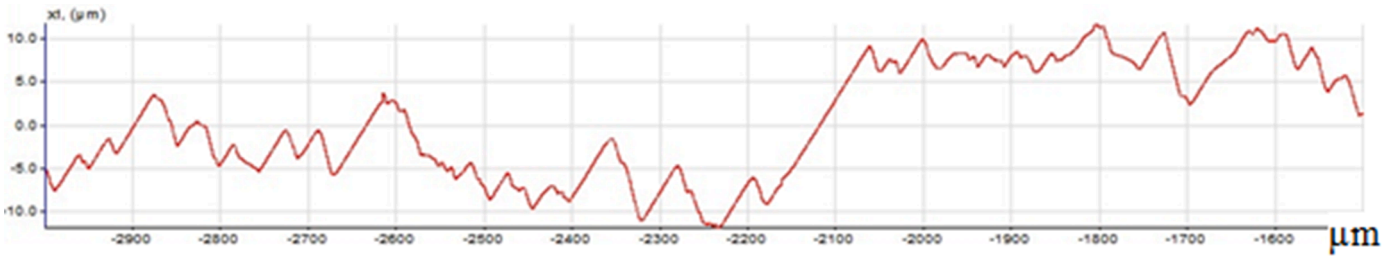


Fig. 7. $Be_{12}Ti$ surface profile before grinding.

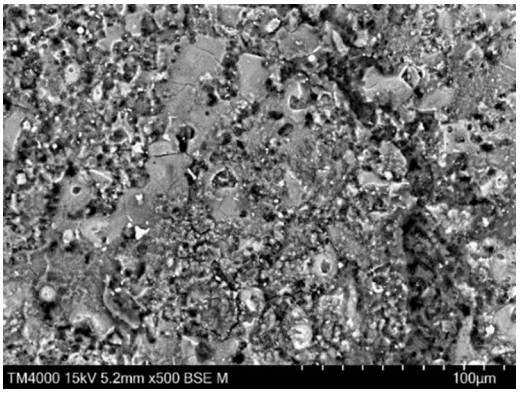


Fig. 8. SEM image of $Be_{12}Ti$ sample surface before grinding, × 500.

measures the change in the amount of gas released from a solid at time t (integral option) or the change in time of gas flow from a sample (differential option).

The differential option of the TDS method is the most suitable to study titanium beryllide samples. Experimental data from TDS spectrum should be curated according to the model representations, which describe the deuterium diffusion process, considering their capture and release from various traps. The accumulation of hydrogen isotopes in the material volume and its desorption depend to a large extent on the parameters of the hydrogen traps. The binding energy of hydrogen isotopes with traps and the concentration of traps are most often determined by fitting the calculated thermal desorption spectrum to the experimental ones [11].

To analyse experimental data, the model is usually used that consider both diffusion and deuterium release from traps (i.e. bound states) during the linear heating [12].

3. Results and discussion

3.1. TDS tests and analysis

Experimental data were obtained for the spectra of gases evolved in the chamber volume for MBe1 and MBe2 samples, saturated under the same conditions and at heating rates of 20 K/min and 10 K/min, respectively. Gases with mass numbers of M3 (HD) and M4 (D_2) were allocated on the spectrum (Fig. 10), and then their temperature dependences were obtained and summed up the deuterium atoms (MD) in the HD and D_2 molecules.

The temperature dependences of two samples, heated at different speeds were compared. As can be seen in the temperature dependence plot (Fig. 11), the flux of deuterium from the samples as the temperature increases is described by the curve with a maximum, asymmetrically extended towards lower temperature. Moreover, the temperature of the

Table 1
The technical specifications of the experimental setup.

Parameter	Value
Operating temperature range	from 293 K up to 1573 K
Residual pressure in the measuring path at a crucible temperature of 1573 °C	10^{-4} Pa
Accuracy of automatic temperature maintenance relative to the setpoint	± 0,5 K
Range of sample heating rates	from 2 up to 50 K/min

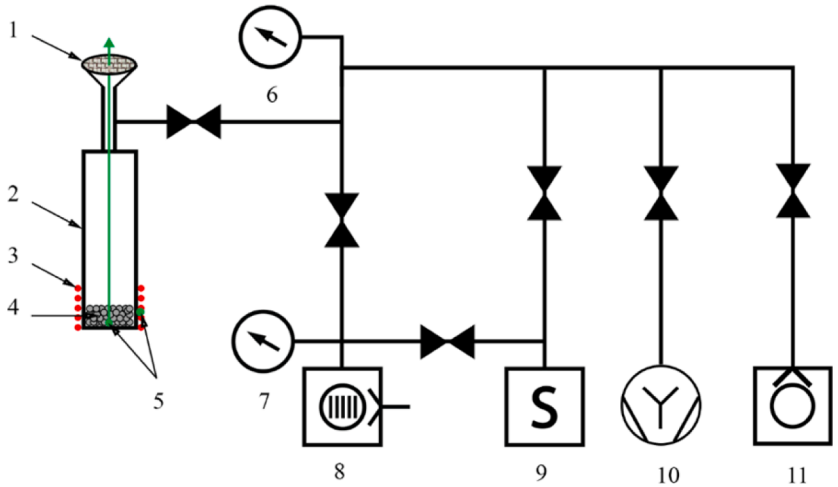


Fig. 9. Scheme of the experimental setup for TDS measurements.
1 – pressure seal for thermocouple; 2 - experimental cell; 3 - heater; 4 - samples; 5 - thermocouples; 6–7 - pressure sensors; 8 – turbomolecular pump; 9 – mass spectrometer; 10 – magnetic discharge pump; 11 – fore pump.

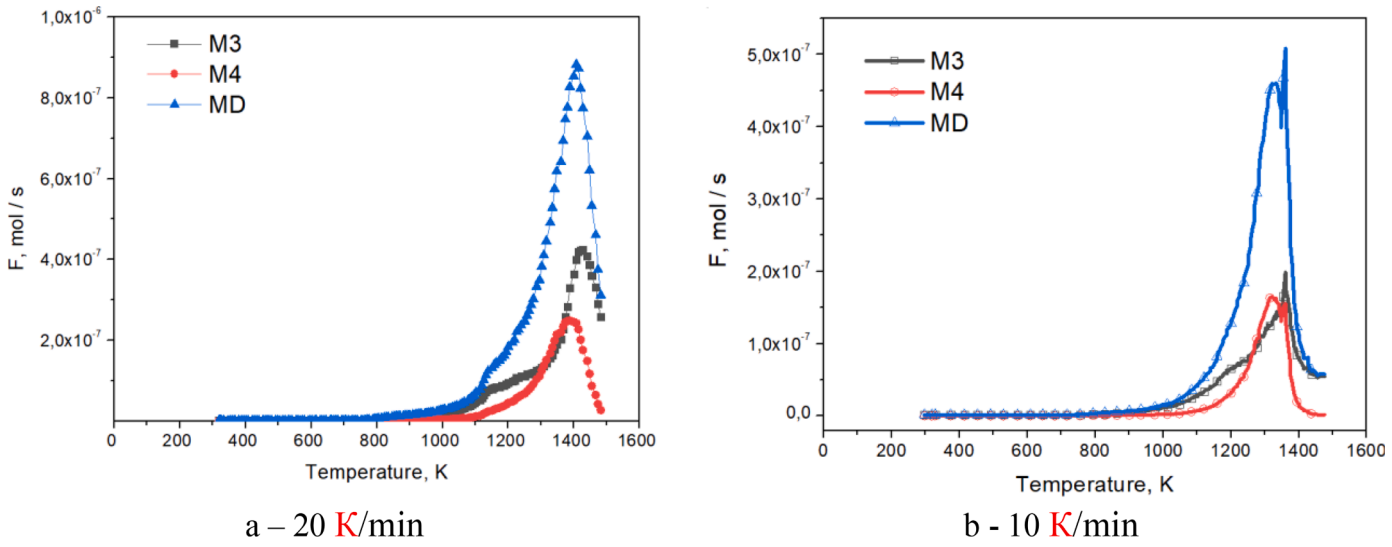


Fig. 10. Experimental data of gas release TDS during linear heating of titanium beryllide sample $Be_{12}Ti$ with different heating rates.
a – dependence of HD and D_2 release from the $Be_{12}Ti$ sample under linear heating at a rate of 20 K/min for the MBe1 sample; b - dependence of HD and D_2 release from the $Be_{12}Ti$ sample under linear heating at a rate of 10 K/min for the MBe2 sample; MD – total number of deuterium atoms.

maximum gas release shifts to a higher temperatures with an increase in the heating rate of the sample, which agrees well with the literature [13, 14]. Peaks of deuterium release at heating rates of 10 and 20 deg/min correspond to the temperatures of 1340 K and 1410 K. The deuterium concentration in the samples equal to ~ 5E-4 mol/g, was calculated from the time dependencies of the TDS spectrum and the mass of the samples.

Experimental data of temperature dependence of deuterium release from milled sample MBe3 with total mass 0.0863 g (Fig. 12) show the beginning of deuterium release at lower temperatures around 750 K than for monolithic sample, and during heating, a superposition of several peaks is observed in the spectrum, which is possibly due to the different length of the deuterium diffusion path from the volume to the surface due to the different sizes of the fragments of the milled sample.

The TDS spectrum of deuterium release from the milled sample and a comparison of the temperature dependence of deuterium fluxes from monolithic and milled samples of titanium beryllide $Be_{12}Ti$ at the same heating rate of 20 K/min are shown in Fig. 12

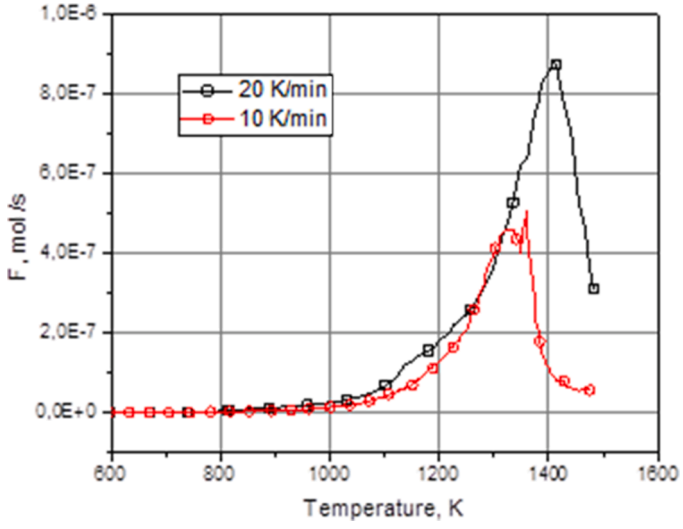


Fig. 11. Temperature dependence of deuterium fluxes from monolithic samples of $Be_{12}Ti$ titanium beryllide at heating rates of 10 and 20 K/min; the deuterium concentration in the samples ~ 5E-4 mol/g.

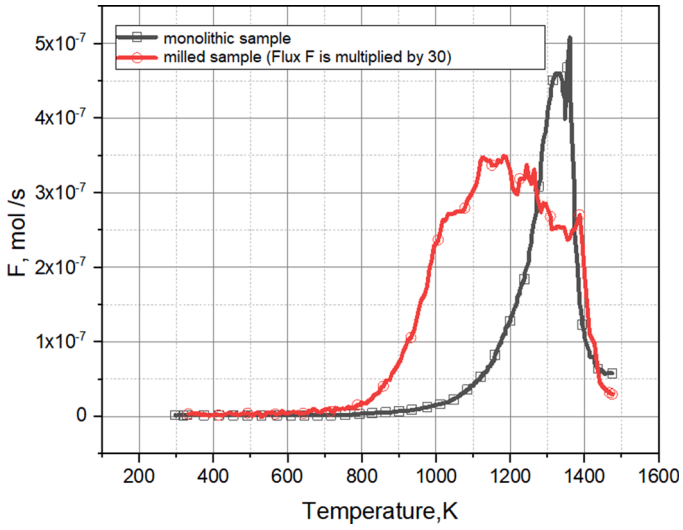


Fig. 12. Comparison of temperature dependences of deuterium fluxes from monolithic and milled samples of titanium beryllide $Be_{12}Ti$ at a heating rate of 20 K/min.

However, despite the fact that the nature of deuterium release has the form of diffusion peaks for monolithic samples (Fig. 11), the diffusion mechanism is apparently not the only mechanism that determines the gas release process. This is evidenced by the almost identical initial growth front of the gas release flux as the sample temperature increases for different heating rates. Such a feature of gas release is characteristic of the process, caused by the release of deuterium from traps, followed by diffusion to the surface and desorption from the sample.

A comparison of the deuterium desorption spectrum from the monolithic and milled samples shows a different deuterium desorption pattern (Fig. 12), which indicates that the assumption of deuterium release from traps as the limiting stage of the gas release process has not been confirmed, i.e. the gas release process is defined by several stages.

3.2. *Simulation of diffusion parameters*

To analyse the experimental data, we used a model that consider both diffusion and deuterium release from traps (i.e., bound states)

during linear heating.

The equations for the diffusion model in the presence of trapped gas sources are defined by the expression:

$$\frac{\partial C}{\partial t} = D(t)\cdot\frac{\partial^2 C}{\partial x^2} + k_1\cdot C_{trap}.$$
$$\frac{\partial C_{trap}}{\partial t} = -k_1\cdot C_{trap}. \quad (1)$$

where C – deuterium concentration; C_{trap} – deuterium source concentration; k_1 - deuterium release constants from traps.

In addition, parameters from time and temperature are determined, that consider the release of deuterium from the bound state:

$$D(t) = D_0\cdot\exp\left(-\frac{E_D}{RT}\right),$$
$$k_1(t) = k_{10}\cdot exp\left(-\frac{E_T}{RT}\right), \quad (2)$$
$$T = T_0 + \beta\cdot t.$$

where T – metal temperature, K; β – sample heating rate; E_D, E_T – activation energies of diffusion and release of deuterium from the trap, respectively, kJ/mol; R – gas constant; k_1 - constants of deuterium release from the bound state in the sample.

Initially, based on Eq. (1), we obtained data on the diffusion parameters, assuming that there are no sources of deuterium C_{trap}=0. The concentration of the diffusant in the metal $C_0(x, t = 0)$ is known from the initial conditions. As an approximation of the model, the boundary conditions of the type I satisfying the conditions of continuous pumping of the diffusant were applied:

$C(x = 0,\ t) = 0,\ C(x = l,\ t) = 0$, where l – sample thickness.

Deuterium spectra for monolithic samples were simulated. Despite a good description of the individual peaks (Fig. 13) within a diffusion-only model (C_{trap}=0), this description of the entire TDS series for different heating rates was not accurate enough.

The obtained parameters of the effective diffusion coefficient (D_0) and activation energy (E_D) are presented in Table 2. These values were overestimated and were in line with literature data on the diffusion of bubble gas complexes in pure beryllium [5]. Considering that there is significant spread E_D for beryllides with different titanium contents, and that E_D is an effective value that includes the activation energies of all deuterium release processes from the sample. Thus, for beryllium, for example, both the experimental and theoretical values of the activation energies of deuterium desorption are $E_{ef} \approx$ 2,0–2,7 eV [12].

In further modelling of the deuterium spectrum for monolithic samples, we assumed that the defining stage of deuterium release is associated with its release from the bound state in the volume of the sample, followed by "rapid" diffusion to the surface and its desorption. Under this assumption, we were able to describe quite well the set of TDS spectra (Fig. 14) and obtain expression (3) for the deuterium release constant from the bound state in the sample:

$$k_1(t) = 22,000\ \left(\frac{1}{s}\right)\cdot\exp\left(-\frac{18,500,0\ \left(\frac{J}{mol}\right)}{RT}\right) \quad (3)$$

The effective desorption energy was also estimated from the TDS results, according to the calculation method used in [8] and justified in [15], which was 4.1 eV. This value was almost equal to the desorption energy obtained by the authors [8] for the desorption of tritium from beryllium irradiated at 700 K.

Here it should be noted that all obtained model parameters have the meaning of effective parameters and do not claim to describe the real mechanism of deuterium release from titanium beryllide samples. Nevertheless, they can be used in a comparative analysis with other

Table 2
Effective diffusion parameters for deuterium in titanium beryllide.

Effective diffusion coefficient, D_0, m^2/s	Diffusion activation energy, E_D,
10 - 60	260 – 280 kJ/mol (2.7–2.9 eV)

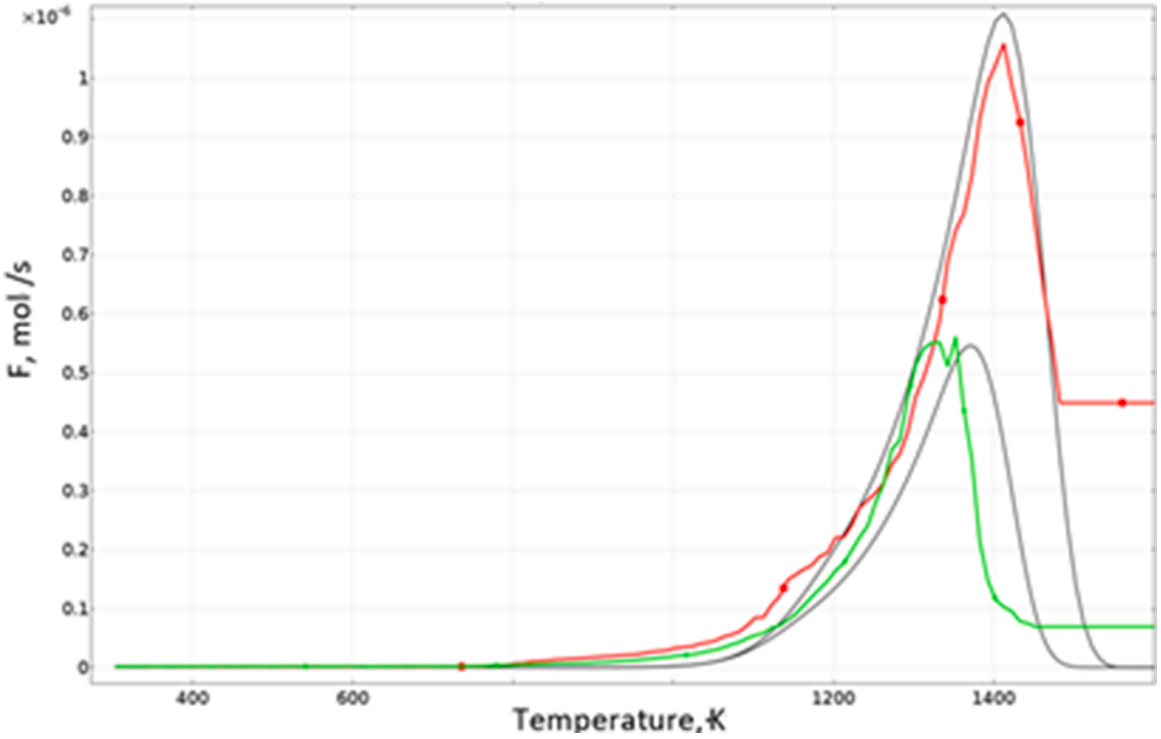


Fig. 13. Simulation of the gas release curve, considering only the diffusion process of deuterium release from $Be_{12}Ti$ titanium beryllide sample at different heating rates: — experiment, 20 K/min — experiment, 10 K/min — calculation.

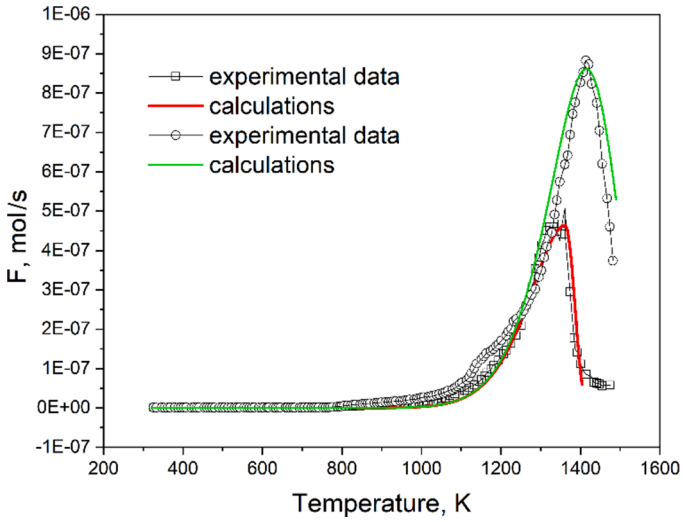


Fig. 14. Simulation results assuming that the defining stage of deuterium gas release is its release from the bound state in the sample volume, followed by "rapid" diffusion to the surface and it's desorption.

experimental data.

4. Conclusions

The parameters of interaction of deuterium with samples of monolithic titanium beryllide $Be_{12}Ti$ manufactured by "Ulba Metallurgical Plant" JSC (Kazakhstan, saturated in a deuterium medium at atmospheric pressure at a temperature of 973 K were studied.

TDS measurements in the temperature range of 293–1473 K at linear heating rates of 10 and 20 K/min were conducted.

The analysis of the TDS spectrum was performed using both a diffusion model and a model of a complex process with deuterium release from traps, diffusion and subsequent desorption.

Under the framework of the diffusion model, the description of the complete series of TDS for different heating rates was not sufficiently accurate. The obtained values of the diffusion coefficients and activation energy were overestimated and corresponded to the literature data on the diffusion of bubbles of gas complexes in pure beryllium

Under the assumption that the defining stage of deuterium gas release is associated with its release from the bound state in the sample volume, followed by "rapid" diffusion to the surface and it's desorption, we were able to describe quite well the set of TDS spectra and obtain an expression for the constant of deuterium release from the bound state in the sample

The mechanism of deuterium retention in the bound state in the sample volume can be associated with both the interaction of titanium with deuterium and the formation of an oxide layer on the grain surface, which is destroyed in the process of recrystallization while heating [7]. For a more profound insight of the interaction of deuterium with titanium beryllide, additional experimental studies of the microstructure of samples are needed.

Here it should be noted that all obtained model parameters have the meaning of effective parameters and do not claim to describe the real mechanism of deuterium release from titanium beryllide samples. Nevertheless, they can be used in a comparative analysis with other experimental data.

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

No data was used for the research described in the article.

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