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# Thermal-radiation processes in the MeIV–H system. Synthesis of hydrides of metals

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## Abstract

A special hermetic chamber was elaborated and constructed to treat materials by the electron beam in vacuum and in hydrogen atmosphere ($P_H = 1$–$2$ atm; 1 atm$=$101 325 Pa). The investigation was carried out on the high current electron accelerator LAE-5 using a focused electron beam. The system of temperature measurement during irradiation was developed. It allowed measurement of the thermal effects taking place under the action of irradiation, and the establishment of the main features of thermal-radiation processes in the Me–H system. As a result of experiments at different doses and power of dose (0.025–1 MRad/s) in vacuum and in the hydrogen atmosphere, the main peculiarities of the processes have been observed, such as: (i) the thermal-radiation synthesis of hydrides of IV group metals of stoichiometric composition – $TiH_2$, $ZrH_2$, and $HfH_2$; (ii) the phenomenon of 'cold synthesis' of hydrides. As a result of this phenomenon hafnium hydride of super-stoichiometric composition, $HfH_{2.4}$, has been obtained.

*Keywords:* Irradiation; Electron beam; Metal hydrides; Combustion synthesis

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## 1. Introduction

In this work, the results of a series of experiments to investigate the effect of an accelerated electron beam on the processes of interaction of IV group metals with hydrogen are presented.

From the literature it is known that irradiation of metals and inorganic materials by an electron beam is accompanied by essential changes of their physical, chemical and mechanical properties (for example – improvement of the catalytic, strength and other characteristics). It is also known that ionization and excitation of electronic and nuclear sub-lattices are the primary effects of solid state irradiation. Under ionizing radiation of sufficient energy, the distortion of structure and/or formation of defects are usually observed in solid states [1–3].

Thermal-chemical-radiation processes in Me–H systems under accelerated electron beam actually are not investigated. In one work [4], information was presented on thermal-radiation synthesis (TRS) of hydrides on the basis of ZrMe (Me – Ni, Co, Fe). The initial intermetallic powder was exposed to the short-term irradiation (1.0–20.0 min) in a hydrogen atmosphere by electrons accelerated up to 2.0 MeV. As a result, intermetallic hydrides $ZrNiH_{2.8}$, $ZrCoH_{2.8}$ and $Zr_{1-y}Fe_yH_x$ were obtained.

Since 1974, the combustion in the systems Me–H, Me–N–H, Me–C–H, Me–Me′–H and others by the method of self-propagating high-temperature synthesis (SHS) has been investigated systematically in our laboratory. It has been the priority direction method of our research. The main outcome of the performed investigations is the development of a complex of scientific tasks on the synthesis of various hydrides [5–7]. More than 150 binary and complex hydrides, as well as hydrogen containing, single-phase multi-component refractory materials were synthesized. The technological processes for production of more useful hydrides ($TiH_2$ and $ZrH_2$) were worked out.

Since 1998, the systematic investigation of thermal-chemical processes in metal–hydrogen systems has been performed in our laboratory.

In formulating the present task, we have taken into account the specific properties of hydrogen that make the

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hydrogen-containing materials particularly sensitive to the impact of the electron beam.

## 2. Experimental

In the investigations, the linear high-current electron accelerator LAE-5 used.

The technical assignment of the special hermetic chamber for processing materials by the electron beam in vacuum and under pressure was worked out. The chamber allowed us to work at a maximum hydrogen pressure up to 3 atm. It was manufactured and installed in front of the LAE-5 in focused electron beam (1 atm=101 325 Pa). The collimated beam ensures even irradiation of all the sample. The experiments were performed under the following parameters of the accelerator: energy of electrons – 4 MeV, average current – 150 μA. The systems for measuring the temperature and heat effects during irradiation were elaborated. The 'S' type Pt/Pt–Rh thermocouple was used. In Fig. 1 the scheme of the experiment is presented.

A tablet ($d$=20 mm, $h$=5 mm) was pressed from metal powder and placed in the chamber on the special support. The chamber was evacuated. Irradiation of the sample in vacuum or in hydrogen atmosphere was carried out with different doses (up to 100 MRad) and dose powers (0.025–1 MRad/s).

Certification of the obtained materials was done using chemical, X-ray (diffractometer DRON-2) and differential thermal (derivatograph Q-1500) analyses.

In the experiments, the poly-disperse powders of Ti, Zr and Hf of grain size down to <100 μm and hydrogen of electrolytic purity were used.

## 3. Results and discussion

### *3.1. Thermal-radiation synthesis of hydrides of IV group metals*

The data have shown that on irradiation of IV group metals in the hydrogen atmosphere, the TRS of metal hydrides occurs. The data of X-ray and chemical analyses testified that hydrides $TiH_2$ and $ZrH_2$ of stoichiometry content, as well as of hafnium hydride of super-stoichiometry content, $HfH_{2.18}$ were obtained.

The measurement of temperature developed at thermal-radiation process in the investigated systems allowed us to establish a number of important regularities, and the mechanism of hydride formation in the accelerated electron beam.

In Fig. 2a–c, the thermograms of thermal-radiation processes at irradiation of Ti, Zr and Hf in hydrogen at the dose power of 0.7 MRad/s are presented. It is seen that in the beginning of irradiation, the temperatures smoothly increase up to 200–400°C with increase of dose up to 20–30 MRad, due to the sample heating caused by irradiation. It is known that on irradiation, a significant part of radiation energy transforms into thermal energy, causing noticeable heating of the material. In our experiments, the temperature developed at preliminary irradiation proved sufficient for initiation of the exothermic reaction $Me+H_2$ in all of the sample volume. On reaching 200–400°C, the temperature of the process jumps up to 880°C for Ti, 760°C for Zr, and 675°C for Hf. It is similar to a 'thermal explosion'. Actually, we registered the high temperature developed in the exothermic reaction initiated by the electron beam in all of the sample volume.

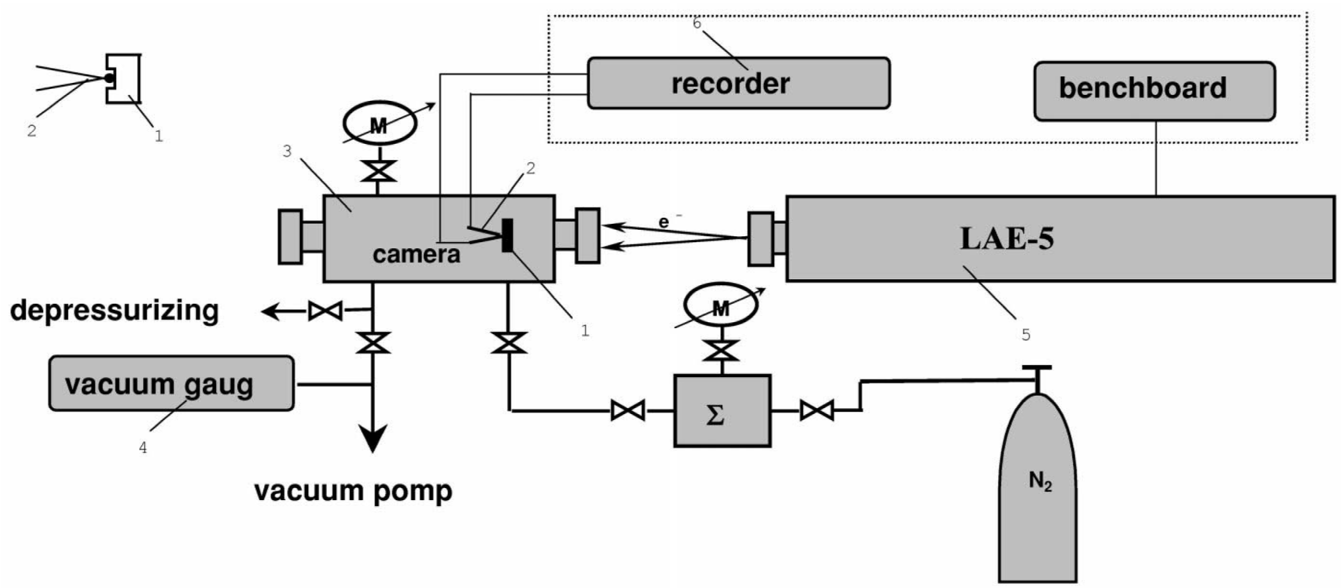

Fig. 1. The general scheme of the experiment. 1 – Sample, 2 – thermocouple, 3 – chamber, 4 – vacuum-gauge, 5 – accelerator of electrons, 6 – thermocouple data recorder.

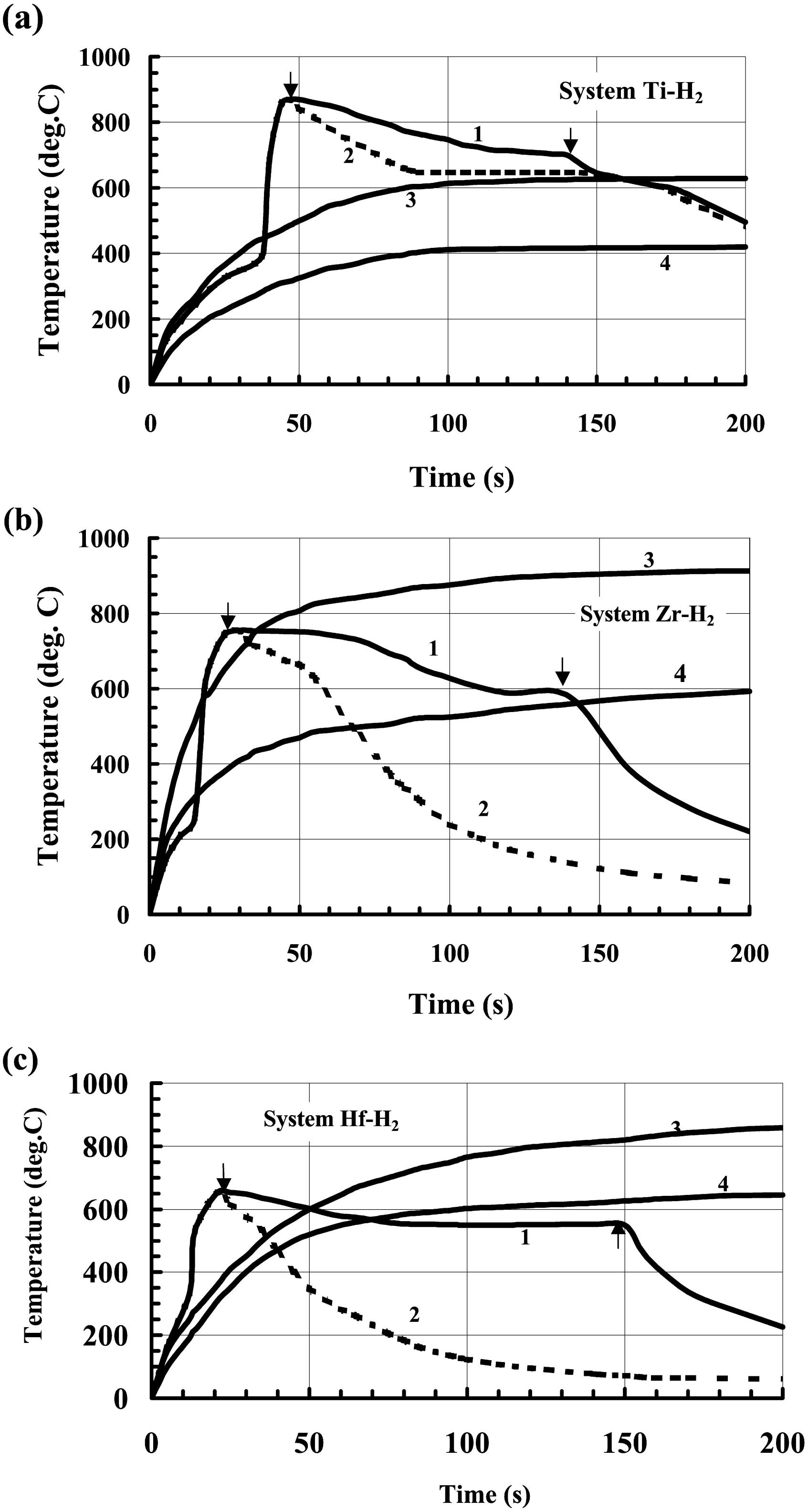

Fig. 2. Thermograms of thermal-radiation processes at irradiation of Ti (a), Zr (b) and Hf (c) in hydrogen: curve 1 – TRS; curve 2 – TRS with beam switched off (dashed lines); curve 3 – irradiation in vacuum; curve 4 – irradiation $MeH_2$ in hydrogen.

On completing the reaction, the temperature smoothly decreases (Fig. 2a–c, the continuous lines 1), even if the irradiation continues and the radiation dose increases. The electron beam was switched off on achieving radiation dose: for titanium – 98 MRad ($t=140$ c), for zirconium – 91 MRad ($t=130$ s) and for hafnium – 105 MRad ($t=150$ s). If the irradiation is stopped on reaching the maximum temperature, faster cooling of the samples occurs (Fig. 2, curves 2 – dashed lines). Then the temperature of the samples decreases down to the room temperature. On some of thermograms a plateau is observed. The cooling curves in general correspond to the known kinetics in the conditions of chemical equilibrium.

In Fig. 2a–c, the dependencies of temperature from the dose at irradiation with the dose power 0.7 MRad/s of IV group metals in vacuum (curves 3), and their hydrides in hydrogen (curves 4), are presented. These experiments can be considered as 'blank', since here any essential chemical interaction is excluded. But they allow one to measure the temperature, developed on the sample due to action of the beam. For temperature calibration in the hydrogen atmosphere, hydrides were used instead of metals, as they do not interact with hydrogen, i.e. they are inert under these conditions. It can be seen that in both cases the temperature smoothly increases with the increases of irradiation dose up to 100 MRad, practically stopping thereafter. Note that the temperature developed in hydrogen atmosphere is much lower than in vacuum.

Let us compare the thermal-radiation and the earlier studied SHS processes in the Me–H system. To realize SHS, for example, in Ti+H, local initiation of combustion in the thin layer of a titanium powder sample is enough. The instantaneous initiation is performed by heating up to 300°C with an electrical spiral. Then, due to layer-to-layer heat transfer, the combustion front propagates through the unheated substance at a rate of 1 cm/s.

In Fig. 3, the thermogram of the SHS process is presented. It is seen that the temperature increased to 730° sharply, then smooth cooling began. The SHS process of metal hydride formation is a two-stage one. In the first stage in the combustion front, a solid solution of hydrogen in titanium is formed. In the second stage, following the combustion stage, along with the cooling of the sample, after-hydrogenation takes place until stoichiometric $TiH_2$ is formed. The above asserts, that TRS and SHS processes (both of them can be easily initiated), are short, and proceed due to the exothermic reaction. The end products of both reactions are hydrides of at least stoichiometric content.

The mechanism of hydride formation in TRS mode is also a two-stage process: in the first, exothermic stage, a solid solution of $H_2$ in metal is formed; in the second, after-hydrogenation stage, hydrogen saturated hydride is formed. However, in SHS, the zone of the exothermic reaction propagates frontally, and in TRS, the reaction proceeds simultaneously in the entire volume of the sample, heated by the electron beam, analogous to the 'thermal explosion'.

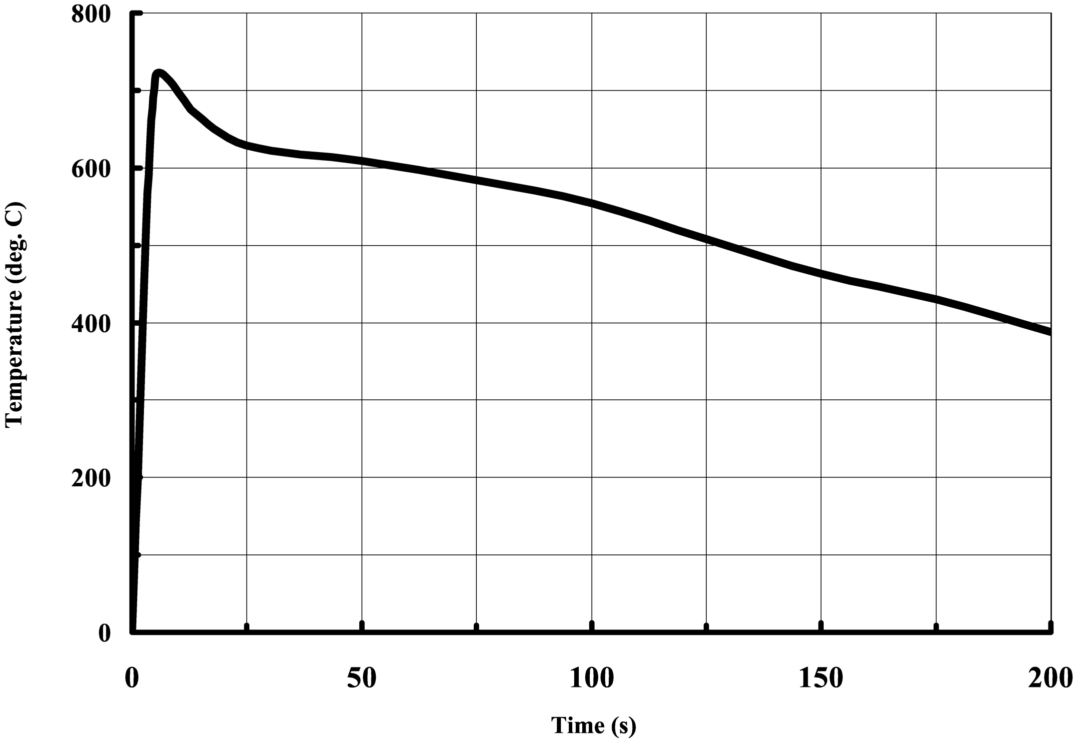

Fig. 3. Thermogram of the SHS process in the Ti–H system.

### *3.2. Some regularities of thermal radiation processes*

In the course of experiments, the dependencies of the temperature of TRS process, and of the hydrogen content in the end products from the dose and the dose power, were determined. The threshold of dose power for realization of TRS reaction has been determined.

In Table 1, the main characteristics of TRS processes are presented. It is seen, that in the Hf–H system, both the temperature of the exothermal reaction start, and the maximum temperature developed in the course of TRS reaction, are lower than those for the systems Ti–H and Zr–H. These temperature variations may be related to the atomic mass of the metal under irradiation. The TRS in these systems proceeds not only due to the exothermic reaction. The contribution of the irradiation to the inter-

Table 1
Characteristics of thermal-radiation synthesis and of cold synthesis in $Me^{IV}$-H systems

| Mode | Dose power (MRad/s)// dose (MRad) | Thermal-radiation synthesis | | | | | Cold synthesis | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $T_{begin}$ TRS (°C) | $T_{reac}$ (°C) | $H_2$, (wt. %) | Formula | Phase state parameters (Å) | Dose | $T_{heat.}$ in vacuum (°C) | $T_{begin}$, CS (°C) | $T_{reac.}$ (°C) | $H_2$, (wt. %) | Phase |
| *Titanium* | | | | | | | | | | | | |
| SHS | – | 300–500 | 700–900 | 4.01 | $TiH_2$ | FCC<br>$a=4.44$ | | | | | | |
| TRS | 0.1 | | | | | No reaction | | | | | | |
| | 0.2 | | | No reaction | | | 40 | 420 | 70 | 725 | 3.95 | $TiH_2$ |
| | 0.3 | | | No reaction | | | 40 | 480 | 70 | 750 | 4.01 | $TiH_2$ |
| | 0.4//40 | 295 | 640 | 4.08 | $TiH_2$ | $a=4.447$ | 40 | 540 | 60 | 760 | 4.08 | $TiH_2$ |
| | 0.5//45 | 305 | 660 | 3.97 | $TiH_2$ | $a=4.447$ | 40 | 540 | 40 | 750 | 4.00 | $TiH_2$ |
| | 0.7//30 | 357 | 695 | 4.03 | $TiH_2$ | $a=4.458$ | 50 | 640 | 80 | 730 | 3.99 | $TiH_2$ |
| | 0.8//30 | 360 | 710 | 3.97 | $TiH_2$ | $a=4.459$ | 60 | 545 | 80 | 550 | 4.17 | $TiH_2$ |
| | 0.9//30 | 375 | 735 | 4.01 | $TiH_2$ | $a=4.458$ | 60 | 570 | 80 | 545 | 4.16 | $TiH_2$ |
| | 1//25 | 420 | 820 | 4.06 | $TiH_2$ | $a=4.461$ | 60 | 555 | 80 | | Not realized | |
| *Zirconium* | | | | | | | | | | | | |
| SHS | – | 300–500 | 900 | 2.07 | $ZrH_2$ | FCT<br>$a=4.976$<br>$c=4.457$ | | | | | | |
| TRS* | 0.1 | | | | | No reaction | | | | | | |
| | 0.2 | 250 | 700 | 2.15 | $ZrH_2$ | FCT | 70 | 400 | 50 | 680 | 2.15 | $ZrH_2$ |
| | 0.3 | 250 | 700 | 1.96 | $ZrH_2$ | FCT | 47 | 650 | 25 | 720 | 2.08 | $ZrH_2$ |
| | 0.4 | 250 | 730 | 2.06 | $ZrH_2$ | FCT | 65 | 680 | 25 | 700 | 2.09 | $ZrH_2$ |
| | 0.5 | 250 | 730 | 2.07 | $ZrH_2$ | FCT | 60 | 720 | 25 | 680 | 2.1 | $ZrH_2$ |
| | 0.6 | 280 | 750 | 2.06 | $ZrH_2$ | FCT | 40 | 800 | 25 | 620 | 2.1 | $ZrH_2$ |
| | 0.8 | 280 | 800 | 1.99 | $ZrH_2$ | FCT | 60 | 910 | 30 | 650 | 2.05 | $ZrH_2$ |
| | 0.9 | 280 | 790 | 2.04 | $ZrH_2$ | FCT | 70 | 895 | 25 | 620 | 1.92 | $ZrH_2$ |
| | 1 | 280 | 790 | 2.11 | $ZrH_2$ | FCT | | | | | | |
| *Hafnium* | | | | | | | | | | | | |
| SHS | – | – | 840 | 1.05 | $HfH_2$ | FCT<br>$a=4.88$<br>$c=4.34$ | – | – | – | | | |
| TRS | 0.05 | No reaction | | | | | 35 | 150 | 60 | 428 | 1.3 | $HfH_{2.35}$ |
| | 0.1 | No reaction | | | | | 35 | 220 | 60 | 428 | 1.23 | $HfH_{2.22}$ |
| | 0.2 | No reaction | | | | | 50 | 355 | 60 | 500 | 1.22 | $HfH_{2.2}$ |
| | 0.3 | 220 | 590 | 1.15 | $HfH_{2.08}$ | FCT | 70 | 423 | 60 | 558 | 1.19 | $HfH_{2.15}$ |
| | 0.4 | 225 | 600 | 1.12 | $HfH_{2.02}$ | FCT | 70 | 490 | 60 | 513 | 1.18 | $HfH_{2.13}$ |
| | 0.5 | 220 | 615 | 1.21 | $HfH_{2.18}$ | FCT | 68 | 505 | 60 | 485 | 1.24 | $HfH_{2.24}$ |
| | 0.6 | 240 | 630 | 1.12 | $HfH_{2.18}$ | FCT | 84 | 707 | 60 | 603 | 1.22 | $HfH_{2.2}$ |
| | 0.7 | 255 | 675 | 1.15 | $HfH_{2.08}$ | FCT | 60 | 615 | 60 | 495 | 1.18 | $HfH_{2.13}$ |
| | 0.8 | 270 | 670 | 1.10 | $HfH_{2.06}$ | FCT | 100 | 915 | 60 | 625 | 1.22 | $HfH_{2.2}$ |
| | 0.9*** | 325 | 695 | 1.20 | $HfH_{2.17}$ | FCT | 100 | 840 | *** | 620 | 1.23 | $HfH_{2.22}$ |

*TRS for Zr was carried out at dose 100 MRad. **TRS for Hf was carried out at doses of 45–55 MRad. ***CS for $HfH_2$ was implemented at room temperature in 3 h after irradiation at dose power 0.9 MRad/s.

action of metal with hydrogen is essential. The below presented data may prove the particular influence of irradiation on the process of synthesis.

Earlier in the investigation of combustion of transition metals in hydrogen, it was found out, that in every group, the tendency of metals to interact with hydrogen in SHS mode is almost independent of the atomic number. The heat of titanium hydride formation ($\Delta H$) is equal to 31.1 Kcal/mol; that of zirconium hydride – 39.7 Kcal/mol, and of hafnium hydride – 9.73 Kcal/mol. At hydrogen pressure ($P_H$) 10 atm, the combustion temperature, $T_c$, for titanium is 700°C, for zirconium – 900°C; for hafnium – 800°C. Most likely, the activation energies of these reactions play an essential role in the interaction of these metals with hydrogen.

At thermal-radiation synthesis of the hydrides of IV group metals, the characters of metals interaction with hydrogen are similar (Table 1). It is worth noticing that, at hydride formation, the temperature of the TRS process is lower than that of the SHS process by nearly 100–200°C.

In the Ti–H system at dose powers of 0.1–0.3 MRad/s and in the Hf–H system at dose powers of 0.05–0.2 MRad/s, TRS reaction does not occur. From 0.4 MRad/s, on accumulating the dose 20–30 MRad, exothermic interaction of metal with hydrogen takes place, and hydrides of titanium and hafnium are formed. Note that, as a result of TRS in the Hf–H system, super-stoichiometric hafnium hydride, $HfH_{2.17}$ (H/Me>2) has been obtained for the first time.

In the Zr–H system TRS is realized at lower dose powers – beginning from 0.2 MRad/s (Table 1).

It should be noted that the temperature profiles of TRS processes at different dose powers are analogous to those in Fig. 2a–c.

In Fig. 4 are presented the dependencies of the temperature, developed in a sample during irradiation, and of the hydrogen content in the end product from the dose power of irradiation of the Zr–H system. Curve 1 characterizes the temperature of the exothermal reaction start; curve 2 – the temperature, developed at exothermal reaction; and curve 3 – the hydrogen content in the end products. It is seen that the main characteristics of TRS (temperature and hydrogen content) do not depend on irradiation dose power. Identical dependencies were observed in the systems Ti–H and Hf–H. Apparently in exothermic interaction of group IV metals with hydrogen, maximally saturated hydrides are formed. Therefore, the hydrogen content does not depend on such an important parameter as the hydrogen pressure (at SHS), and the irradiation dose power (at TRS). With regard to the independence from the dose power of both the temperature of reaction start and the maximum temperature of reaction, we can say the following. During irradiation, as soon as the temperature necessary for starting the exothermic reaction is reached, the sharp temperature jump occurs. This temperature depends on the heat effects of hydride formation.

### 3.3. *'Cold synthesis' of hydrides*

The investigation of the influence of the preliminary irradiation of metals on the TRS process revealed a new, unexpected phenomenon. After irradiation of titanium in vacuum by a dose power of 0.4 MRad/s, as soon as the sample temperature reaches 500–550°C (at dose 30–40

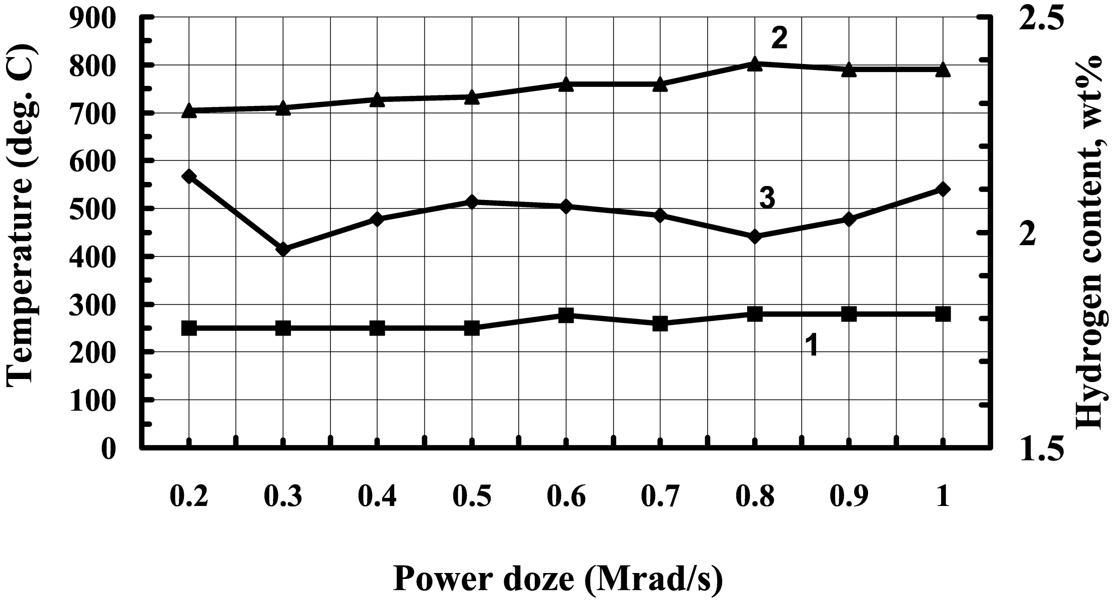

Fig. 4. The dependencies of the temperatures (or reaction start, 1, and of developed in reaction, 2) and of the hydrogen content, 3, in the end product from the dose power of irradiation of the Zr–H system.

MRad), the electron beam was turned off. After cooling the sample down to 60°C, the chamber was filled with hydrogen. In 10–30 s, a sharp jump of temperature was registered by the thermocouple up to 750°C (Fig. 5). Then a reaction analogous to the TRS process followed. A result of this reaction was titanium hydride, $TiH_2$. This phenomenon was not observed, if the preliminary irradiation of titanium in vacuum was performed by dose powers 0.1 MRad/s (total dose 40 MRad) and 1.0 MRad/s (total dose 60 MRad). In Table 1, the characteristics of the discovered reaction are presented.

For the system Zr–H, an identical phenomenon was observed when irradiation in vacuum was performed over a wider range of dose power (0.05–1 MRad/s). In this system, the exothermal reaction after preliminary irradiation began at room temperature (Table 1).

In the Hf–H system, after irradiation in vacuum even by very low dose power (as low as 0.025 MRad/s), the filling of the chamber with hydrogen at room temperature brought to effective exothermic interaction of hafnium and hydrogen. If the dose power of irradiation in vacuum is increased from 0.025 up to 0.9 MRad/s, the temperature of sample heating grows from 150 up to 840°C. The temperature developed in the sample after hydrogen filling, i.e. the temperature of reaction increases too.

One should note that in each case, after irradiation in vacuum, the sample was cooled down. The data in Table 1 indicate higher dose the sample has taken in vacuum higher the end temperature of reaction after hydrogen filling.

The observed reaction was named ‘cold synthesis’ (CS). Currently, we do not have any definite explanation for the nature of this phenomenon.

Apparently, at irradiation of metal in vacuum by electron beam of indicated dose power, energy is accumulated due to crystal lattice tension and/or deformation and structural defects.

The ‘deformed’ metal particles are, obviously, chemically more active and, appearing in hydrogen environment, they interact with it at low temperature (20–60°C) without any external intervention (without reaction initiation as in SHS, or electron beam irradiation as in TRS in hydrogen, or baking as in traditional furnace technology). Worth noting is, that at room temperature, this ‘non-equilibrium’ state of metal crystal lattice is held for rather a long time: in titanium – for 15 min, in zirconium – for 3 h, in hafnium – for 4 h.

It was found that the ability to retain the radiation damage (the damage lifetime) is mainly conditioned either by the dose power of the preliminary irradiation, or by the total dose, or by the temperature developed during irradiation in vacuum. For instance, at irradiation of Hf in vacuum with dose power 0.2 MRad/s up to dose 70 MRad ($T_{heat}$=355°C), CS takes place at 60°C with the maximum reaction temperature 600°C. However, the reaction does not take place in 3 h after irradiation. At irradiation by 0.3 MRad/s up to dose 42 MRad ($T_{heat}$=380°C), CS takes place even in 3 h after irradiation with the $T_{reac}$=655°C.

Table 2 presents the data concerning the CS of hafnium hydride at irradiation of hafnium with dose power 0.5 MRad/s.

At sequential irradiation of the same hafnium sample

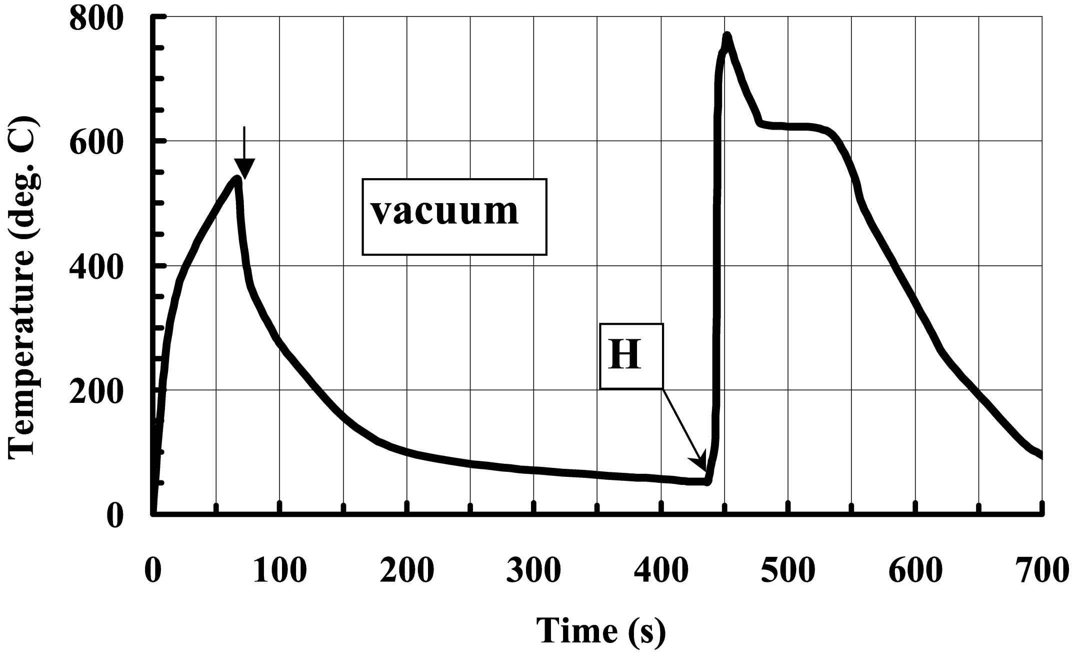

Fig. 5. The thermogram of hafnium hydride ‘cold synthesis’ (dose power of preliminary radiation 0.7 MRad/s).

Table 2
Characteristics of hafnium hydride cold synthesis at irradiation at dose power 0.5 MRad/s

| Dose (MRad) | Temperature in vacuum (°C) | Time of hydrogen feeling | Temperature of exo-reaction (°C) | $H_2$ content (mass %) | Formula, phase |
|---|---|---|---|---|---|
| 68 | 505 | 15 min (100°C) | 485 | 1.24 | $HfH_{2.24}$, FCT |
| 75 | 650 | 30 min | 620 | 1.13 | $HfH_{2.04}$, FCT |
| 75 | 680 | 1 h | 550 | 1.13 | $HfH_{2.04}$, FCT |
| 75 | 590 | 2 h | 590 | 1.24 | $HfH_{2.24}$, FCT |
| 100 | 630 | 3 h | 600 | 1. 28 | $HfH_{2.31,}$ FCT |
| 75 | 655 | 4 h |  | No reaction |  |
| 100 | 685 | 4 h | 620 | 1.10 | $HfH_{1.99}$, FCT |

with various dose powers (0.1, 0.2, 0.3 and 0.4 MRad/s, total dose 175 MRad), the CS took place at 60°C. The temperature of reaction in this case reached 600°C. The hydrogen content in the received hafnium hydride was 1.34 wt. % and confirmed the hydride content as $HfH_{2.42}$.

Thus, hafnium hydride of super-stoichiometric content ($HfH_{2.0-2.42}$), rich in hydrogen, has been produced for the first time in 'cold synthesis' reaction.

## 4. Conclusions

As a result of the performed investigations:

- The thermal-radiation syntheses of titanium, zirconium and hafnium hydrides were performed.
- The main features of TRS were defined, and the mechanisms of hydride formation in the accelerated electron beam were revealed.
- The new phenomenon of 'cold synthesis' was discovered, beginning at room temperature due to exothermic interaction of preliminary irradiated metal with hydrogen.
- The super-stoichiometric hafnium hydride, $HfH_{2.42}$ was synthesized for the first time.

## Acknowledgements

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