

Kinetics of gas release from titanium beryllide irradiated to different neutron fluences

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ABSTRACT

The binary intermetallic compound of beryllium and titanium (Be₁₂Ti) is an advanced material for DEMO-type fusion reactors as a neutron multiplier in breeder blankets. Titanium beryllide has a high melting point (~1873 K), high oxidation resistance and low swelling, which makes it attractive for use in fission and fusion reactors. The study of its behavior in a radiation field is of utmost importance from the point of view of practical application of titanium beryllide in nuclear installations.

Based on this, at the Institute of Nuclear Physics (Kazakhstan), R&D is underway to characterization of industrial fabricated titanium beryllide (Be₁₂Ti) at JSC “Ulba Metallurgical Plant” (Kazakhstan). Titanium beryllide samples were irradiated in the WWR-K reactor core to accumulate two integral neutron fluences of $1.6 \times 10^{25} \text{ m}^{-2}$ and $2.3 \times 10^{25} \text{ m}^{-2}$, in an inert environment and at low temperature to minimize gas migration from the samples.

The present work presents the results of post reactor studies to determine the amount of generated tritium and helium in irradiated Be₁₂Ti samples. The studies were carried out by thermodesorption spectrometry (TDS) method with mass-spectra control of gas composition. Mass spectra of gas extraction from titanium beryllide irradiated to different neutron fluences and heated up to 1173–1223 K at different heating rates (5, 10, 20 K/min) are presented. The kinetic parameters of tritium desorption from Be₁₂Ti were determined, and the activation energy was estimated for each fluence achieved.

1. Introduction

Titanium beryllide (Be₁₂Ti), due to its physical and chemical properties, is currently the main candidate material for the neutron breeder of the DEMO fusion reactor blanket to replace pure metallic beryllium [1–5]. The operating conditions of the blanket material in a fusion reactor are high temperature and radiation field, mainly neutron [4,5]. Due to which the blanket material must be resistant to these external factors.

There are several methods for producing titanium beryllide, the main ones being plasma sintering, arc-melting, hot isostatic pressing, powder metallurgical. Due to the fact that the properties of the material, among other things, depend on the production method, its characterization

under operating conditions is relevant and in demand. An important specific characteristic of titanium beryllide is the gas evolution of tritium and helium, which are produced during interaction with neutrons and affect its service life [6–9].

Neutrons interact with beryllium through nuclear reaction channels ($n;2n$) ($\gamma;n$). In addition, for neutrons with energy $E \geq 0,71 \text{ MeV}$ there is a reaction (n,α), which leads to a series of reactions leading to the formation of He-6, which soon decays into Li-6. And the interaction of Li-6 with neutrons produces T and He:

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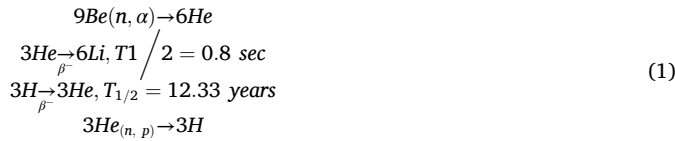
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For neutrons with energy $E \geq 2.7$ MeV, the reaction is $(n, 2n)$:



During neutron irradiation of Be_{12}Ti , helium accumulates in the crystal lattice, causing the formation of pores and cracks, which leads to deterioration of mechanical properties and is a significant problem in its long-term use in fusion plants. Tritium also affects embrittlement, but its effect is generally less significant than that of helium, but depends on the specific irradiation conditions and parameters.

Kazakhstan has a unique opportunity to produce Be_{12}Ti at the JSC "Ulba Metallurgical Plant" (JSC UMP) for the needs of fusion and hydrogen energies. Thus, the technology of Be_{12}Ti production by the industrial method of vacuum hot pressing has been developed at JSC UMP [10]. However, in accordance with international rules, after the start of serial production of the material, R&D is required to confirm the performance of the material under the conditions of its operation.

In accordance this, at the Institute of Nuclear Physics (Kazakhstan) on the basis of the WWR-K research reactor, R&D was initiated to investigate the radiation resistance of Be_{12}Ti fabricated at JSC UMP [11, 12]. This paper presents the experimental results of the study of the effect of different neutron doses on the kinetics of gas release from titanium beryllide. The features of the study lies in the application of TDS method combined with vacuum extraction to analyze the gas release kinetics from titanium beryllide samples irradiated at relatively low temperature (353 ± 15) K that allow to minimize release gases during irradiation. The study covers the analysis of gas desorption at different heating rates and for different neutron doses, which allows to study in detail the effect of irradiation parameters on desorption processes occurring in titanium beryllide samples.

2. Methods and materials

2.1. Characteristics of the investigated samples

The tested samples are plates made of beryllium intermetallide (Be_{12}Ti). Within the framework of the study, identical samples were studied, which were irradiated to different neutron fluences, and divided into two groups: a batch with the first level of accumulated fluence (F-I) and a batch with the second level of accumulated fluence (F-II). The main characteristics and irradiation conditions of the samples in the WWR-K reactor of the considered samples are given in Table 1:

Table 1
Characteristics and irradiation conditions of the investigated samples.

Parameter	Samples F-I	Samples F-II
Sample density, g/cm ³	2.18	2.13
Temperature, K	353 ± 15	
Irradiation environment	Inert gas	
Thermal neutron flux, cm ⁻² s ⁻¹	5.7×10^{13}	
Fast neutron flux, cm ⁻² s ⁻¹	2.4×10^{13}	
Thermal neutron fraction, %	53	
Fast neutron fraction, %	23	
Dose, dpa	1.9	2.9
Total neutrons fluence, n/cm ²	1.6×10^{21}	2.3×10^{21}
Accumulated thermal neutron fluence, cm ⁻²	8.2×10^{20}	1.2×10^{21}
Accumulated fast neutron fluence, cm ⁻²	3.5×10^{20}	5.2×10^{20}
Helium production, appm	134	201
Tritium production, appm	2	4

2.2. Procedure of TDS experiments

Thermodesorption studies of titanium beryllide included a series of experiments with different heating rates (5, 10, 20 K/min) to the target temperature of 1173–1223 K under deep vacuum conditions by vacuum extraction. Control measurements without sample (blank experiments) were performed before each experiment to improve the accuracy. A total of nine complete experiments were performed.

An upgraded thermodesorption spectroscopy setup [13,14] was used in this work, in which a residual gas analyser RGA-100 was used to measure the composition of the gas component and to check for tightness, which allows registering gases in the mass range from 1 to 100 a.u. m. The principle of gas registration is based on the fact that a small fraction of gas molecules is ionised, the resulting positive ions are separated by mass and measured. The detector is a Faraday cylinder. Fig. 1 shows a schematic diagram of the RTD unit.

The unit is equipped with an Agilent TPS-Flexy-304FS high-vacuum pumping station, which is based on a Turbo-V 304-FS turbomolecular pump and an Agilent Technologies IDP-7 dry scroll pump. A nitrogen trap has been installed, which increases the sensitivity of the system by minimising the methodological influence of water components on measurements of other gases.

2.3. Characterization of samples

Irradiated samples were subjected to preliminary preparation and characterization before TDS-experiments. The mass of samples before and after TDS-experiments was measured using electronic laboratory scales VIBRA HT-224RCE (measurement range: 0.01–120 g). The discreteness of mass measurement is 0.0001 g. The appearance of the samples was evaluated using Canon EOS R6 camera with RF 100 mm macro lens. The EOS R6 camera is equipped with a 20.1 MP CMOS image sensor.

Weighing of the samples before and after the TDS experiments showed that no significant changes in mass were recorded (the changes did not exceed the measurement error), which indicates that the tightness of the oxide film was preserved.

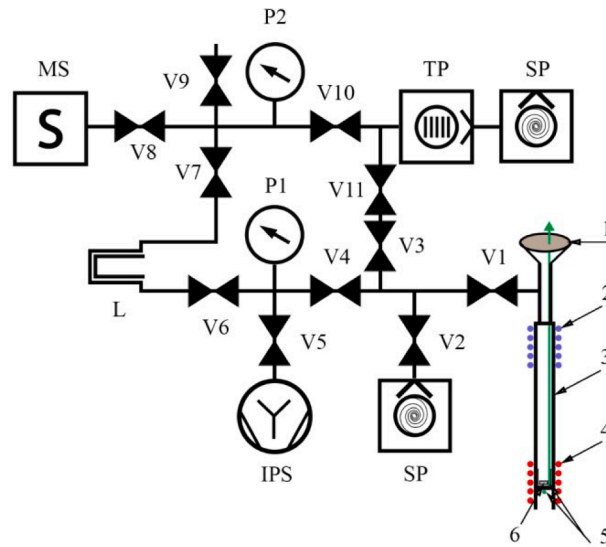
Inspection of the appearance (see Table 2) revealed a change in coloration: side A, facing the crucible, darkened, and on side B the yellow tints noticeable before the experiment disappeared. These changes may be related to thermal exposure and possible chemical reactions on the surface of the samples.

3. Results

As a result of TDS experiments, detailed data on the time dependence of mass spectra, absolute pressure measurements in the chambers of the mass spectrometer (P2) and experimental device (P1), and temperature characteristics were obtained. Analysis of the mass spectra allowed us to determine the main gas components and their partial pressures. Decoding of the M4 signal and comparison of the data obtained with previous studies showed the absence of helium emission [15]. This indicates that all the accumulated helium remains in the sample, and its desorption is possible only at higher temperatures.

Fig. 2 shows the results of the TDS experiments at a heating rate of 10 K/min, which are used to illustrate the differences between the outgassing from the sample and the effects associated with the methodology and setup features. This allows us to focus on key data, minimizing redundant visualization in subsequent experimental data, which is particularly important for accurate estimation of kinetic parameters and analysis of the mechanisms of radiation-induced gas desorption from the sample.

When comparing the data from the TDS experiments and blank curves (see Fig. 2), it can be clearly seen that a significant amount of gases, including H₂, CO and CO₂, are released from the structural materials. At the same time, the content of inert atmospheric gases such as



1 – sealed inlet; 2 – cooling, 3 – experimental cell; 4 – heating device; 5 – thermocouples; 6 – sample; P1-P2 – pressure sensors; TP – turbomolecular pump; MS – mass spectrometer; IPS – magnetic discharge pump; SP – volute pump; L – nitrogen trap.

Fig. 1. Schematic diagram of the TDS unit.

N₂ and Ar remains at background levels in all experiments and throughout the temperature range, indicating that the system is highly airtight.

The significant changes in the behaviour of O₂ and CH₄ are due to the release of moisture from the sample, which was stored under atmospheric conditions after irradiation. The level of released moisture can be estimated from the difference between the pressure values P1 and P2. Sensor P1, located upstream of the nitrogen trap in the experimental device (ED) chamber, records the absolute pressure, reflecting the presence of moisture in the system. At the same time, P2, which measures the pressure in the mass spectrometer (MS), shows lower values because it is located downstream of the nitrogen trap and corresponds to an already drained medium relative to the pumping system.

Moisture evaporates almost instantaneously from the sample as soon as the temperature exceeds 373 K. Then, according to its physico-chemical properties, it condenses on the cold inner surfaces of the ED along the pumping path. As the temperature increases, more and more area of the EA is heated, resulting in continuous evaporation of H₂O into the gas phase until the moisture flux reaches the cooling zone of the ED. Thus, the presence of moisture affected the behaviour of the previously discussed H₂, CO and CO₂ gases and also contributed to the increase in the concentration of O₂ and CH₄, which are products of water interaction with the ED structural materials.

It is important to note that the presence of moisture and molecular hydrogen (H₂) in the gas phase significantly affected the yield of tritium in HT form. As can be seen from the results, this release channel dominates the yield in the form of T₂ because the reaction rate of HT formation is orders of magnitude higher than that of the second channel.

These observations highlight the complex relationship between degassing processes, temperature changes and chemical reactions on the surface of titanium beryllides, which is important to take into account in further studies and interpretation of the results of TDS experiments.

Next, we detail the separation of tritium-containing HT and T₂ molecules to evaluate the effect of heating rate on the shift of their desorption peaks (see Fig. 3).

The obtained results confirm the expected regularity: in experiments with F-II samples, a more intensive emission of tritium-containing gases is observed compared to F-I samples. The analysis of temperature

dependences showed that for F-I samples the HT release front is fixed at about 800 K, and T₂ - at 850 K. In F-II samples these values are shifted towards lower temperatures: about 750 K for HT and 800 K for T₂. It is noteworthy that in most cases the release peak of the T₂ molecule occurs later than HT. This fact may indicate differences in the release mechanisms due to the reduction of H₂O and H₂ in the gas phase, so the probability of associations of tritium atoms on the surface increases. Having made this assumption, we can proceed to estimate the total amount of tritium, which will allow us to quantify its yield from the material. These data will also serve as a basis for further analyses of the kinetic parameters of the desorption process.

The release peaks of tritium-containing molecules for both batches of samples are observed at the same temperatures (see Fig. 4). This leads to the important conclusion that tritium release from the samples is primarily determined by diffusion through the BeO oxide film, which, according to the manufacturer's data, forms a sealed layer on the material surface.

Next, the time-dependent curves of tritium atom release were integrated, and the obtained results were normalised to the mass of the samples (see Table 3). The analysis showed that the total tritium release from F-II samples is more than twice as high as that for F-I samples. This pattern becomes even more noticeable when recalculated per unit mass of the sample, which allows us to clearly distinguish the differences in specific gas yield.

Minor deviations in the specific amount of outgassing between experiments with different heating rates can be explained by variations in the surface area of the samples (see Table 2). This parameter plays a key role in the outgassing rate, especially in TDS experiments. Samples with a larger or more porous surface have an increased contact area, which favours an enhanced release of adsorbed and embedded gases during heating. These features must be taken into account when interpreting results and comparing data to avoid errors due to differences in the geometry and surface morphology of the samples.

To estimate the activation energy (E_a) and pre-exponent (k_0), as well as to compare the obtained parameters with literature data, we used the method of kinetic analysis based on the Arrhenius equation, which is generally represented as follows:

$$k = k_0 \cdot e^{-E_a/RT} \quad (3)$$

Table 2
Inspection of the appearance of samples before and after TDS studies.

Samples	F-I		F-II	
Phase	before TDS	after TDS	before TDS	after TDS
5 K/min	No. 1		No. 2	
Side A				
Side B				
10 K/min	No. 3		No. 4	
Side A				
Side B				
20 K/min	No. 5		No. 6	
Side A				
Side B				

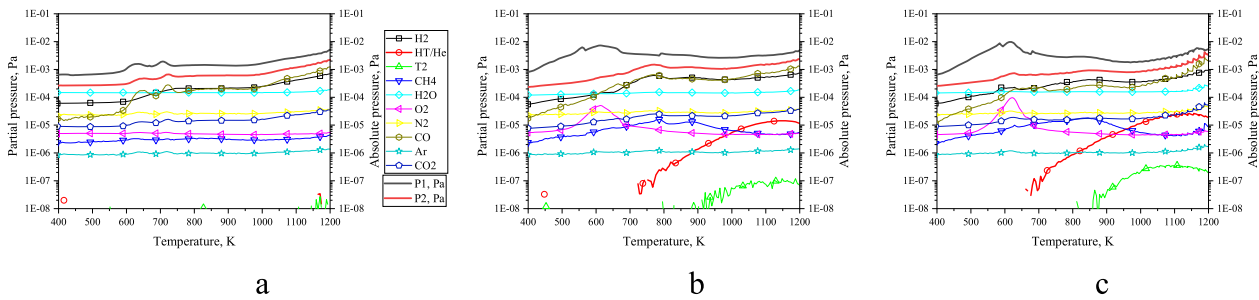


Fig. 2. Results of TDS experiments at a heating rate of 10 K/min: a - blank experiment, b - sample 2 (F-I), c - sample 5 (F-II).

where k - reaction rate coefficient, k_0 - pre-exponential multiplier, E_a - is the reaction activation energy (eV/mol), R - is the universal gas constant 8.617×10^{-5} eV/K, T - is the temperature in Kelvin.

Further, the parameters of the rate constant for the separation of tritium-containing molecules at different heating rates were determined:

$$k = \frac{dQ(t)}{dt} \cdot \frac{1}{A \cdot Q(t)} \quad (4)$$

where $Q(t)$ is the concentration of tritium-containing molecules, A is the area of the sample under study. To determine the pre-exponential factor, we prologue Eq. (3):

$$\lg(k) = \lg(k_0) + \frac{E_a \cdot 0.4343}{RT} \quad (5)$$

Now let us plot the dependence of $\lg(k)$ on $1000/T_{peak}$ - desorption peak temperature, which further allows us to describe the kinetics of

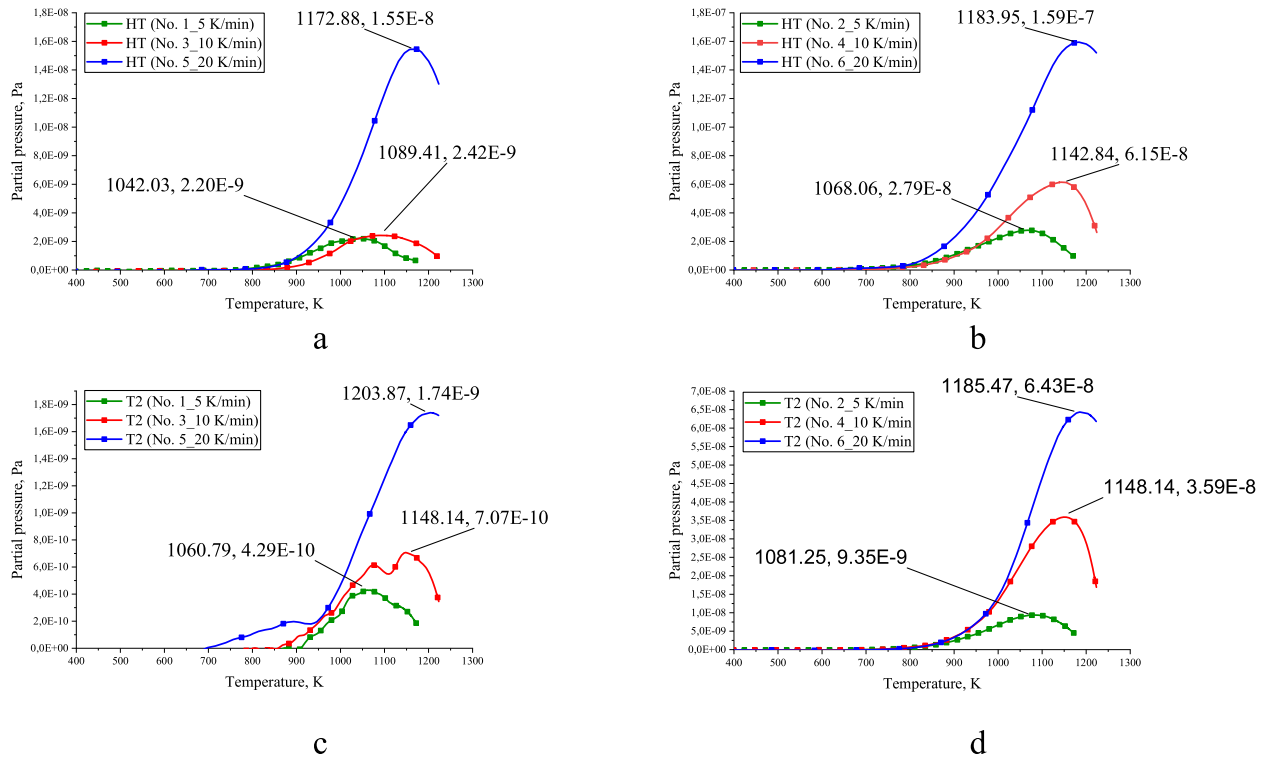


Fig. 3. Effect of heating rate on the temperature peaks of HT and T₂ release for samples: a & c - F-I, b & d - F-II.

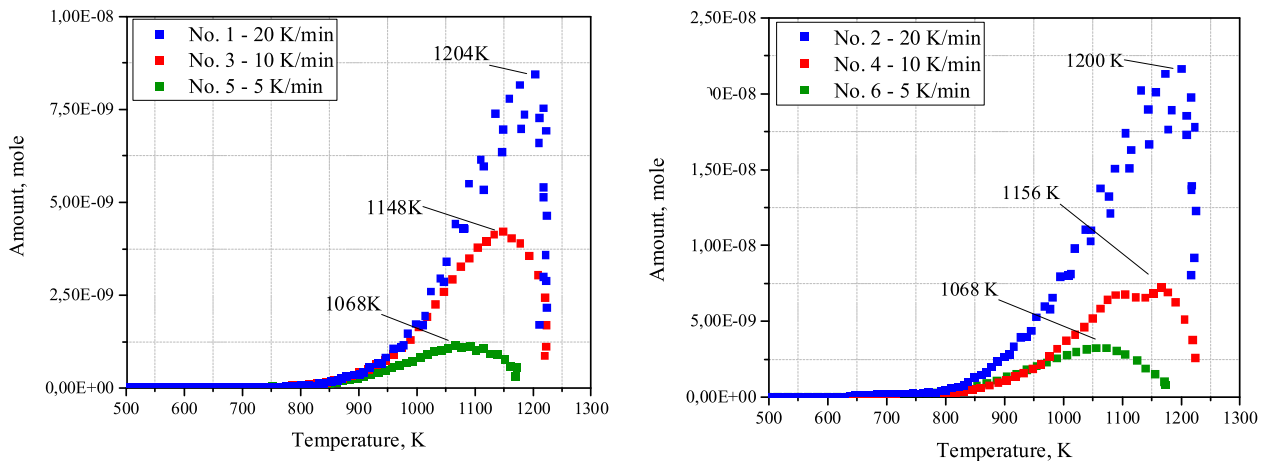


Fig. 4. Determination of emission peaks of tritium-containing molecules for F-I and F-II titanium beryllides.

Table 3

Amount of tritium released and specific characterisation.

Heating rate, K/min	F-I		F-II	
	Integral Q(t), mole	Q(t)/m, mole/g	Integral Q(t), mole	Q(t)/m(g), mole/g
5	2.93 E-06	5.57 E-05	9.53 E-06	2.05 E-04
10	5.47 E-06	8.64 E-05	1.03 E-05	1.44 E-04
20	5.41 E-06	8.68 E-05	1.60 E-05	1.89 E-04

tritium release in the process of desorption. Fig. 5 shows that the dependence of $\lg(k)$ on $1000/T_{peak}$ has good linearity for different heating rates of 5, 10 and 20 K/min. The slope of this dependence allows us to calculate - E_a activation energy, and the crossing point - k_0 pre-exponential factor (Arrhenius coefficient).

Analysis of the data from Table 4 confirms the relevance of the previously proposed mechanisms of outgassing. The values of E_a for samples F-I and F-II, taking into account the measurement error, are close and practically overlap, indicating similar mechanisms of tritium desorption in samples subjected to different levels of neutron irradiation.

At the same time, k_0 is significantly different, indicating a change in the frequency of successful desorption acts. This may be due to micro-structural changes in the material after irradiation, such as an increase in the concentration of radiation defects and changes in the oxide film density.

The close values of E_a confirm that the main mechanism of tritium release remains unchanged and the outgassing process is controlled by diffusion through the BeO oxide layer. The structural changes of the material caused by the fluence increase do not critically affect the

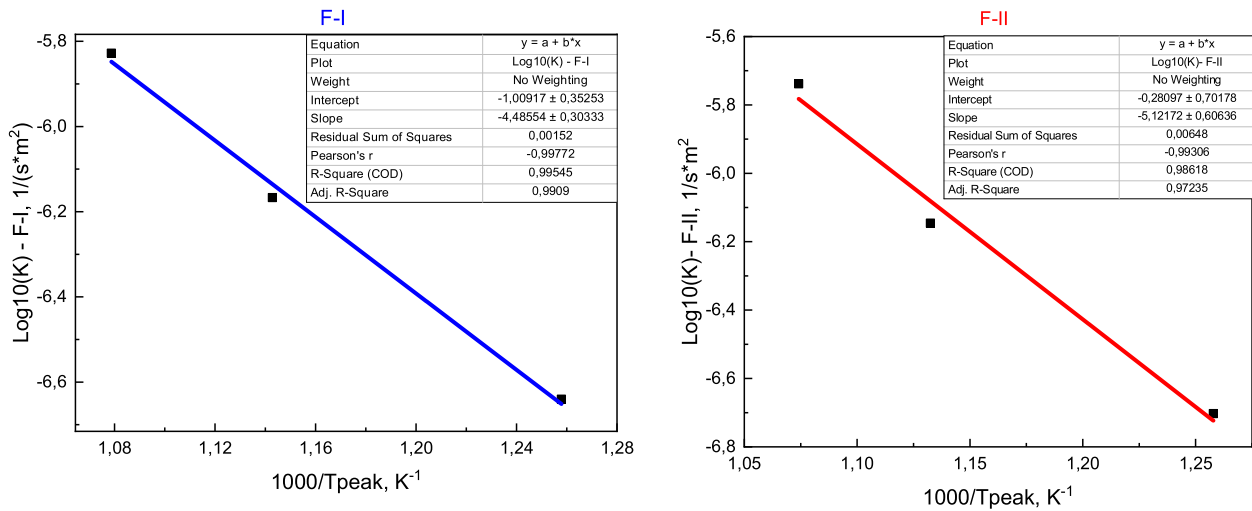


Fig. 5. Dependence of $\lg(k)$ on $1000/T_{\text{peak}}$ for F-I and F-II titanium beryllides.

Table 4

Kinetic parameters of tritium desorption from Be_{12}Ti .

Parameter	F-I	F-II
$k_0, (\text{s} \cdot \text{m}^2)^{-1}$	0.10 ± 0.02	0.52 ± 0.10
E_a, eV	0.89 ± 0.08	1.02 ± 0.09

desorption energy barrier.

However, the significant increase in k_0 from F-I to F-II indicates an increase in the tritium release rate. This may be due to an increase in the concentration of defects in the material, which leads to the formation of additional active centers for tritium release and facilitates its migration through the sample structure.

4. Discussion

In this study, titanium beryllide material Be_{12}Ti , fabricated by industrial vacuum hot-pressing method, is investigated after irradiation to various neutron doses. In titanium beryllides, tritium release occurs more easily than in pure Be due to the weaker bonding of tritium to the BeO oxide film and the influence of titanium, which favours accelerated diffusion. In contrast, pure Be has higher tritium retention due to its strong oxide film which limits the gas release. The TDS results showed that the kinetics of tritium release is dependent on the heating rate, and its yield is controlled by diffusion through the BeO oxide film. It is observed that the emission maxima of tritium-containing molecules shift towards higher temperatures with increasing heating rate. This fact is supported by the results of a study conducted as part of thermal desorption tests for Be, Be_5Ti and Be_7Ti materials [16].

In a study [17], the tritium desorption energy for irradiated beryllium (Be) and titanium beryllide (Be_7Ti) samples was determined to be 3–4.1 eV. These results show some discrepancy with our data. Possible factors explaining this difference are the difference in the levels of damaged doses and temperature conditions. In this work, the samples were irradiated to damaged doses in the range of 23–38 dpa and at higher temperatures (710–1040 K), while in our studies the samples were irradiated to doses of 1.9–2.9 dpa at a low temperature of 353 K. This significant gap in irradiation doses and temperature regimes may affect the tritium desorption characteristics, since higher irradiation levels and temperatures may lead to different desorption mechanisms related to defects occurring in the material.

5. Conclusion

Gas emission from titanium beryllide samples irradiated to different fluences of neutrons at 353 K and by the TDS method to temperatures of 1173–1223 K has been studied.

The use of thermodesorption spectroscopy in combination with vacuum extraction allowed a detailed study of the outgassing processes from irradiated Be_{12}Ti samples, which represents a unique approach and contributes to a deeper understanding of the influence of irradiation on outgassing kinetics.

The investigations showed that the kinetics of tritium release depends on the heating rate and diffusion through BeO , and the presence of H_2O and H_2 in the gas phase favours the predominance of HT release over T_2 .

In samples with a fluence of $1.6 \times 10^{25} \text{ n/m}^2$, HT emission starts at 730 K and T_2 at 890 K, while in samples with a fluence of $2.3 \times 10^{25} \text{ n/m}^2$, the emission front shifts to lower temperatures, 610 K for HT and 780 K for T_2 , indicating radiation-induced changes in the outgassing mechanisms.

The kinetic parameters of tritium desorption from Be_{12}Ti were determined in this study, where the activation energy for F-I was 0.89 eV and for F-II was 1.02 eV. The differences in k_0 indicate an enhanced tritium yield at high fluence, while E_a remains unchanged, indicating the dominance of diffusion through BeO and the influence of fluence on the process kinetics.

Overall, the data obtained will aid in further research and optimisation of titanium beryllide as a material for fusion reactors, and provide key insights for the development of more efficient gas management techniques under such conditions.

Finding

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CRedit authorship contribution statement

A. Shaimerdenov: Writing – review & editing, Project administration, Funding acquisition. **A. Akhanov:** Writing – original draft, Software, Methodology, Data curation, Writing – review & editing. **S. Askerbekov:** Validation, Methodology, Investigation, Writing – review & editing. **T. Kulsartov:** Validation, Supervision, Conceptualization. **M. Aitkulov:** Investigation, Data curation. **D. Sairanbayev:** Formal

analysis. **A. Nessipbay:** Investigation, Data curation. **Zh. Bugybay:** Investigation. **I. Kenzhina:** Investigation. **B. Shakirov:** Investigation. **M. Kylyshkanov:** Resources. **M. Podoinikov:** Resources.

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

The data used is not confidential.

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