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Study of hydrogen sorption and desorption processes of zirconium beryllide $ZrBe_2$

Yergazy Kenzhin [a], Inesh Kenzhina [a,b,*], Timur Kulsartov [a,c], Zhanna Zaurbekova [a,c], Saulet Askerbekov [a], Yuriy Ponkratov [a,c], Yuriy Gordienko [c], Alexandr Yelishenkov [a], Sergey Udartsev [d]

[a] Kazakh-British Technical University, Almaty, Kazakhstan
[b] Satbayev University, Almaty, Kazakhstan
[c] Branch "Institute of Atomic Energy" of National Nuclear Center of the Republic of Kazakhstan, Kurchatov, Kazakhstan
[d] JSC "Ulba Metallurgical Plant", Ust'-Kamenogorsk, Kazakhstan

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ABSTRACT

One of the promising intermetallic compounds for use in nuclear and fusion reactors, as well as in hydrogen energy technology, are intermetallic compounds of beryllium with metals such as Ti, V, Zr and Nb. Beryllium-based intermetallics are not only a promising material for blankets of future fusion reactors, but can also be utilized in other areas of the nuclear industry, including fission reactor reflectors, rocket and space technology. The interest in studies 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 under neutron irradiation, and is caused by the need for understanding the processes arising from such interaction.

In this work, hydrogen sorption and desorption processes of zirconium beryllide $ZrBe_2$ produced by industrial technologies at JSC "Ulba Metallurgical Plant" (JSC UMP) were investigated. The experiments were performed by the Sievert's and thermal desorption spectrometry (TDS) methods. In TDS experiments deuterium was chosen for samples saturation to reduce the possible error in determining fluxes associated with the release of hydrogen from the elements of the vacuum chamber.

An equation for hydrogen solubility in zirconium beryllide $ZrBe_2$ was obtained by processing the experimental data obtained by the Sievert's method:

$$S = (0.46 \pm 0.1)\frac{mol}{m^3 \bullet Pa^{\frac{1}{2}}} \bullet \exp\frac{(-13.3\pm0.5)\frac{kJ}{mol}}{RT}$$

The temperature intervals of formation and decomposition of two different hydride phases (deuterides) of zirconium beryllide $ZrBe_2$ at ~ 600 K and ~ 900 K have been established in TDS experiments.

1. Introduction

Beryllides are intermetallic compounds of beryllium with other metals, for example, zirconium, tantalum, titanium, nickel or cobalt, and are a promising but insufficiently studied materials, the use of which is promising in nuclear, fusion and hydrogen energy, for example, as functional elements of fusion reactor blankets, in beryllium reflectors of fission nuclear reactors, as well as materials for storing hydrogen in the hydrogen industry and energy [1].

Interest in such intermetallic compounds, in particular, is due to their ability to absorb large amounts of hydrogen, forming hydrides, which makes it possible to reduce the volume of the system by three times compared to the volume of storage in gas cylinders [2,3].

These properties of intermetallic compounds began to be used to solve the safety problems of hydrogen energy as an alternative to storing hydrogen in the gas phase, which is associated with a high explosion hazard and its negative impact on the properties of structural materials (hydrogen embrittlement). Intermetallic compounds used as hydrogen batteries consist, as a rule, of two types of metals: hydride-forming (Ti, Mg, Zr, rare-earth metals, etc.) and catalysts (Ni, Cr, Fe, Mn, etc.).

In addition, Be-containing intermetallic compounds involving secondary metals such as Ti, V, Zr and Nb are also attracting attention as a

* Corresponding author.
E-mail address: kenzhina@physics.kz (I. Kenzhina).

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promising alternative to pure Be-based neutron multipliers in nuclear fusion applications due to their lower chemical reactivity with steam, which can dramatically reduce the release of hydrogen during a loss of coolant accident [4–6].

Computational studies using density functional theory (DFT) calculations [7] showed lower energy costs for hydrogen dissolution in $Be_{12}Ti$ and $Be_{12}V$ shells, indicating easier dissolution of tritium from crystalline shells. However, it is still unclear why beryllide compounds release hydrogen isotopes so easily and have reduced energy costs for hydrogen dissolution. Therefore, from a fundamental point of view, it is important to have more experimental data to predict the properties of beryllides.

In the studies by Maeland and Libowitz [8,9] of zirconium beryllide $ZrBe_2$ as a hydrogen storage material, it was found that it can rapidly sorb hydrogen at atmospheric pressure H_2 and room temperature to form hydride $ZrBe_2H_{1.5}$. In addition, $ZrBe_2H_{1.5}$ was found to have negligible hydrogen dissociation pressure (<1 kPa) at room temperature. One isotherm of hydrogen absorption (above the critical temperature) at 588 K has been reported [8], which gives some additional indications on the stability of $ZrBe_2H_x$ with respect to hydrogen content, with hydrogen pressure varying from 3 Pa at $x \approx 0.2$ to 0.1 MPa at $x \approx 1$. In another study by authors Kodibagkar et al. [10] it was reported that all hydrogen can be effectively removed at 973 K in vacuum.

Considering the urgency of the task of creating new materials based on beryllium, JSC UMP has developed an industrial technology for creating beryllides such as $TiBe_{12}$, $ZrBe_2$, $CrBe_{12}$, Ta_2Be_{17}, $TaBe_2$ and $NbBe_2$. Full-scale studies of titanium beryllide $TiBe_{12}$ were carried out jointly by scientists from Karlsruhe Institute of Technology (KIT) and JSC UMP [11–13], who confirmed the promising properties of this material for using it as a neutron multiplier in the helium-cooled breeding blanket (HCPB) concept for EU DEMO. However, relatively little experimental research has been carried out to study the interaction of zirconium beryllide $ZrBe_2$ with hydrogen.

Therefore, in order to use zirconium beryllide $ZrBe_2$ in various branches of engineering, it is important to study its characteristics of hydrogen sorption and desorption processes depending on temperature, pressure, cyclicity of sorption/desorption processes, the influence of the impurity gases composition with significant changes in the physical properties of the material during its saturation with hydrogen and a number of other characteristics. This aim can be achieved by more complete experimental and theoretical studies.

The present work presents a methodology for studying the processes of hydrogen sorption and desorption performed on samples of zirconium beryllide $ZrBe_2$ provided by JSC "Ulba Metallurgical Plant". The experiments were performed by the Sievert's and TDS methods.

2. Materials and methods

2.1. Test sample

The test sample of zirconium beryllide $ZrBe_2$ is a compacted porous material fabricated by hot vacuum pressing technique at JSC "UMP" (Ust'-Kamenogorsk, Kazakhstan).

Since the studies were carried out using the Sievert's and thermal desorption spectrometry (TDS) methods, several samples were prepared. The Sievert's method study of zirconium beryllide $ZrBe_2$ was performed on crushed samples with an average particle size of 2–4 mm with a total weight of 0.3631 g (Fig. 1).

For TDS studies, small zirconium beryllide samples weighing 0.01825 g and 0.0248 g were used (Fig. 2).

In Fig. 3 the results of X-ray phase analysis of the test samples $ZrBe_2$ are presented. The intensity of the diffraction peaks is presented as the square root of the original values.

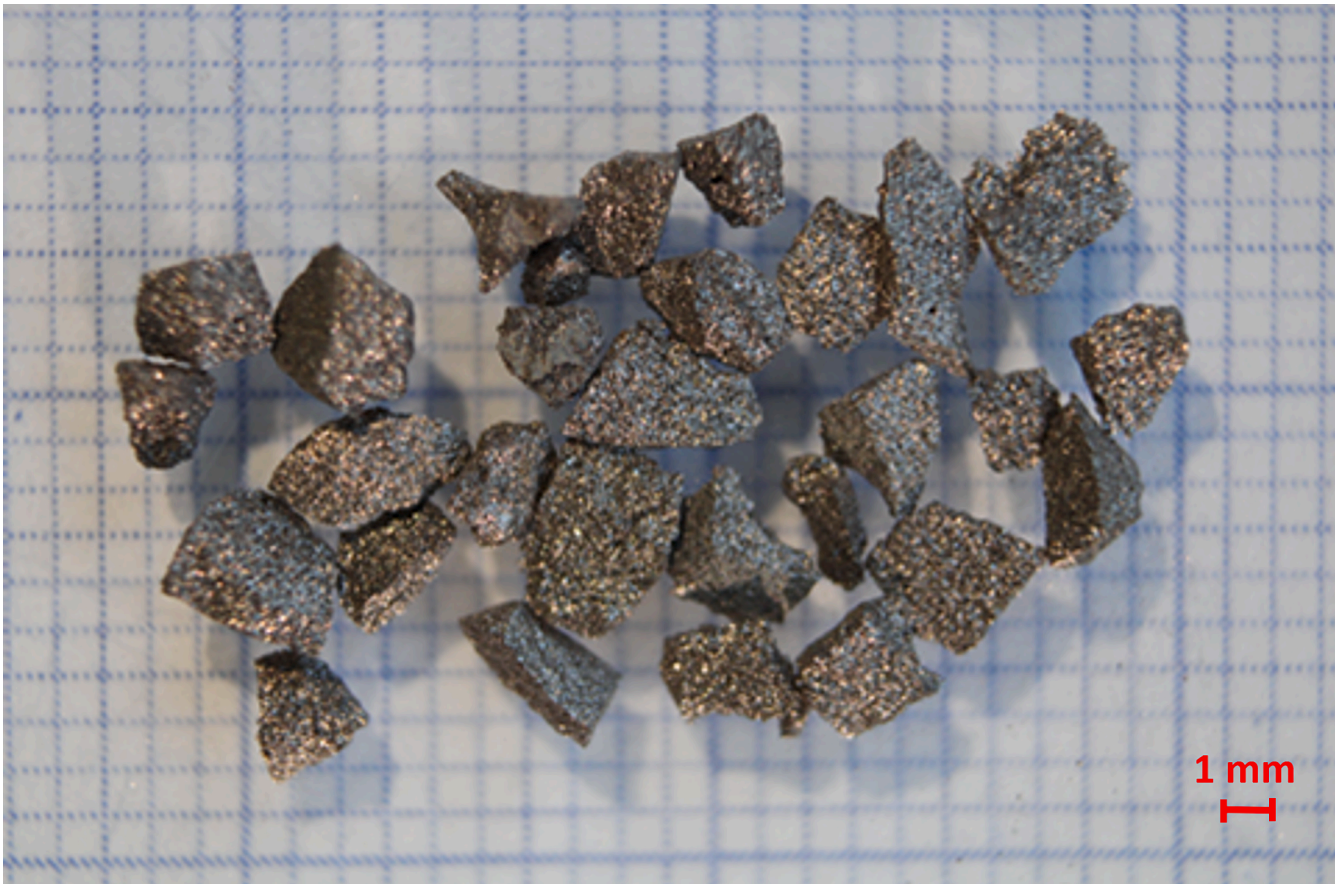

Fig. 1. Crushed sample of zirconium beryllide $ZrBe_2$ weighing 0.3631 g.

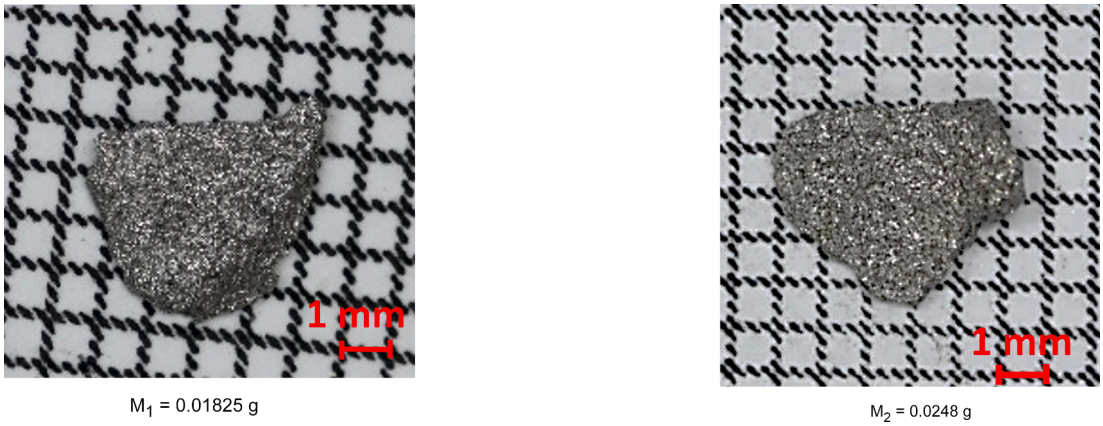

Fig. 2. Zirconium beryllide $ZrBe_2$ samples in TDS experiments.

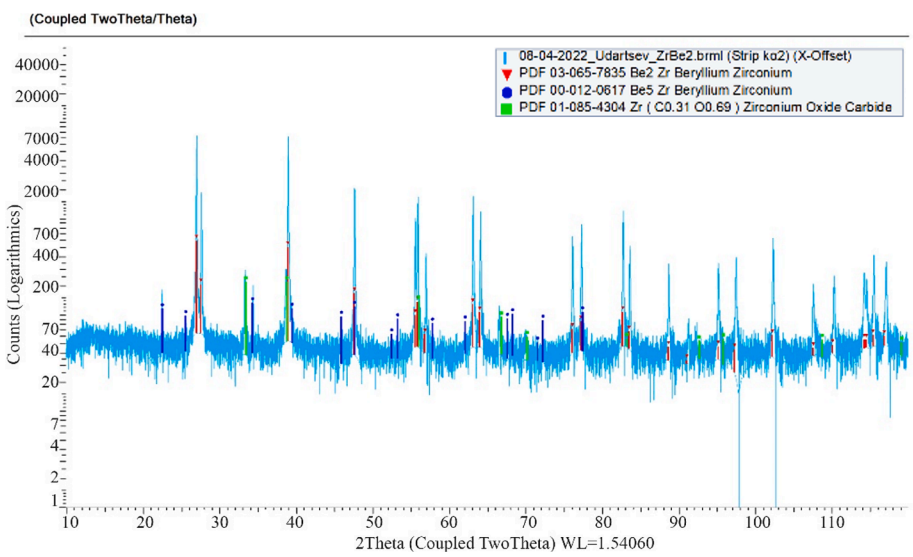

Fig. 3. Results of X-ray phase analysis of zirconium beryllide $ZrBe_2$ (blue line is the experimental curve obtained by scanning; red line – characteristic peaks for $ZrBe_2$; dark blue line – characteristic peaks for $ZrBe_5$).

The results of X-ray diffraction analysis of zirconium beryllide (the blue line in Fig. 3 is the experimental curve obtained by scanning) showed that the diagrams contain mainly peaks characteristic for $ZrBe_2$ (red line) and $ZrBe_5$ (dark blue line) compounds. It is worth saying that the presence of a 2–3 % hexagonal impurity phase of $ZrBe_5$ was discovered by Hauback, B.C. et al. [14], who obtained neutron powder diffraction patterns for various types of samples $ZrBe_2H_{1.5}$, $ZrBe_2H_{1.2}$ and others at different temperatures.

It should be noted that the $ZrBe_5$ compound is the next intermetallic compound after $ZrBe_2$ with a higher ratio of the number of atoms Be/Zr = 5, which can be formed during the synthesis process (according to phase diagrams, see Fig. 4).

The presence of $ZrBe_5$ may arise as a result of an error in weighing the initial components of the mixture, or partial losses during their mixing and pressing, which leads to a deviation from the stoichiometric mass ratio. Also, the reason for the presence of $ZrBe_5$ may be the inhomogeneity of the distribution of components in the sample after pressing, as a result of which the synthesis of this beryllide became possible. The mixture may contain zirconium oxycarbide $ZrC_{0.31}O_{0.69}$, which could initially be present in the powder, formed during its manufacture.

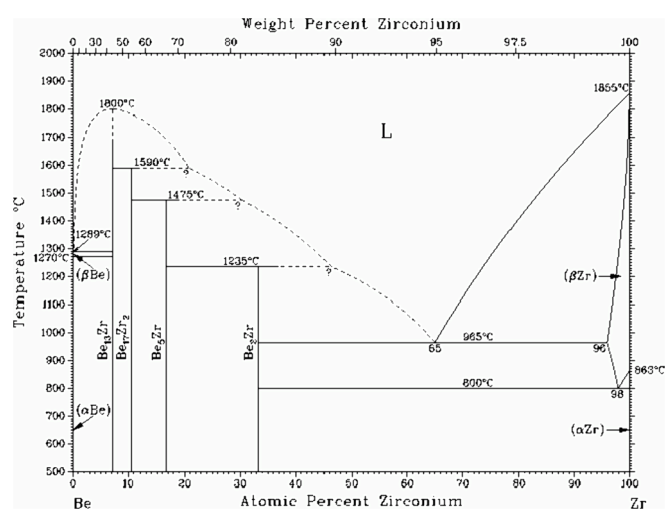

Fig. 4. Be-Zr phase diagram [15].

2.2. The Sievert's method experiments

The most common method for studying hydrogen sorption by materials is the Sievert's method [16,17], in which the sample is saturated from a gas atmosphere. This method consists in calculating the change of hydrogen content in the sample by the change of pressure and temperature based on the equation of state. The advantage of the volumetric method is that it is relatively simple and inexpensive. At the same time, the accuracy of volumetric methods is potentially higher than that of gravimetric methods because they measure large changes in gas pressure rather than small changes in mass. The gravimetric method allows you to determine the amount of hydrogen absorbed by a sample by measuring the change in its mass. This method is considered technically more complex than the volumetric Sievert's method, since it is necessary to use highly sensitive microbalances and more strictly select the mass of the sample so that it is possible to record even small changes due to absorbed hydrogen.

Fig. 5 shows a schematic diagram of the experimental setup that allows studying materials by the Sievert's method. The samples were loaded into a stainless steel Cr18Ni10Ti capsule and then placed in the experimental cell with an ohmic heater allowing heating the samples up to 873 K. The samples temperature is measured by two K-type thermocouples (made of chromel–alumel alloy) through the AD8495 module. AD8495 (Analog Device) is a precision thermocouple amplifier with cold junction compensation for K-type thermocouples, provides voltage output U, V and linear conversion $T,^{\circ}C = \frac{(U-1250)mV}{5mV/^{\circ}C}$ with accuracy ± 1 °C. Hydrogen is supplied to the chamber through V5 and V6 valves, hydrogen pressure relief from the system to the atmosphere is performed through V2 valve, vacuum pumping is performed through V3 and V4 valves. Pressure is registered by piezo-resistive pressure transducers P1 and P2, with measuring range from 0 to 10 MPa and accuracy ± 0.5 % FS (full scale).

Volumes of the gas-vacuum system of the experimental setup were measured sequentially using helium. First, the volume of the experimental cell was measured by filling with alcohol; then, filling the other volumes of the setup with helium at a known pressure, and using the ideal gas equation, their volumes were calculated. As a result, the volume of the working part of the setup with the sample and pressure sensor R1 + R2 was determined to be equal to $1.15 \cdot 10^{-7}$ m^3, with open V4 valve and closed V3 valve.

Previously, a series of experiments on the effect of experimental cell's temperature on the gas pressure increase in the measuring volume without the samples were performed. These data were necessary to make a correction for the change in hydrogen pressure during the experiments with the test samples.

Taking into account the accuracy of determining chamber volumes (±0.1 cm^3) and the influence of temperature in the chamber with samples on the pressure value, the error in measuring the absolute pressure level in the chamber was less than 100 Pa (at low (~500 Pa) pressures and samples' temperatures less than 473 K) and less than 1000 Pa (at high (~10^5 Pa) pressures at samples' temperatures above 673 K).

Since during the heating experiments the pressure varied from ~ 300 Pa to $2 \cdot 10^5$ Pa, the total hydrogen content in the samples at different temperatures was measured with an accuracy of no worse than 5 %, and the largest error was at the maximum temperature of the samples.

The Sievert's method study to determine the sorption characteristics of zirconium beryllide $ZrBe_2$ samples consisted in obtaining experimental data on the dependence of hydrogen pressure change in vacuum chamber on temperature during stepwise heating of the samples from 273 K to 873 K.

Initially, several cycles of experiments were carried out with different hydrogen pressures in the chamber, but the beryllide samples absorbed hydrogen poorly. Therefore, to start the process of hydride formation in the test samples, a series of experiments on the activation of the $ZrBe_2$ samples were carried out first. A method for activating samples of zirconium beryllide $ZrBe_2$ was developed, which consists of exposing beryllide samples to high pressure of hydrogen (about 1.4 MPa) and high temperatures about 800 K, with periodic pumping out of the hydrogen medium, with exposure under these conditions for more than two hours. Continuous real-time pressure monitoring was performed during the activation procedure. Two cycles of samples activation were carried out. Fig. 6 shows a diagram of the activation mode of the samples, after which their ability to absorb hydrogen significantly was recorded.

After activation a series of experiments using Sievert's method was carried out, in some of which the samples were heated linearly (at a rate of 5–20 K/min), in other experiments the samples were heated stepwise at 50–100 degrees, holding on each temperature level for up to 20 min. Then the stage of the experiment with samples cooling was analyzed, which was carried out by a stepwise decrease in temperature through 50–100 degrees with holding at each level for up to 20 min.

2.3. TDS experiments

Another method for studying the interaction characteristics of hydride-forming materials is the method of thermal desorption spectroscopy (TDS), which determines the parameters of the process of hydrogen and its isotopes desorption from a pre-saturated sample during

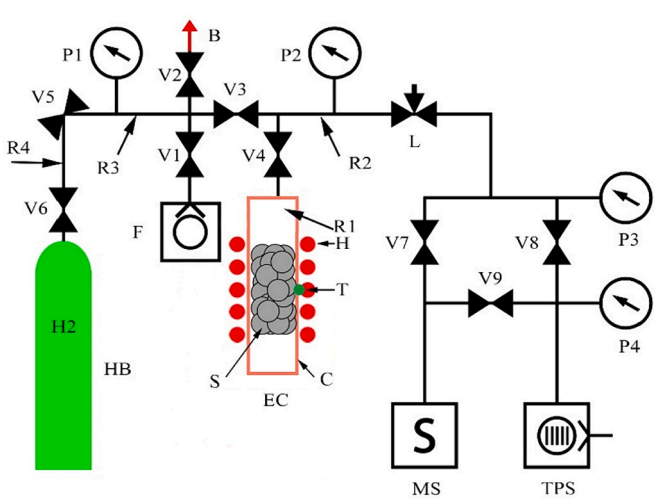

Fig. 5. Schematic diagram of setup by Sivert's method. HB – hydrogen cylinder, EC – experimental cell, R – working volumes, P – pressure sensors, V – vacuum valves, TPS – turbomolecular pump, MS – mass spectrometer, F – forevacuum pump, H – heater, T – thermocouple fixturing point, C – working chamber, S – samples, B – excess pressure discharge, L – mechanical leak-valve.

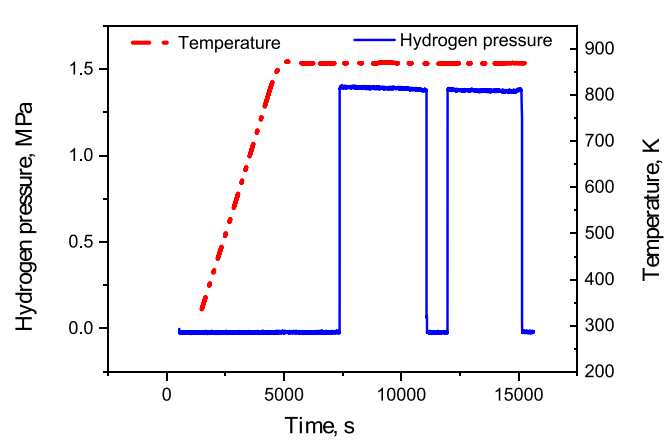

Fig. 6. Activation process of $ZrBe_2$ samples.

programmed linear heating with mass spectrometric registration of the released gases. The experimental setup and methodology are described in detail in [18].

In TDS experiments, pre-activated zirconium beryllide $ZrBe_2$ samples after high-temperature degassing at 1073 K were saturated with deuterium at 873 K and ~ 60 torr pressure for 5 h. Then the samples were cooled to a temperature of 293 K and the working chamber was pumped down to ~ 10^{-6} torr. After that, the TDS measurements were started in the mode of programmed linear heating to 1273 K at rates of 5 and 10 K/min with samples of M_1 and M_2, respectively (Fig. 2).

Deuterium was chosen for sample loading up to saturation level, since TDS experiments were supposed to be carried out in vacuum conditions, and there was an intention to reduce the possible error in determining fluxes associated with the release of hydrogen from the crucible elements and holders in a vacuum chamber when the sample was heated. As further studies showed, deuterium fluxes were significantly higher than the background hydrogen release.

3. Results and discussion

3.1. Results of the Sievert's method studies

After activation of the $ZrBe_2$ zirconium beryllide sample, a series of experiments were carried out to measure the dependence of hydrogen pressure change in the setup chamber during linear heating to a temperature of 873 K of the pre-hydrogenated sample, followed by stepwise cooling, the results of which are presented in Fig. 7.

As can be seen from the Fig. 7, as the temperature increases, degassing of the samples occurs and hydrogen is released into the experimental chamber. In the presented experiments, the maximum hydrogen content in beryllide was observed at room temperature and corresponded to the hydrogen content x = 0.82 ($ZrBe_2H_{0.82}$).

The dependence of pressure change rate in the chamber with samples is shown in Fig. 8. This graph was obtained by differentiating the smoothed dependence of pressure change in the chamber during linear heating of the samples. It should be noted that the maximum rate of hydrogen evolution occurs at a temperature of about 580 K, which may indicate the decomposition of the hydride phase.

The temperature dependence of the hydrogen content x in zirconium beryllide Be2ZrHx is presented in Fig. 9 (the calculation of the hydrogen content in the samples was carried out for the hydrogen pressures of the samples obtained at a constant temperature). The authors suppose that up to a temperature of 600 K hydrogen in the samples was in some intermediate hydride phase, after which the hydride decomposes and hydrogen is released into the gas phase; and after temperatures above 673 K the main amount of hydrogen is in a state not bound into a hydride.

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As a result of processing the experimental data measured by the Sievert's method, an equation for hydrogen solubility was obtained for temperature range from 673 to 873 K (Fig. 10). Solubility was calculated for temperature regions above 623 K, where it is assumed that hydrogen is mainly present in a non-hydride state:

$$S = (0.46 \pm 0.1)\frac{mol}{m^3 \bullet Pa^{\frac{1}{2}}} \bullet \exp\frac{(-13.3 \pm 0.5)\frac{kJ}{mol}}{RT},$$

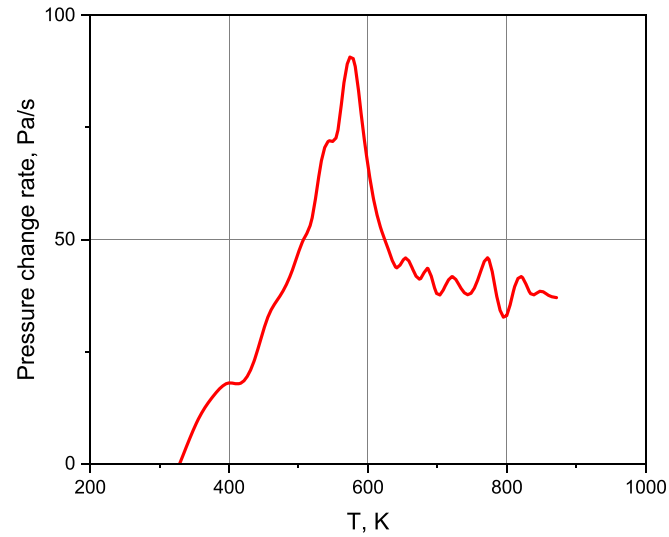

Fig. 8. Dependence of the rate of hydrogen pressure change in the chamber at a linear increase in the samples temperature of 5 K/min.

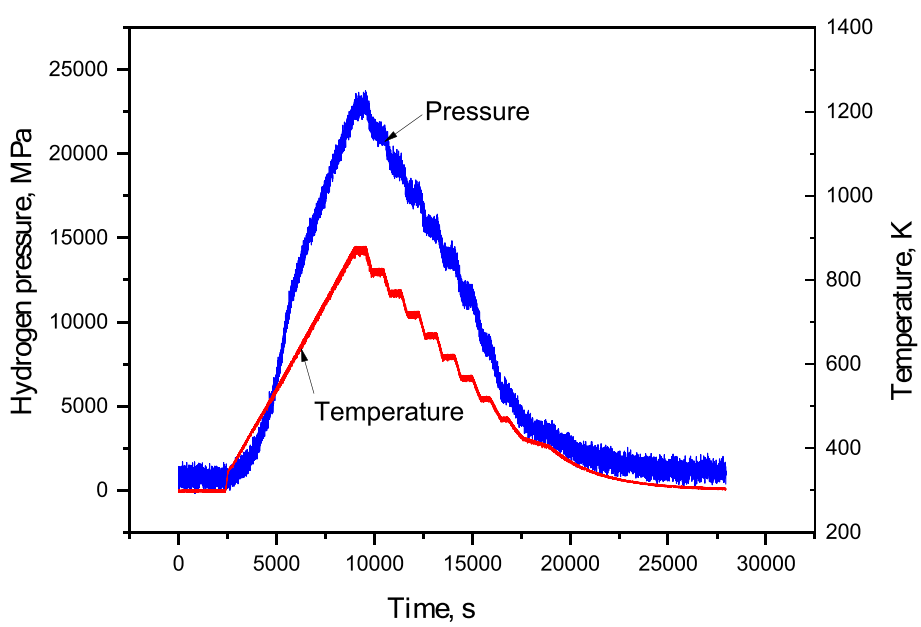

Fig. 7. Dependence of hydrogen pressure change in the setup chamber during linear heating and subsequent cooling of $ZrBe_2$ zirconium beryllide sample by Sievert's method.

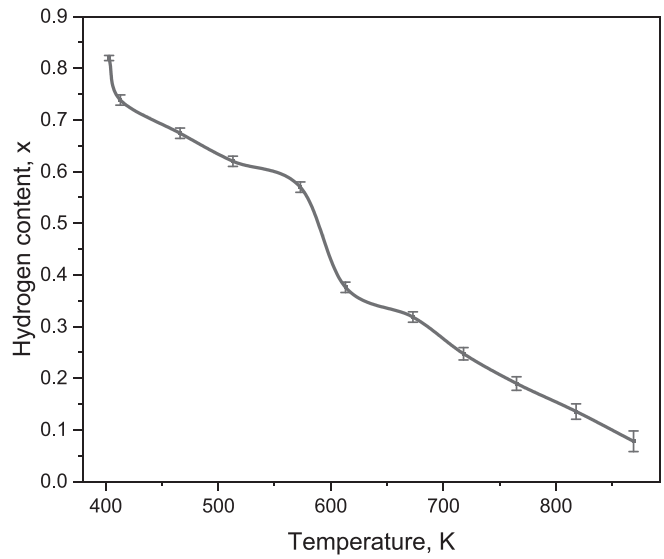

Fig. 9. Temperature dependence of the hydrogen content x in zirconium beryllide $ZrBe_2H_x$.

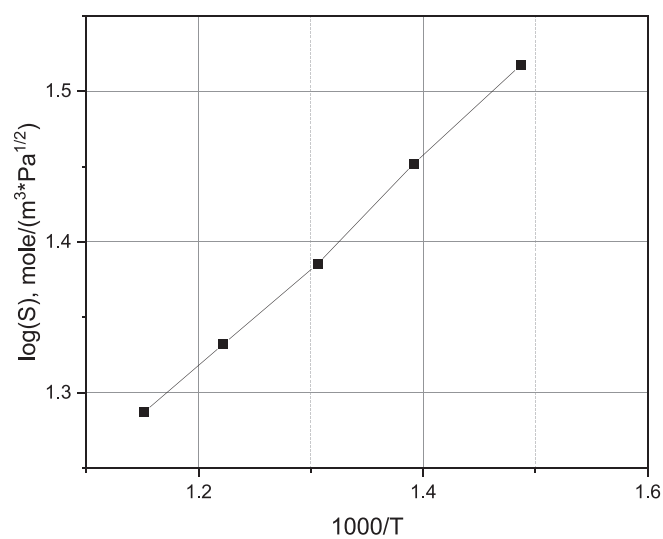

Fig. 10. Temperature dependence of hydrogen solubility S in $ZrBe_2$.

where $S_0 = 0.46 \pm 0.1$ (mol/($m^3 \cdot Pa^{1/2}$)) is the solubility constant, H_s = -13.3 ± 0.5 kJ/mol is the heat of solution.

Hydrogen solubility was calculated from the dependences of pressure changes when cooling samples for each temperature level:

$$S(T) = \frac{\nu_0 - P(T) \bullet V_{ch}/(R \bullet T)}{V_s \bullet \sqrt{P(T)}},$$

where ν_0 is the amount of hydrogen supplied to the chamber with samples (mol); P(T) is the quasi-equilibrium pressure of hydrogen in the chamber at sample temperature T (Pa); V_{ch} and V_s are the volumes of the saturation chamber and samples, respectively (m^3).

3.2. Results of TDS experiments

In studies of zirconium beryllide $ZrBe_2$ samples by thermodesorption spectroscopy, TDS spectra of deuterium desorption in the process of linear heating with heating rates of 5 and 10 K/min were obtained, which are presented in Fig. 11. It should be noted that the integral amounts of released deuterium coincided quite well for two samples M_1 and M_2 (taking into account the samples' masses), and the content of deuterium corresponded to $ZrBe_2D_{0.94}$ compound.

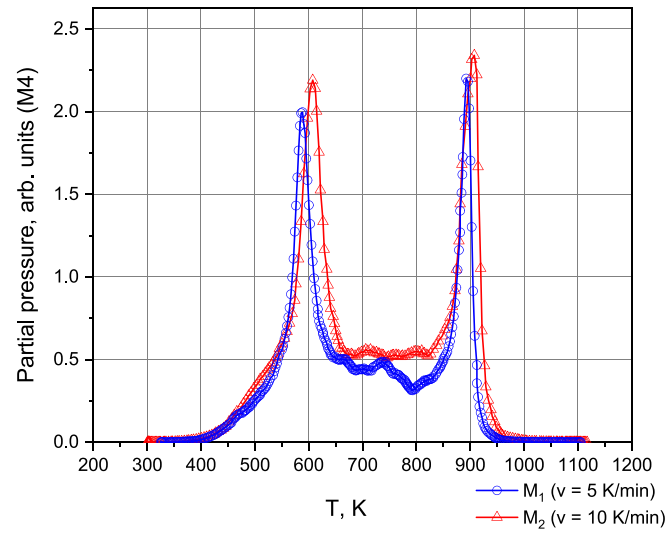

Fig. 11. Deuterium release spectrum from $ZrBe_2$ in TDS experiments at different sample heating rates 5 and 10 K/min.

It was found that the deuterium release has a non-diffusive character and has two peaks at temperatures of ~ 600 K and ~ 900 K, which suggests that two phases with different decomposition temperatures are formed in the process of hydride formation (Fig. 11). It is worth noting that the second phase (at a higher temperature) can be formed after the decomposition process of the first phase, during degassing of the samples. The process of gas release in this case can be described by diffusion in the presence of traps of limited capacity in the samples (McNabb Foster model); capture into traps and release from them will be determined by parameters having an Arrhenius dependence.

Conclusion

Experimental studies of the sorption characteristics of zirconium beryllide $ZrBe_2$ have been conducted by the Sievert's method and the thermal desorption method.

The technique of activation of zirconium beryllide $ZrBe_2$ samples before experiments has been worked out.

An expression for hydrogen solubility in zirconium beryllide $ZrBe_2$ was obtained by processing the experimental data obtained by the Sievert's method:

$$S = (0.46 \pm 0.1)\frac{mol}{m^3 \bullet Pa^{\frac{1}{2}}} \bullet \exp\frac{(-13.3 \pm 0.5)\frac{kJ}{mol}}{RT}.$$

The temperature intervals of formation and decomposition of two different hydride phases (deuterides) of zirconium beryllide $ZrBe_2$ at ~ 600 K and ~ 900 K have been established in TDS experiments.

In summary, this paper presents the results of the first studies of zirconium beryllide $ZrBe_2$ produced by JSC UMP, and is focused on the study of samples of zirconium beryllide fabricated in real industrial production; sets the impetus for more detailed studies of this material (and similar beryllides) in the future.

CRediT authorship contribution statement

Yergazy Kenzhin: Writing – review & editing, Writing – original draft, Investigation. **Inesh Kenzhina:** Writing – original draft, Investigation. **Timur Kulsartov:** Writing – review & editing, Investigation, Formal analysis, Conceptualization. **Zhanna Zaurbekova:** Writing – review & editing, Investigation. **Saulet Askerbekov:** Visualization, Methodology, Investigation. **Yuriy Ponkratov:** Resources,

Methodology, Investigation. **Yuriy Gordienko:** Resources, Methodology, Investigation. **Alexandr Yelishenkov:** Investigation. **Sergey Udartsev:** Resources, Methodology, Data curation.

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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