

Comparative analysis of high-temperature corrosion processes of beryllides of different compositions

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ABSTRACT

Beryllium intermetallic compounds, such as titanium beryllide (Be_{12}Ti), chromium beryllide (Be_{12}Cr), and zirconium beryllide (Be_2Zr), exhibit exceptional physicochemical properties, making them promising materials for diverse scientific and energy applications. Among them, Be_{12}Ti is the leading candidate for neutron multiplier use in future European projects employing the Helium Cooled Pebble Bed (HCPB) concept and solid blanket systems of ITER and DEMO-type reactors, due to its high melting point, radiation-induced swelling, low activation, and excellent corrosion resistance. To broaden the scope of material selection, exploring alternative compounds has gained importance. Having properties similar to Be_{12}Ti , chromium beryllide Be_{12}Cr demonstrates potential as a possible option, including high thermal and radiation resistance, although its corrosion resistance in vapor-gas medium requires further research. Beyond fusion applications, beryllides have potential in other domains. For example, Be_2Zr exhibits remarkable properties for hydrogen energy, such as forming stable hydride phases, making it an excellent candidate for hydrogen storage systems. These investigations are especially relevant for advancing hydrogen and fusion energy technologies in Kazakhstan. Ulba Metallurgical Plant JSC, a leader in beryllium material production, synthesizes not only Be_{12}Ti , Be_{12}Cr and Be_2Zr , but also less studied beryllides.

This study performs a comparative analysis of high-temperature corrosion in beryllides with varying compositions. A series of experiments were conducted to investigate the corrosion mechanisms under vapor-gas mediums with different isotopic compositions using non-isothermal heating across a wide temperature range. Key features of beryllide corrosion were revealed, including time-dependent changes in sample mass and gas-phase composition during linear heating. Corrosion behaviors of different beryllide compositions were established, and temperature-dependent reaction rates determined. These findings enhance understanding of beryllide corrosion properties, providing a scientific basis for their potential in fusion and hydrogen technologies.

1. Introduction

Selecting materials for fusion reactors (FR) is a key task that directly impacts the advancement of fusion energy. This selection is guided by harsh criteria: materials must endure prolonged exposure to intense radiation and thermal loads, ensure efficient tritium generation, be cost-

effective to produce, ensure safe operation, utilization and disposal. Satisfying these requirements is essential for achieving the sustainability and efficiency needed for the future of fusion energy systems.

Beryllium intermetallic compounds, particularly titanium beryllide (Be_{12}Ti), are considered as promising functional materials for solid blanket systems in ITER and DEMO-type reactors [1–3]. Titanium

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beryllide (Be_{12}Ti) possesses a unique combination of properties, including a high tritium breeding ratio, low activation, a high melting point (1550 °C), excellent corrosion resistance, thermal stability, and resistance to radiation-induced swelling. These attributes position it as a leading candidate for functional material applications in the blankets of future FR [4,5].

An alternative to Be_{12}Ti is chromium beryllide (Be_{12}Cr) [6], as both materials share similar physical and chemical properties [7], making them promising candidates for the utilization in the solid blanket systems of future FR [8]. While the melting point of Be_{12}Cr (1340 °C) is lower than that of Be_{12}Ti , the phase diagram of Be_{12}Cr , as presented in [9], is simpler and does not involve a peritectic reaction. However, chromium beryllide (Be_{12}Cr) remains a relatively less-studied material, especially in the context of its corrosion resistance and behavior in vapor-gas medium.

Another interesting beryllium intermetallic compound is zirconium beryllide Be_2Zr . In [10], the interaction of several beryllium-rich intermetallic compounds with hydrogen was investigated. The authors found that such compounds as Be_2Ti , Be_2Zr , and Be_2Hf form stable hydride phases, which opens prospects for their use in hydrogen energy for hydrogen storage.

Specialists of “Ulba Metallurgical Plant” JSC, a leading enterprise in the production of beryllium-containing materials, are involved in the synthesis and production of not only Be_{12}Ti , Be_{12}Cr , and Be_2Zr , but also other less-studied beryllides, such as Be_2Ti , Be_2Cr , Be_3Nb , Be_2Nb_3 , and Be_{12}Nb . This highlights the significant potential of these materials for applications in both fusion [6,11,12] and hydrogen energy systems [13]. An essential stage in the research of beryllium-containing materials involves conducting experiments to study their behavior under conditions simulating operation in FR, including prolonged irradiation in neutron fields of nuclear reactors. These experiments are crucial for assessing the durability and functional characteristics of materials under extreme conditions, which is fundamental for their application in nuclear power engineering [14–16]. Notably, some of these studies have also been carried out in the Republic of Kazakhstan [17–21], where significant expertise and experience in this field have been developed.

One of the major challenges limiting the use of beryllide-based functional materials in breeder blankets of future FR is their susceptibility to high-temperature corrosion [22]. A particularly critical concern is the interaction of these materials with water vapor of varying isotopic compositions present in the operating medium. The operational conditions of solid-state breeders involve high-temperature heating up to 1000 °C, which can significantly impact the structural integrity and performance characteristics of these materials. The limited availability of data on their behavior under such extreme conditions poses a significant barrier to their assessment and implementation in fusion and hydrogen technologies.

Previous studies on beryllium and its alloys have demonstrated that corrosion under such conditions can result in substantial changes in the mass, structure, and chemical composition of the materials [23–25]. For instance, [26–29] has highlighted that high-temperature corrosion significantly affects the properties of beryllium samples, posing a limitation to their use in FR and hydrogen energy [30,31].

As previously noted, beryllium intermetallic compounds such as Be_{12}Ti , Be_{12}Cr and Be_2Zr are highly promising for applications in blanket systems and hydrogen energy due to their exceptional properties. However, there is a significant lack of data on the behavior of these beryllides when exposed to water vapor with varying isotopic compositions. This gap hinders a comprehensive assessment of their performance characteristics and potential implementation in fusion and hydrogen technologies. Understanding the nature and mechanisms of corrosion processes in these materials is critical to evaluating their applicability in these fields. Conducting comprehensive studies that simulate real operating conditions will not only understand the mechanisms by which beryllides interact with chemically active gases and water vapor, but also provide the foundation for recommendations to

enhance their resource durability and operation.

Earlier, in [32], we investigated the high-temperature corrosion of titanium beryllide (Be_{12}Ti) in both plate and crushed sample forms. During these experiments, new thermal data were obtained, describing the corrosion processes of Be_{12}Ti samples across a wide temperature range and at various heating rates in the presence of water vapor within the purge gas. The present study serves as a logical continuation of this research, focusing on the high-temperature corrosion behavior of chromium beryllide (Be_{12}Cr) and zirconium beryllide (Be_2Zr).

The purpose of this study is to perform a comparative analysis of the high-temperature corrosion processes in beryllides of various compositions. The paper presents experimental results on the corrosion behavior of Be_{12}Cr and Be_2Zr beryllides under the influence of vapor-gas media with different isotopic compositions. Time-dependent mass changes in the samples were measured, the composition of the gas phase during heating at 10 °C/min rates up to 1400 °C was analyzed, and the temperature dependences of corrosion reaction rates were determined. These findings contribute to the understanding of the degradation mechanisms of beryllides and offer valuable insights for optimizing their application under the operational conditions of FR and hydrogen energy systems. Consequently, the results of this study not only expand the current knowledge of corrosion processes in beryllides of various compositions, but also provide a foundation for developing recommendations on the selection and preparation of promising functional materials for future fusion and hydrogen technologies.

2. Experimental part

2.1. Materials and methods

Crushed samples of Be_{12}Cr and Be_2Zr beryllides were used as investigated materials. The crushed beryllide samples represented a heterogeneous fraction with particle size distribution from 0.5 to 4 mm and different particle shapes. The appearance of Be_{12}Cr and Be_2Zr beryllide samples before corrosion experiments is shown in Fig. 1a and 1b, respectively. Crushed samples of Be_{12}Cr and Be_2Zr beryllides were used to increase the contact surface area between the material and the vapor-gas medium. This approach facilitates a more detailed examination of corrosion processes at microlevel, enabling a deeper understanding of material's behavior under such conditions.

A series of high-temperature corrosion experiments with crushed samples of Be_{12}Cr and Be_2Zr beryllides were conducted using the TiGrA experimental complex [33], which integrates thermogravimetric analysis (TG), differential scanning calorimetry (DSC), and mass spectrometric (MS) measurements in a single experiment.

Comprehensive materials science analyses were performed to assess the impact of high-temperature corrosion on the structure and phase composition of the beryllides. Microstructural changes were examined using a Leica M125C stereomicroscope, while phase composition analysis was carried out with an Empyrean diffractometer from Malvern Panalytical Ltd., equipped with a PIXcel1D linear detector. The phase composition of the surface was identified using the PDF-2 ICDD Release 2004 database.

2.2. Technique of high-temperature corrosion experiments with beryllide samples

In order to expand the capabilities and the range of conducted measurements, the TiGrA facility was modernized, in the course of which a module for creating a gas purge mixture with specified parameters was designed and manufactured. This module includes a spiral fore pump with a vacuum path and a valve for separating the gas system collector from the vacuum part, a collector with shutoff valves, a humidifier, a pressure control manovacuum meter, two small tanks for chemically active gases, a humidity generator MHG and a buffer tank for creating mixtures with a specified composition. The created module was

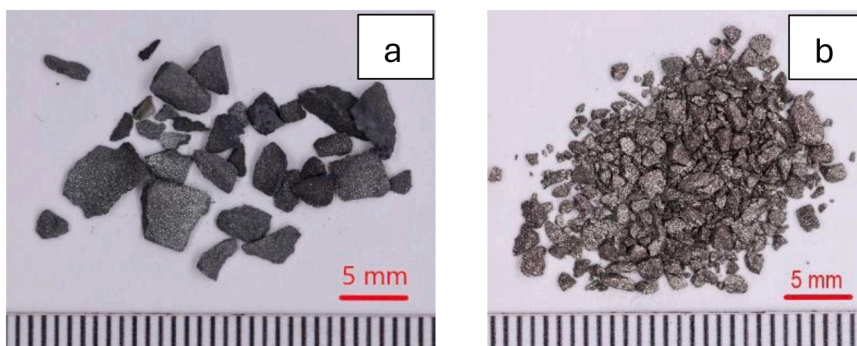


Fig. 1. Appearance of crushed Be_{12}Cr (a) and Be_2Zr (b) samples before corrosion experiments.

integrated into the standard system of gas supply to the reaction chamber of TiGrA facility and allows to create mixtures of chemically active gases: hydrogen (up to 0.13 kPa in the purge mixture based on inert gas); oxygen (up to 1.3 kPa), water vapor (up to 4 kPa). Fig. 2 shows a general view of the TiGrA facility after modernization.

High-temperature corrosion experiments of Be_{12}Cr and Be_2Zr beryllide samples were performed at the modernized TiGrA facility, according to the general scheme of experiments presented in Fig. 3.

The high-temperature corrosion testing procedure involved sequential stages using the same crushed beryllide samples, with regular mass measurements conducted throughout the experiments for control purposes. Before initiating the tests, the TGA-DSC 3+ module was heated, and the measuring cell was stabilized. The beryllide samples were then placed in a crucible, loaded on the platinum substrate of the TGA-SDTA ceramic sensor. To achieve the objectives of the study, a temperature program was set prior to the experiment, which included linear heating of the samples from 100 °C to 1200 °C (1400 °C for Be_{12}Cr samples) at a rate of 10 °C/min followed by cooling at a rate of 40 °C/min. The inert gas argon was used as the reaction gas, with additions of heavy water vapor (D_2O) and water vapor mixture ($\text{D}_2\text{O}+\text{H}_2\text{O}$), which was fed into

the chamber at a specified flow rate. The experimental mass change measurements, heat flux measurements and real-time mass spectroscopic data, obtained during the experiments were stored and further processed using the STARE software integrated in TGA-DSC 3+.

The following section presents the results of high-temperature corrosion experiments with Be_{12}Cr and Be_2Zr beryllide samples.

3. Results

3.1. Results of corrosion experiments with Be_{12}Cr samples

A sequential series of three high-temperature corrosion experiments was performed on crushed chromium beryllide Be_{12}Cr samples, with each experiment varying in the composition of the vapor-gas mixture. Table 1 provides a summary of the experimental conditions for this series. All experiments were conducted within a temperature range of 100 °C to 1400 °C, maintaining a total water vapor pressure in the chamber at approximately 2 kPa. These consistent and stable conditions ensured a reliable evaluation of the kinetic characteristics of the interaction between chromium beryllide Be_{12}Cr and the vapor-gas mixtures.

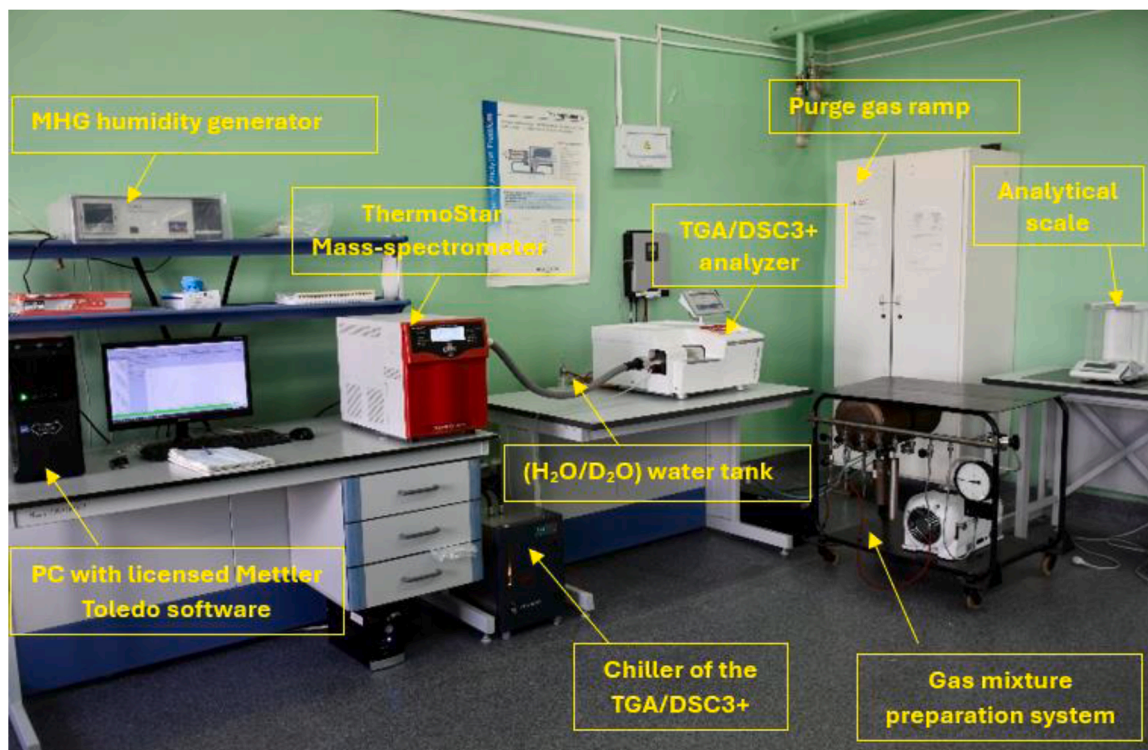


Fig. 2. General view of the modernized TiGrA facility with a module for preparation and supply of gas and vapor-gas mixtures.

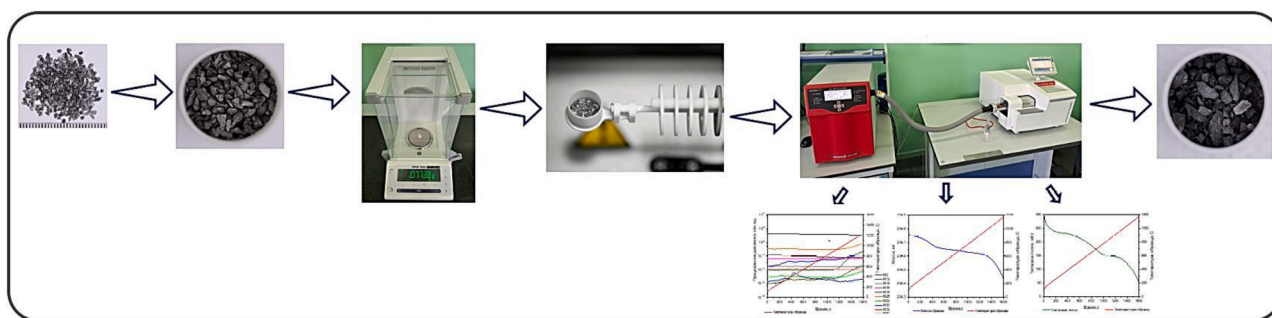


Fig. 3. Scheme of corrosion experiments on Be₂Zr sample example.

Table 1

Experiment condition with Be₁₂Cr sample.

Experiment	Temperature range	Compound of vapor-gas mixture	Mass, mg
N ^o 1	from 100 °C to 1400 °C	Ar(10 ⁵ Pa) + D ₂ O(1.2·10 ³ Pa) + HDO(800 Pa)	244.25
N ^o 2		Ar(10 ⁵ Pa)+D ₂ O(1.3·10 ³ Pa) + HDO(10 ³ Pa)	264.54
N ^o 3		Ar(10 ⁵ Pa)+D ₂ O(700 Pa)+HDO (1.2·10 ³ Pa) +H ₂ O(10 ³ Pa)	279.33

Fig. 4 illustrates the results of high-temperature corrosion experiments with Be₁₂Cr samples, including diagrams of corrosion kinetics (a, b) and the appearance of the samples after the corrosion experiment (c). Throughout the experiments, data on gas release, mass changes, and heat flux were recorded.

First Experiment: The vapor-gas mixture consisted of argon (Ar), heavy water (D₂O), and partially HDO (800 Pa). Early in the experiment, notable gas release, including D₂ and HD, indicated the onset of active interaction between water vapor and the sample surface. Corrosion initiated earlier in this experiment than in the subsequent ones. Post-experiment, the samples displayed a color change, but their shape and structural integrity were preserved, indicating minimal structural degradation.

Second Experiment: The second experiment confirmed the primary observations from the first. The sample's initial mass of 264.54 mg increased to 279.33 mg, resulting in a mass gain of ~ 15 mg. The gas release pattern, including D₂ and HD, contrasted with the initial experiment, suggesting consistent corrosion mechanisms in this vapor-gas medium.

Third Experiment: The third experiment introduced a more complex vapor-gas mixture, resulting in additional molecular hydrogen H_2 release alongside D_2 and HD. In this experiment, which involved a mixture of water and heavy water vapor, the $Be_{12}Cr$ sample exhibited a mass gain of 14.40 mg. This indicates more active chemical processes occurring with the presence of H_2O . Such results indicate a significant influence of mixture composition on kinetics of processes.

Fig. 5 shows typical DSC curves for Be₁₂Cr sample obtained during heating and cooling.

Analysis of the temperature dependence of the change in the heat flux of the sample during purging with a vapor-gas mixture revealed the following features:

During heating, starting from the temperature of 1306.16 °C an endothermic heat effect was observed, probably related to the phase transition. The heat of reaction was $-50.46 \cdot 10^3$ J/g.

During cooling, an exothermic heat effect was observed at 1318.22 °C with a reaction heat of $42.73 \cdot 10^3$ J/g, indicating that energy was released during the reverse phase transformation or other related processes.

The DSC curves obtained for each experiment with the Be₁₂Cr sample show well-defined peaks of heat flux deviation from the trend. These

peaks indicate the presence of phase transformations in chromium beryllide samples at various stages of the experiment, which allows us to identify the temperature boundaries and the nature of the changes that occur.

One of the most significant thermal effects observed is the appearance of peaks in the temperature range from 1300 °C to 1350 °C. This phenomenon may have several explanations. According to current data [34,35], the melting point of chromium beryllide (Be_{12}Cr) is approximately 1338 °C, as confirmed by the phase diagram of the Be-Cr system [9], where Be_{12}Cr is shown to congruently melt at around 1338 °C. However, the results of corrosion experiments demonstrated that Be_{12}Cr samples retained their shape and did not melt even after three series of heating to 1400 °C.

Another possible explanation for these thermal effects is the rearrangement of the electronic structure of beryllium at temperatures above 1250 °C. It is known that the electronic configuration of beryllium in its ground state ($1s^2 2s^2$) can change through the transition of one $2s$ electron to the $2p$ level, resulting in a $1s^2 2s^1 2p^1$ configuration. In pure beryllium, this rearrangement leads to a transformation of the crystal lattice from hexagonal close-packed (hcp) to cubic. It is plausible that in the binary compound Be_{12}Cr , a similar rearrangement of beryllium's electronic structure above 1250 °C could cause a modification of the original tetragonal crystal lattice to a different structure.

To clarify the mechanisms of high-temperature processes in Be₁₂Cr samples after corrosion interactions with water vapor, post-materials studies were carried out.

3.2. Results of post-materials studies with Be_{12}Cr samples

Fig. 6 shows the results of X-ray phase analysis of the Be₁₂Cr sample before and after corrosion experiments.

X-ray structural studies were conducted on both the surfaces of individual fragments or sets of fragments and on powder samples. The powder samples were prepared by further grinding representative quantities of fragments in an agate mortar with excess alcohol. A layer of finely dispersed powder was then applied to the surface of a glass substrate for analysis.

Analysis of the viewing XRD revealed that diffraction data obtained from the surfaces of individual fragments or sets of fragments were practically identical to those obtained from powder samples prepared by additional grinding in an agate mortar. However, analyzing the surfaces of some fragments posed challenges due to the need for sufficient surface area. This often resulted in diffraction patterns of insufficient quality or required significantly extended experiment durations.

The diffraction data characterizes both the initial phase composition of the samples and the changes occurring as a result of the corrosion experiments. The initial phase composition was predominantly represented by the corresponding beryllide Be_{12}Cr agrees well with the data given in [6]. After corrosion testing, changes in the sample composition were noted, including a reduction in the intensity of the diffraction peaks corresponding to the primary Be_{12}Cr phase (Reference Code

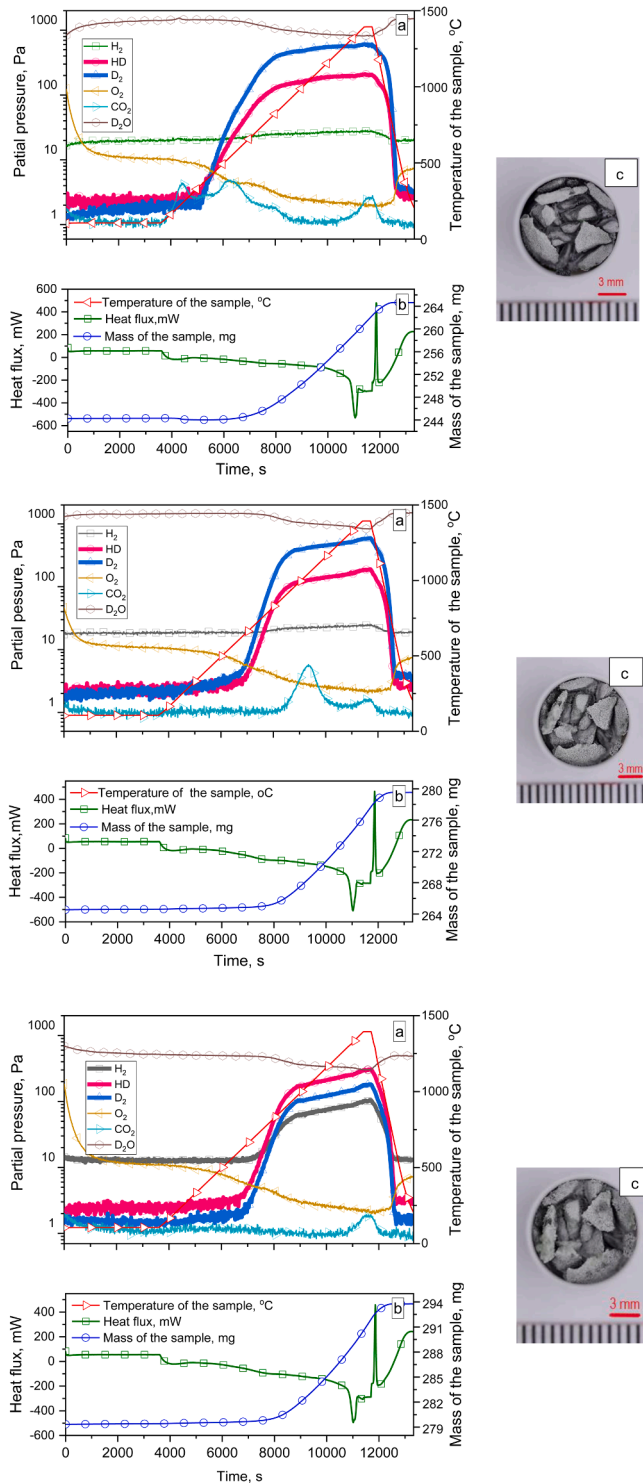


Fig. 4. Results of a series of high-temperature corrosion experiments (left) and appearance of crushed Be_{12}Cr samples after corrosion experiment (right).

03–065–2769). Additionally, new diffraction peaks associated with corrosion products, specifically oxide crystalline phases, were observed. These included low-temperature modifications of BeO (bromellite, Reference Code 01–078–1560) with notable peaks at 38.5° , 41.2° , and 43.9° .

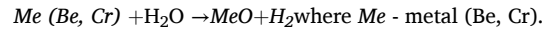
Fig. 7 shows the results of microstructural studies of the surface of crushed Be_{12}Cr samples before and after corrosion experiments.

As shown in the Fig. 7, the surfaces of the samples underwent noticeable changes due to exposure to the vapor-gas medium. Initially,

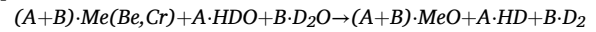
the sample surfaces were uniform, displaying the characteristic structure typical of chromium beryllide. However, after the corrosive exposure, a white coating formed on the surface, indicating the formation of corrosion products.

The color change and the appearance of a corrosion coating indicate active interaction of the Be_{12}Cr surface with the components of the vapor-gas mixture at high temperatures. This suggests the formation of oxide or hydroxide phases, confirming the intensity of the chemical processes taking place.

The reaction of the interaction of the samples with water vapor can be described by the following equation:



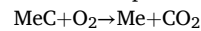
A similar reaction occurs with heavy water vapor, which can be represented as:



where, A and B denote stoichiometric coefficients depending on the ratio of the vapor-gas mixture.

These reactions are key in the corrosion process, since they make a significant contribution to the mass increase observed during the experiments. The mass increase is due to the formation of oxide phases on the surface of the samples (BeO), as well as the release of gases H_2 , D_2 , HD , which were recorded during the experiments.

In addition, the formation of carbon dioxide CO_2 on the surface of samples can occur as a result of the reaction of carbon-containing or hydrocarbon impurities. This process can be described by the equation:



The presence of even a small amount of carbon impurities on the surface of the samples can influence the reaction, which must be considered when interpreting the results of the corrosion experiments. The detection of CO_2 formation indicates that oxidation processes occur alongside the primary reactions between the metals and water vapor.

Following the completion of the corrosion experiments on chromium beryllide (Be_{12}Cr), the next stage of the study focused on examining the behavior of zirconium beryllide (Be_2Zr) under similar conditions.

3.3. Results of corrosion experiments with Be_2Zr samples

A series of three experiments was conducted using a single crushed Be_2Zr sample, during which the composition of the vapor-gas mixture was varied. The experiments followed the same methodology used for Be_{12}Cr , including non-isothermal exposure of the sample to the vapor-gas medium during linear heating. However, the upper temperature limit was reduced to 1200°C due to the differing characteristics of Be_2Zr .

The total mass of the sample prior to the first corrosion experiment was 371.87 mg. Table 2 provides the specific conditions for the experiments conducted with the Be_2Zr sample.

Fig. 8 presents the results of corrosion experiments with Be_2Zr samples, including diagrams of corrosion kinetics and gas release (a, b), as well as the appearance of the samples after the corrosion tests (c). This visual comparison enables assessment of surface changes resulting from exposure to the vapor-gas medium.

The diagram for the first experiment illustrates the interaction of zirconium beryllide (Be_2Zr) with water vapor. The corrosion behavior observed was generally similar to that of Be_{12}Cr in previous experiments. During heating, the release of gaseous corrosion products such as D_2 and HD was recorded, accompanied by a mass increase in the samples, suggesting the formation of new surface phases, likely BeO . Additionally, the kinetics of CO_2 release differed from the Be_{12}Cr experiments, potentially due to variations in the chemical composition of surface impurities or the specific reactions with carbon-containing compounds on the surface of Be_2Zr .

In the second experiment, the mass of the Be_2Zr sample increased from 388.36 mg to 406.46 mg, consistent with previously observed trends.

In the third experiment, which utilized a mixture of water vapor, the

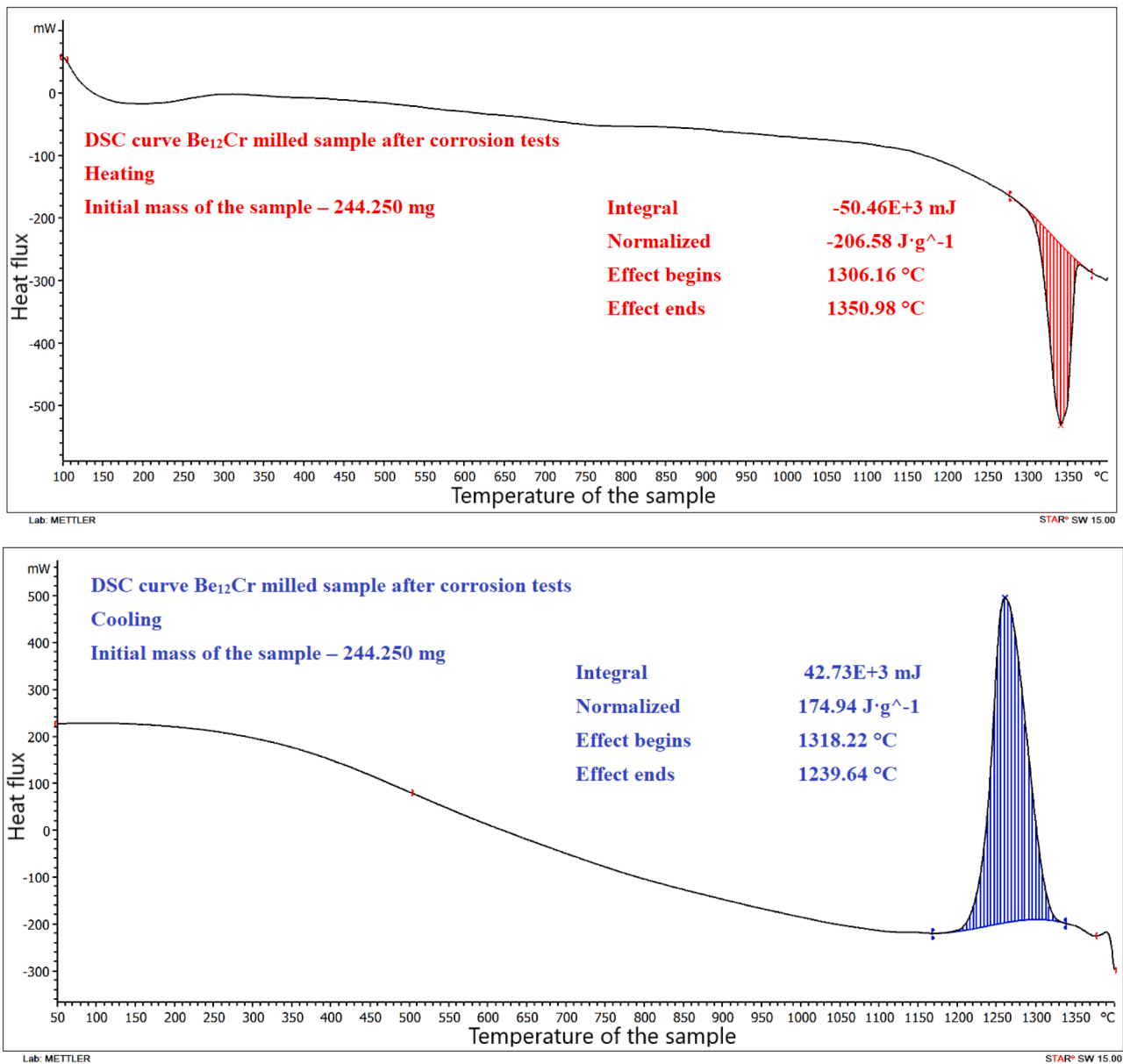


Fig. 5. Temperature dependence of heat flux variation of crushed Be₁₂Cr samples under vapor-gas mixture purging during heating (red) and cooling (blue).

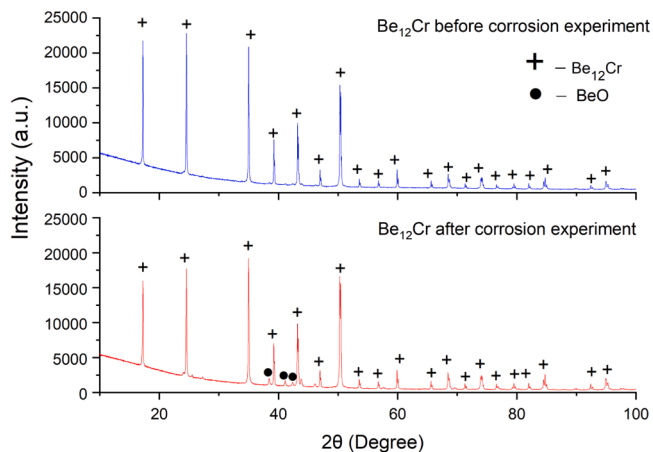


Fig. 6. XRD results of the Be₁₂Cr sample before and after corrosion experiments.

mass increased by 20 mg. Notably, the release of D₂ and H₂ molecules exhibited distinct patterns, indicating differences in the mechanisms of hydrogen isotope interactions with the Be₂Zr sample.

Across all experiments, no thermal effects were recorded, suggesting the absence of significant endothermic or exothermic reactions under the given conditions.

After completing the corrosion experiments on Be₂Zr samples, post-experimental materials science examination was conducted to examine changes in the structure and phase composition of the sample surface.

3.4. Results of post-materials studies with Be₂Zr samples

Fig. 9 shows the results of X-ray phase analysis of the Be₂Zr sample before and after corrosion experiments.

The initial phase composition of the samples before the corrosion impact is represented mainly by the target beryllide Be₂Zr (Ref. Code 03-065-7835), which is confirmed by the high intensity of its diffraction peaks. Impurity phases in the initial state appear weakly, including a small peak of beryllium oxide BeO at 2θ = 57.6°, related to bromellite

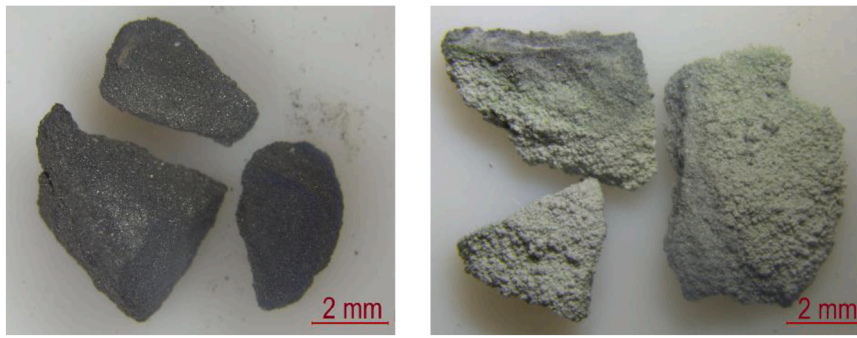


Fig. 7. Microstructural studies of the surface of crushed Be₁₂Cr samples before (left) and after corrosion experiments (right).

Table 2

Experiment condition with Be₂Zr sample.

Experiment	Temperature range	Compound of vapor-gas mixture	Mass, mg
N ^o 1	from 100°C to 1200°C	Ar(10 ⁵ Pa) + D ₂ O(1.2·10 ³ Pa) + HDO(800 Pa)	371.87
N ^o 2		Ar(10 ⁵ Pa)+D ₂ O(1.3·10 ³ Pa) + HDO (10 ³ Pa)	388.36
N ^o 3		Ar(10 ⁵ Pa)+D ₂ O(700 Pa)+HDO (1.2·10 ³ Pa) +H ₂ O(10 ³ Pa)	406.67

(Ref. Code 01–072–0290).

After corrosion exposure, significant changes in the phase composition occurred. In particular, a clear decrease in the intensity of the Be₂Zr peaks is noticeable from Fig. 9. The decrease in the intensity of the Be₂Zr peaks is obviously associated with the formation of surface layers of corrosion products (oxides BeO and ZrO₂). New peaks corresponding to oxide phases appeared on the diffraction patterns after the corrosion experiment:

- BeO (Reference Code 01–072–0290): low-temperature modification of beryllium oxide (bromelite) appears as distinct peaks, including a characteristic peak at $2\theta=38.5^\circ, 43.9^\circ, 57.6^\circ$
- ZrO₂ (Reference Code 01–078–0047): monoclinic modification of zirconium oxide (baddeleyite) appears as new diffraction peaks at $2\theta = 28.1^\circ, 31.4^\circ, 34.1^\circ, 38.8^\circ$, indicating the interaction of zirconium with oxygen.

Analysis of the phase composition showed that changes in the phase composition after corrosion effects are explained by the formation of surface oxide layers, which are formed as a result of chemical reactions of the components of the vapor-gas medium with the material. The formation of oxides probably occurred predominantly in the active areas of the surface, which correlates with the increase in the mass of the samples recorded during the experiments.

Fig. 10 shows the results of microstructural studies of the surface of crushed Be₂Zr samples before and after corrosion experiments.

The presented microstructural images reveal several notable features. Before the corrosion experiments, the surface of the Be₂Zr samples appeared relatively smooth and uniform, with minimal signs of damage. There were no visible traces of oxide deposits or evidence of interaction with an aggressive medium. However, after exposure to the vapor-gas medium, the surface structure of the Be₂Zr samples exhibited changes, including minor roughness and localized traces of interaction with the medium. Notably, unlike beryllides with a tetragonal crystal structure (Be₁₂Ti, Be₁₂Cr), the Be₂Zr samples did not develop a pronounced oxide layer.

These differences can be attributed to the characteristics of the crystal structure of zirconium beryllide [36]. The hexagonal crystal lattice (P6/mmm) of Be₂Zr is marked by a denser atomic packing, which

likely inhibits the diffusion of oxygen and other reactive components of the vapor-gas medium into the material. In contrast, the tetragonal crystal structure (I4/mmm) typical of Be₁₂Me compounds facilitates more active oxide formation due to the lattice structure's inherent properties. These and related assumptions are further discussed in detail in Section 4.2.

4. Discussion

4.1. Analysis of the results obtained with crushed Be₁₂Cr samples

To analyze the experimental data from the three sequential corrosion experiments with a Be₁₂Cr sample, the release of corrosion products was systematically processed. The data were converted into normalized temperature dependences, which illustrate the variations in the partial pressures of the corrosion products and the rate of change in the sample's mass as a function of temperature. The processed results are presented in Fig. 11, providing a comprehensive description of the corrosion behavior of Be₁₂Cr under the experimental conditions.

From the graphs in Fig. 11, it is evident that the curves for hydrogen isotope release and the rate of mass change of the Be₁₂Cr sample during linear heating in gas-vapor mixtures are nearly identical. This similarity is observed for different compositions of gas-vapor mixtures.

The coincidence in the shape of the graphs showing the pressure of corrosion products and the rate of mass change during three heating cycles indicates that the corrosion process follows a single mechanism and reaction pathway. During linear heating, the interaction of the Be₁₂Cr sample with water vapor occurs on its surface, where oxygen from the water vapor is adsorbed, and hydrogen and deuterium dissociate and are released into the reaction chamber as H₂, HD, and D₂ molecules.

For each of the three heating experiments, the effective rate constant of the reaction between water vapor and the Be₁₂Cr sample was calculated using the specified formula.

$$K(T) = \frac{\frac{dm}{dt}(T)}{S \cdot P} \quad (1)$$

Where $\frac{dm}{dt}(T)$ - rate of change of sample mass at temperature T, mg/s, S - sample area, m², (The total geometric surface area of the samples used in the calculation was assessed by constructing a 3D model for each sample from the batch, followed by determining its surface area and then summing the surface areas of all the samples).

P - water vapor pressure, Pa.

The calculated values of the constant are shown on the graph in Arrhenius coordinates (Fig. 12).

The graph in Fig. 12 demonstrates that the effective interaction constant for the first experiment (first heating) is significantly higher than for the subsequent two heating cycles. However, the constants for the second and third experiments align closely with each other, despite differences in the composition of water vapor, as long as the total water

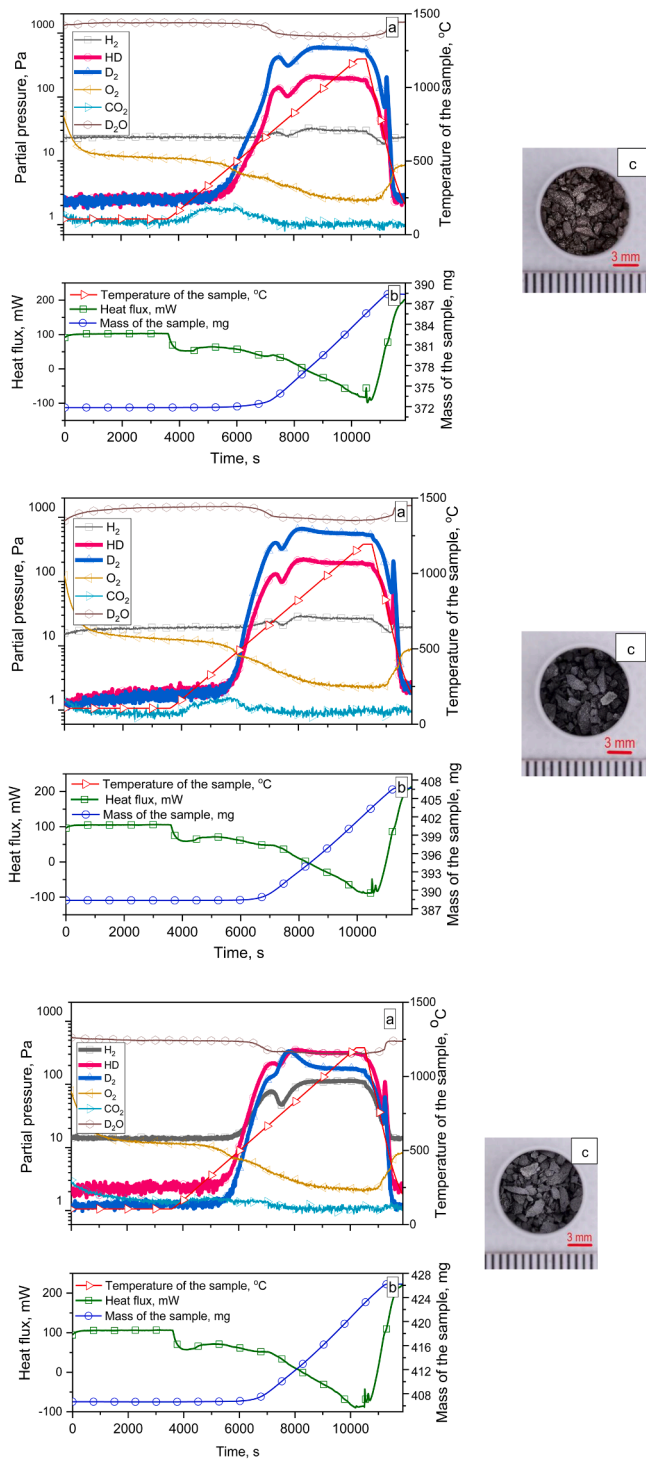


Fig. 8. Results of a series of high-temperature corrosion experiments (left) and appearance of crushed Be_2Zr samples at after corrosion experiment (right).

vapor pressure remains constant.

During the second and third heating cycles of the crushed Be_{12}Cr sample, distinct regions can be observed in the dependence of the effective interaction constant. In the low-temperature region (up to 900 °C), the slope of the curve is steeper, indicating more intense interaction within this temperature range. In contrast, in the high-temperature region (above 900 °C), the slope becomes more gradual, suggesting a decrease in the reaction rate or the potential achievement of equilibrium within the system.

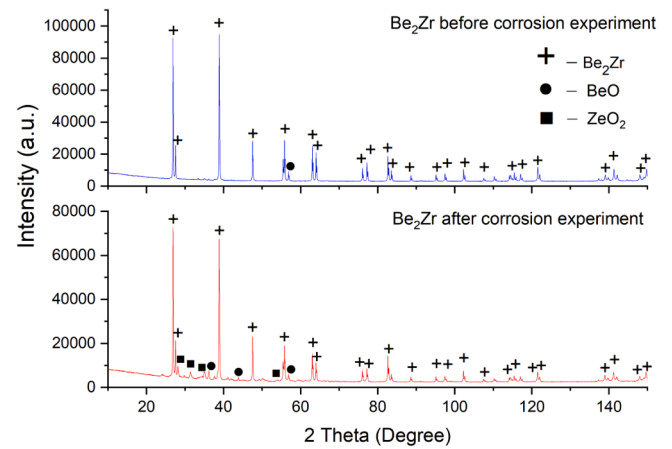


Fig. 9. XRD results of the Be_2Zr sample before and after corrosion experiments.

4.2. Analysis of the results obtained with crushed Be_2Zr samples

The corrosion process of beryllide zirconium Be_2Zr begins at temperatures above 400 °C. At this stage, oxygen-free beryllium and zirconium atoms are formed on the surface of the sample. These processes are accompanied by the development of corrosion interaction affecting the internal areas of the material grains, which is schematically shown in Fig. 13.

Free beryllium or zirconium atoms formed on the surface of the sample begin to interact with oxygen atoms according to the following reactions:



The terms on the right-hand side of Eqs. (2–7) represent reaction products, including the formation of an oxide layer and the release of H_2 and D_2 molecules, some of which may remain on the sample surface. H_2 and D_2 molecules may dissociate, react with each other, and be re-adsorbed on the sample surface as HD molecules. As a result of reactions (2–7), the mass of the sample increases because oxygen from the water vapor reacts with the material and remains in it as oxides such as BeO or ZrO_2 .

During linear heating of the Be_2Zr sample, the release of hydrogen isotopes passes through a distinct peak at a temperature of 740 °C. This peak is associated with the slowing down of the corrosion process, caused by the formation of a layer containing hydroxyl groups OD and OH on the surface of Be_2Zr . This layer blocks further interaction of water vapor with beryllide surface.

When the temperature exceeds 800 °C, hydroxyl groups associate with hydrogen and deuterium atoms, causing the desorption of D_2O and H_2O molecules from the sample surface. Simultaneously, the “wedge-shaped” cracking process [37] occurs, breaking down the protective oxide layer. This breakdown increases the concentration of free Be and Zr atoms along the grain boundaries, making the material susceptible to grain boundary corrosion.

Grain boundary corrosion is thought to be driven by two main factors:

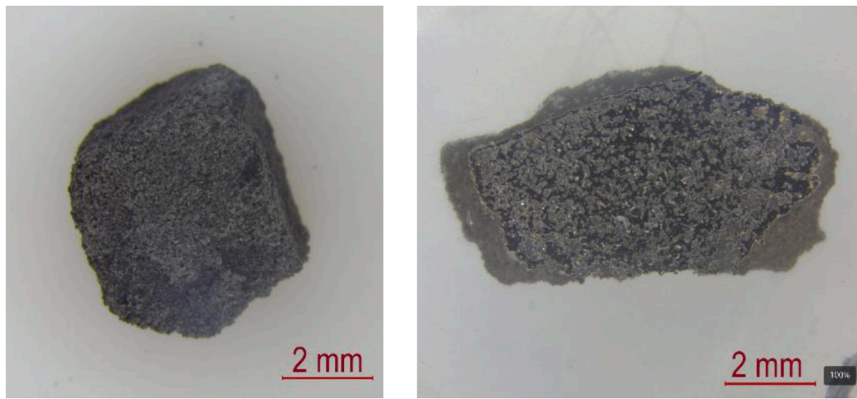
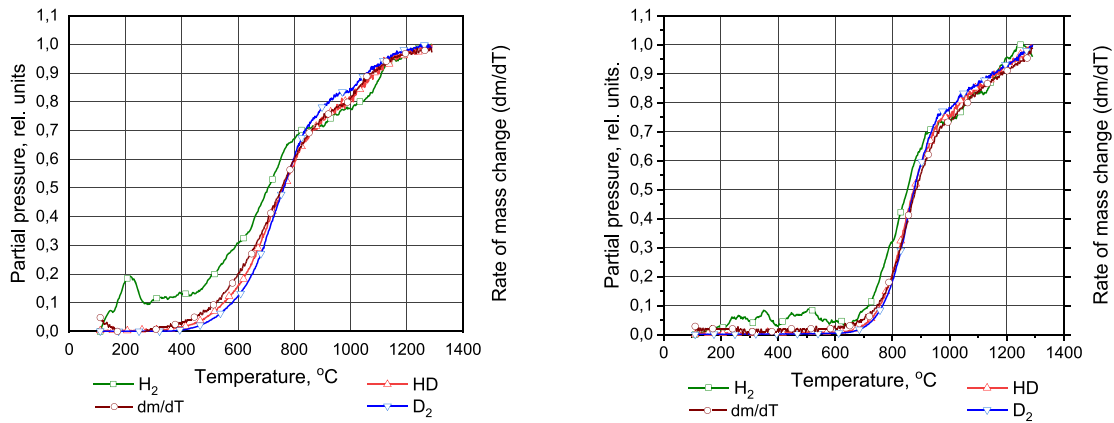
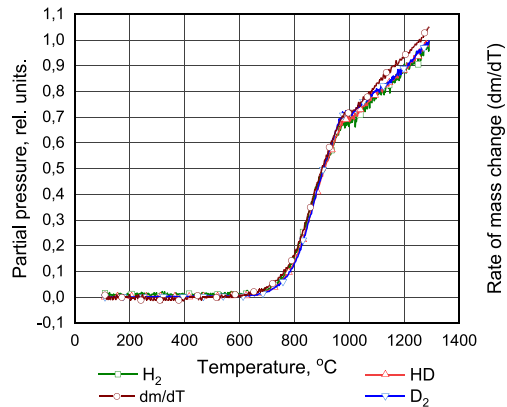


Fig. 10. Microstructural studies of the surface of crushed Be_2Zr samples before (left) and after corrosion experiments (right).



a) First experiment – the first heating of the Be_{12}Cr samples during purging with $\text{Ar} + \text{D}_2\text{O} + \text{HDO}$ mixture

b) Second experiment – the second heating of the Be_{12}Cr samples during purging with $\text{Ar} + \text{D}_2\text{O} + \text{HDO}$ mixture



c) Third experiment – the third heating of the Be_{12}Cr samples during purging with $\text{Ar} + \text{D}_2\text{O} + \text{HDO} + \text{H}_2\text{O}$ mixture

Fig. 11. Normalized temperature dependences of the change in pressure of corrosion products (H_2 , HD , D_2) and the rate of change in the mass of a crushed sample of Be_{12}Cr during sequential heating of the sample in different gas-vapor medium.

1. Cracking of the oxide film along grain boundaries: Mechanical stresses generated during the process lead to the destruction of the oxide film at the grain boundaries.

2. Increased chemical activity at grain boundaries: Structural imperfections and high reactivity make these regions particularly prone to corrosion.

Once initiated, grain boundary corrosion can become self-sustaining

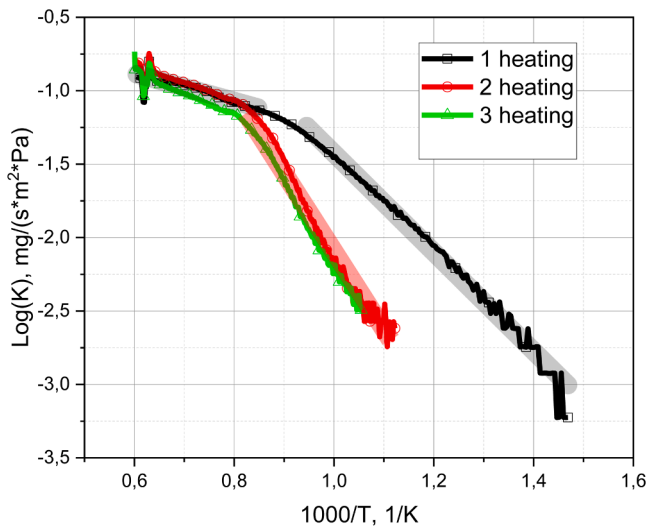


Fig. 12. Temperature dependences of the effective constant of interaction of water vapor with a Be_{12}Cr sample during three sequential heating in different vapor-gas medium.

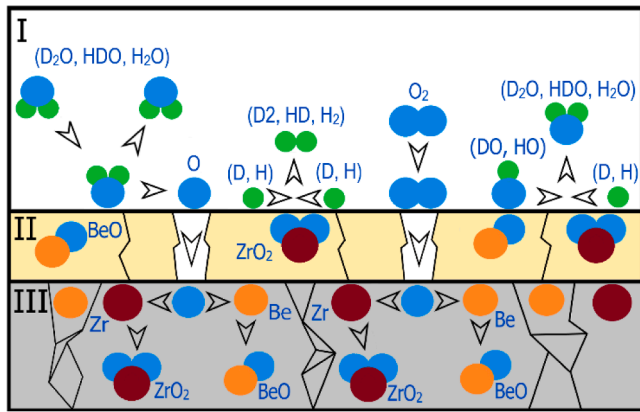


Fig. 13. Scheme of corrosion processes during the interaction of zirconium beryllide with a vapor-gas mixture.

due to the significant difference in the specific volumes of Be and BeO (~ 1.6). This volume mismatch generates stresses in areas where metal transforms into oxide, leading to the formation of an oxide "wedge" that separates metal grains and promotes further destruction [38]. These processes result in accelerated oxidation of the material following the breakdown of the protective oxide layer. Further research is required to develop strategies for enhancing the material's resistance to corrosion under such conditions.

When the sample temperature reaches 920°C and above 1200°C , an equilibrium process is established for the release of hydrogen isotopes (H_2 , D_2 , and HD) as well as D_2O and H_2O molecules. This indicates that the concentration of free Be and Zr atoms on the sample surface reaches a level where all oxygen atoms from water vapor molecules immediately interact with these free atoms. The formation of free beryllium and zirconium atoms primarily occurs along the split surfaces of the sample.

Upon cooling, the corrosion rate remains stable down to 650°C . However, the release of D_2 and HD molecules decreases significantly, likely due to the formation of hydroxyl groups (OD and OH) on the split surfaces. Below 750°C , the oxidation reaction nearly ceases as the concentration of free Be and Zr atoms decreases, resulting in stabilization of the sample surface and the cessation of degradation.

Based on this mechanism of interaction between water vapor and the Be_2Zr surface, the corrosion rate depends on three key factors:

1. Water vapor pressure ($P_{\text{D}_2\text{O}, \text{H}_2\text{O}}$): Higher pressure increases the number of water vapor molecules interacting with the surface.
2. Concentrations of free Be and Zr atoms ($N_{\text{Be,Zr}}$): These atoms act as reaction centers for oxygen interaction.
3. Interaction constants $K(T)$: These depend on temperature, increasing as the temperature rises, which accelerates corrosion processes.

For all three sequential experiments with the crushed Be_2Zr sample, the effective interaction constant of water vapor with the sample was calculated and is presented in Fig. 14 as Arrhenius dependences. The graph shows that in the low-temperature region, the interaction constant for the first experiment (first heating in the $\text{Ar} + \text{D}_2\text{O} + \text{HDO}$ medium) is noticeably lower than for the subsequent experiments. In these later experiments, the interaction constants for water vapor with the Be_2Zr sample align closely with each other.

The observed effects can be attributed to the presence of an initial carbide film on the surface of zirconium beryllide (Be_2Zr), which acts as a barrier to corrosion interactions. During the first experiment, this carbide film is destroyed and replaced by oxide films, altering the nature of the corrosion processes. A similar phenomenon was observed in experiments with Be_{12}Cr , where a discontinuity in the Arrhenius dependence of the effective interaction constant indicated a change in the reaction mechanism.

In the corrosion experiment with the Be_2Zr sample, when purged with a vapor-gas mixture of $\text{Ar} + \text{D}_2\text{O} + \text{HDO} + \text{H}_2\text{O}$, an isotope effect was observed in the release of corrosion products, including H_2 and D_2 molecules. The differences in the release patterns of hydrogen and deuterium molecules highlight the impact of the isotopic composition of the vapor-gas mixture on the kinetics of corrosion processes.

To illustrate the differences more clearly, Fig. 15 presents the normalized temperature dependences of the pressure changes for corrosion products during experiments with a Be_2Zr sample purged with the vapor-gas mixture of $\text{Ar} + \text{D}_2\text{O} + \text{HDO} + \text{H}_2\text{O}$. This visualization emphasizes the variation in the release behavior of hydrogen and deuterium molecules.

These dependencies allow us to identify the features of the interaction of hydrogen isotopes with the surface of the sample and the mechanisms of their release in different temperature ranges.

4.3. Comparative analysis of high-temperature corrosion processes of beryllides of different compositions

This section summarizes the results of the study on the corrosion processes of various beryllides and presents the calculated parameters of the effective interaction constant, including the pre-exponential factor (K_0) and the activation energy (E_a). The calculated parameters for the effective interaction constant of water vapor with different beryllides are compiled in Table 3.

It is important to note that the data for Be_{12}Ti were taken from our previous study [32] and are included here for comparison with the newly obtained results for Be_{12}Cr and Be_2Zr . This generalization enables a comprehensive comparison of the corrosion behaviors and reaction kinetics of these materials.

For beryllides with stabilized surfaces, it has been observed that activation energy values are significantly higher in the low-temperature range compared to the high-temperature range, indicating distinct corrosion mechanisms operating in these temperature regions.

The mechanism of beryllide (beryllium) corrosion in water vapor is well-discussed in [37] and is primarily attributed to the destruction of the oxide film along grain boundaries. This mechanism suggests that corrosion progresses predominantly along the grain boundaries, where cracking of the oxide film occurs at the edges of the underlying grains [39].

Once corrosion begins, the process accelerates due to the high volume ratio of beryllium to beryllium oxide (~ 1.6), which induces considerable stresses as the metal converts to oxide. These stresses lead

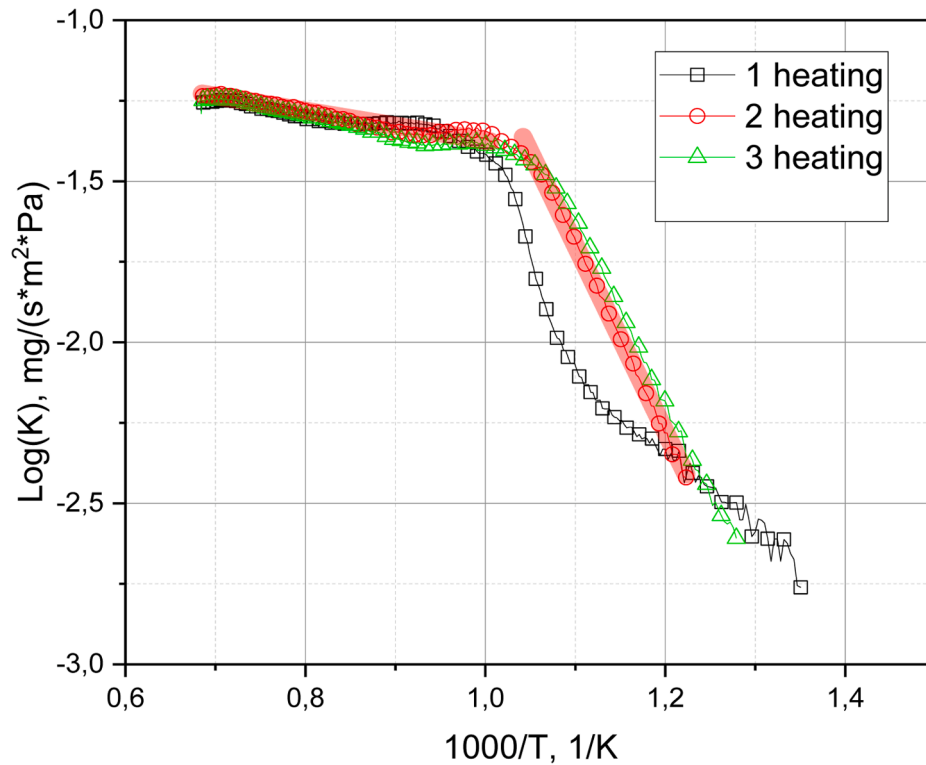


Fig. 14. Temperature dependences of the effective constant of interaction of water vapor with a Be₂Zr sample during three sequential heating in different vapor-gas medium.

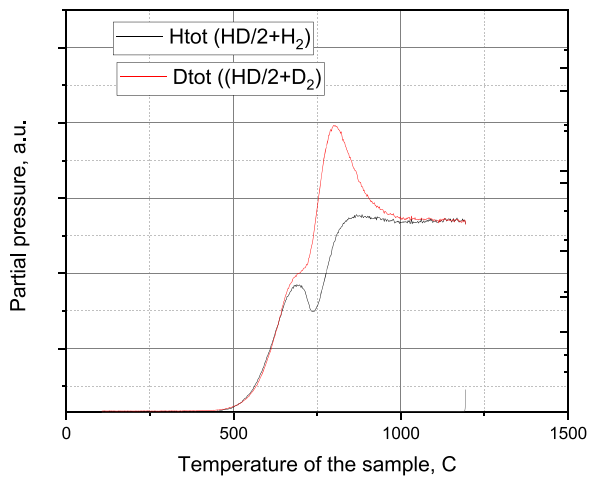


Fig. 15. Normalized temperature dependences of the change in pressure of corrosion products in experiments with a Be₂Zr sample during purging with an Ar + D₂O + HDO + H₂O vapor-gas mixture.

to the formation of an oxide "wedge" that separates the grains, further increasing the oxidation rate after the protective film is destroyed. Additionally, hydrogen diffusion into the oxide film may contribute to its degradation, as thinning of the oxide layer at higher temperatures can be explained by an increased proton diffusion rate and the attainment of a critical hydrogen concentration [40].

Fig. 16 presents a diagram developed to illustrate the proposed mechanism of wedge-shaped corrosion, emphasizing the destruction of the oxide film along grain boundaries and its impact on the progression of corrosion.

At the initial stage (I), no corrosion processes are observed because the oxide film effectively prevents the chemisorption and dissociation of

Table 3

Results of calculation of parameters of effective constant of interaction of water vapor with various beryllides.

Material	Condition	Temperature range, °C	K ₀	E _a
Be ₁₂ Ti	Before corrosion	500–1400	0.85	40
	After corrosion	750–1000	2200	130
		1000–1400	36	88
Be ₁₂ Cr	Before corrosion	400–850	87	64
		850–1400	0.48	18
	After corrosion	650–850	4050	111
Be ₂ Zr		850–1400	0.48	18
	After corrosion	500–650	45,900	90
		650–1200	0.12	18

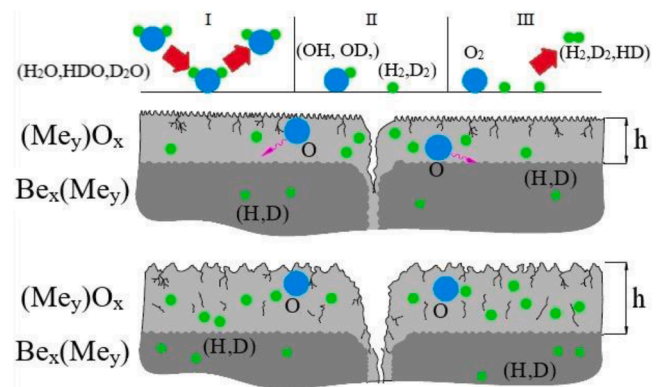


Fig. 16. Scheme of the corrosion of beryllides during linear heating and purging with a vapor-gas mixture.

water vapor molecules. During the second stage (II), adsorption of water vapor molecules on the material's surface begins. These molecules dissociate into oxygen and hydrogen (or deuterium) atoms. At the third stage (III), hydrogen and deuterium atoms associate, forming H_2 , HD and D_2 molecules that leave the surface. Simultaneously, some hydrogen and deuterium atoms penetrate the oxide film. Oxygen atoms react with unblocked beryllium atoms, forming BeO and increasing the thickness of the oxide film.

As the temperature rises, the stresses arising in the oxide film reach critical values, leading to the formation of cracks, particularly near the beryllide grain boundaries. These cracks expose additional unblocked beryllium atoms, initiating active corrosion processes.

Thus, the dynamics of beryllide corrosion are determined by complex interaction of a number of factors, including the stability of the oxide film, temperature conditions, and the mechanical properties of the material. Understanding these mechanisms is critical for developing materials with enhanced resistance to aggressive environments.

5. Conclusion

The conducted study enabled a comparative analysis of high-temperature corrosion processes in beryllides of various compositions under the influence of a vapor-gas medium. The experimental results confirmed the significant influence of both material composition and environmental conditions on the kinetics of corrosion processes, mechanisms of oxide phase formation, and changes in sample mass.

The results of the corrosion experiments demonstrated the formation of a thin BeO oxide layer was observed on the surface of all studied samples, with the additional formation of a ZrO_2 layer identified for Be_2Zr .

The data analysis allowed us to determine the key corrosion parameters and calculate the effective constants of the interaction of water vapor with the studied beryllides. In addition, the constants of the release of molecules containing hydrogen isotopes (H_2 , HD, D_2) arising as a result of the corrosion reaction were calculated.

Based on the results of the experiments, a "wedge-shaped" corrosion mechanism was proposed that explains the sequence of stages of destruction of the oxide film and the active interaction of water vapor with the surface of beryllides. It was found that the main corrosion parameters, such as the activation energy and the reaction rate constant, significantly depend on the composition of the material and temperature. These findings contribute significantly to understanding the mechanisms of beryllide corrosion and support the development of materials with enhanced corrosion resistance for use in fusion and hydrogen energy systems. Future research will focus on refining the understanding of corrosion mechanisms, investigating the effects of various additives on the corrosion resistance of beryllides, and modeling their behavior under conditions close to real operation.

CRedit authorship contribution statement

Timur Kulsartov: Validation, Formal analysis, Conceptualization. **Inesh Kenzhina:** Writing – original draft, Project administration, Funding acquisition. **Kuanysh Samarkhanov:** Writing – review & editing, Investigation. **Yergazy Kenzhin:** Formal analysis. **Yuriy Gordienko:** Resources, Methodology. **Yuriy Ponkratov:** Visualization, Validation. **Yevgeniy Chikhray:** Software, Investigation. **Vadim Bochkov:** Investigation, Data curation. **Zhanna Zaurbekova:** Writing – review & editing, Formal analysis. **Alexander Yelishenkov:** Validation, Software. **Meiram Begentayev:** Project administration. **Saulet Askerbekov:** Data curation. **Sergey Udartsev:** Resources. **Mikhail Podoinkov:** 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.

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Data availability

Data will be made available on request.

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