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INFLUENCE OF PLASMA TREATMENT ON EROSION HARACTERISTICS AND STRUCTURE OF REVERSIBLE HYDROGEN GETTERS

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Introduction

In the paper [1] an analysis of bombardment of cathodes of gas discharge devices with high-energy plasma particles was carried out. Those cathodes were manufactured on the base of reversible low-pressure getters of hydrogen. It was shown that bombardment of the cathodes by particles stimulated the hydrogen desorption from the surface. In this case the quantity of stripped hydrogen, resulted due to both a thermal decomposition of hydride phases and an ion stimulated desorption, was proportional to the flux of incident particles. In that way the internal feedback between energy flux to the electrode surface and intensity of gassing was provided. In other words, a protective gas-dynamic target was formed close to the surface of the reversible sorbents of hydrogen. This target essentially decreased the sputtering yield of a material. Such systems could be used for high hydrogen concentration buildup in the plasma facing materials. As it was shown in work [2], there is a possibility of decreasing the carbon erosion due to the hydrogen shielding. This shielding effect arises as the result of accumulation of high dose of hydrogen at near surface layers of material during special regime of high-flux hydrogen ion bombardment. But it is not clear whether such mechanism can be realized when using intermetallides and metals. So, it was of a great interest to consider the possible mechanisms of high hydrogen content buildup in such systems.

In this work the results of investigations of plasma processing of $Zr_{55}V_{40}Fe_5$ alloy, modified by 3% of B_2O_3 and preliminary saturated by hydrogen, are discussed.

Experimental results and discussion

The specified above material with additional binding dopant (copper powder in quantity 40 % from total weight of a composite) was pressed under the room temperature. The pressure of pressing and duration of composite exposure under the pressure were about 0.3 GPa and 5 minutes accordingly. The sample of pressed composite had a form of cylinder of 20 mm in diameter and 5 mm in thickness Total content of hydrogen accumulated in the sample was 2,5 dm^3.

Pulsed nitrogen plasma streams processing was used for modeling the material behavior under high power heat load. The duration of plasma stream generation was (3-5) μs. The ion energy was 2 keV, plasma density ~ $2x10^{14}$ cm^{-3}, specific power of plasma stream up to 10 MW/cm^2, and plasma energy density varied in the range of (10-30) J/cm^2.

It was shown that treatment by pulsed plasma streams with specific power value, similar to one expected for disruption process in tokamak-reactor, led to melting of surface layer and significant weight losses of processed sample (up to 0.53 mg/cm^2 per pulse). Electron microscope images of the sample surface, obtained before and after plasma irradiation, are presented in Fig. 1.

After bombardment of samples with 7 pulses the phase analysis of both sample surfaces, irradiated by plasma and non-irradiated, was carried out. The analysis was carried out by difractometer DRON-2 with use the Cu-K_α radiation. The registration of diffraction picture was performed in an interval of Bragg angles 2θ = 20-150° in a mode of continuous record on a plotter tape.

Obtained difractograms were scanned and translated into a digital form under simultaneous corrective actions for elimination of a background and drift of a zero line. As a result the difractogram profiles were obtained as pairs of values "Bragg angle 2θ° (with step in the reflex region 0,1°) - intensity I_e (mm)".

Analysis of difractograms was carried out with use of the specialized software with realizing the algorithm of minimization of the divergence factor R [3], which calculated by formula:

$$R = \sqrt{\frac{\sum \left(I_e - I_c\right)^2}{\sum I_e^{\,2}}}, \qquad (1)$$

Here I_e, I_c - experimentally observed and calculated intensities accordingly.

Calculated intensities I_c were approximated by expression [4]:

$$I_c = I_c^0 \exp\left[-\left(\frac{2\theta - 2\theta_0}{\Delta}\right)^2\right], \qquad (2)$$

Where I_c^0 and $2\theta_0$ - calculated intensity and Bragg angle of maximum of peaks accordingly, Δ - halfwidth of a peak measured at the peak height where its intensity is by e times lower than maximum one.

The calculated intensity of a maximum of j^{th} peak of i^{th} phase was determined as:

$$\left(I_c^0\right)_{ij} = W_i I_{ij}^0, \qquad (3)$$

Here I_{ij}^0 - theoretical intensity of peak maximum, W_i - weight factor that is proportional to the mass portion of the i^{th} phase (in the case of absence of the intensities distortions).

The difractograms analysis was carried out on the base of known models of material structure (definition of the theoretical intensities I_{ij}^0) with the help of the software package "Crystal Structure Determination", developed in the Lvov State University (the algorithms of calculations are given in work [5]). Bragg angles were obtained when taking into account the symmetry and periods of phases lattice [3,4].

To provide the minimum value of factor R at the process of calculations there was carried out the adjustment of Δ values (Δ was accepted identical to all present phases) as well as the weight factors of intensity W_i and periods of a lattice for each phase. Adjustment was carried out with use of a simplex - method [6]. The step of a computational grid at calculations of the factor R from the expression (1) was $0{,}01^o$.

For difractograms assignment of indice the earlier received data on the phase-structure and the hydrogen sorption characteristics of hydride forming materials of similar structure were used [7].

Characteristic of phases for both irradiated and non-irradiated sides of a material sample, obtained as a result of difractograms assignment of indice (Fig. 2,3) are given in the Table 1. High values of the divergence factors are caused, apparently, by distortions of lines intensity due to texturing, which, as is known [2], can achieve of dozens of times. At that, as it is possible to see from the Fig. 2,3, the intensity of copper lines considerably surpass the intensity of the lines of other phases, that complicates the assignment of indice additionally. Nevertheless, on the base of the completed analysis it is possible to conclude the following.

The irradiation of a sample leads to appreciable (on the level of 15 %) broadening of lines in difractogram and simultaneous reduction of absolute intensities of the lines. It testifies that sample side, irradiated by pulsed plasma stream, has fine grained, and probably partially amorphous structure. According to the data of the electron microscopy, the pulsed plasma stream treatment leads to melting the sample surface under a condition of the barbotage of desorbed hydrogen bubbles through the melt and subsequent its high speed cooling after the plasma pulse action.

The periods of the copper lattice for both non-irradiated and irradiated sides of the sample exceed the appropriate reference data (0,3615 nm). Moreover, for irradiated side the period is even higher. Probably, it is caused both by an introduction of nitrogen from incident plasma stream into a crystal lattice of copper and by its subsequent diffusion to the depth of the sample.

The lattice periods of hydride forming intermetallic phases, namely Laves phase λ_2–$Zr(V,Fe)_2H_X$ and phase $Zr_3(V,Fe)_3OH_X$, are higher than periods of initial intermetallides (0,7396 nm and 1,2156 nm respectively), but are lower than lattice periods of completely saturated hydrides (0,7886 nm and 1,2656 nm). For irradiated side of sample the periods of a lattice of these phases were appreciably decreased in comparison with initial state. Comparison of the obtained results (see Table) with the literature [5] allows to estimate degree of saturation by hydrogen of a Laves phase as 72 % before irradiation and 64 % after irradiation. The data for the η–phase are 49%

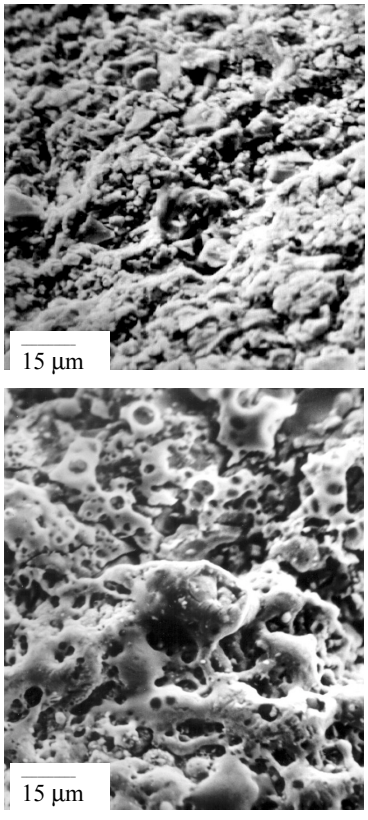

Fig. 1. Images of the sample surface before and after plasma treatment

Table. Phase content of the sample

Characteristic			***Value***	
			Nonirradiated side	Irradiated side
Divergence factor *R*			0,458	0,528
Average halfwidth of peak, Δ, ^{o}			0,30	0,34
Phase (symmetry group)	Cu (F m 3m)	Lattice period,nm	a=0,36187	a=0,36215
		W_i	0,1256	0,08197
		$\frac{W_i}{\sum W_i}$, %	76,21	64,21
	λ_2–$Zr(V,Fe)_2H_X$ (F d 3 m)	Lattice period,nm	a=0,77496	a=0,77110
		W_i	0,02502	0,02186
		$\frac{W_i}{\sum W_i}$, %	15,18	17,12
	$ZrCu_5(H_X)$ (F 4 3 m)	Lattice period,nm	–	a=0,70549
		W_i	–	0,01207
		$\frac{W_i}{\sum W_i}$, %	–	9,95
	ε–ZrH_X (I 4)	Lattice period,nm	a=0,3621 c=0,4469	a=0,3626 c=0,4552
		W_i	0,01147	0,00546
		$\frac{W_i}{\sum W_i}$, %	6,96	4,28
	η–$Zr_3(V,Fe)_3OH_X$ (F d 3 m)	Lattice period,nm	a=1,23991	a=1,23152
		W_i	0,007534	0,00630
		$\frac{W_i}{\sum W_i}$, %	4,57	4,93

and 30 % respectively. It testifies that intensive desorption of a hydrogen from these hydride phases takes place during bombardment. Besides, desorption of hydrogen from zirconium hydride probably takes place also. During a bombardment the weight factors of the intensity of the Laves phase and the η-phase (normalized to the sum of the weight factors of all phases) vary unsignificantly. At the same time similar parameter for a copper decreases as much as more than 10 %, and for a zirconium hydride it decrease by 1,5 times. At that a phase of $ZrCu_5$ intermetallide with a little bit increased period of a lattice in comparison with the reference data (0,687 nm) appears in the irradiated sample. It can be caused by introduction of hydrogen or nitrogen atoms into material. The most probable explanation of this fact is the interaction of zirconium hydride with the melt of copper during a pulse of plasma processing. The melting temperature of copper is much lower in comparison with other components of a composite. In result the $ZrCu_5$ intermetallide of system Zr – Cu, which is the most enriched by copper, is formed during melt crystallization.

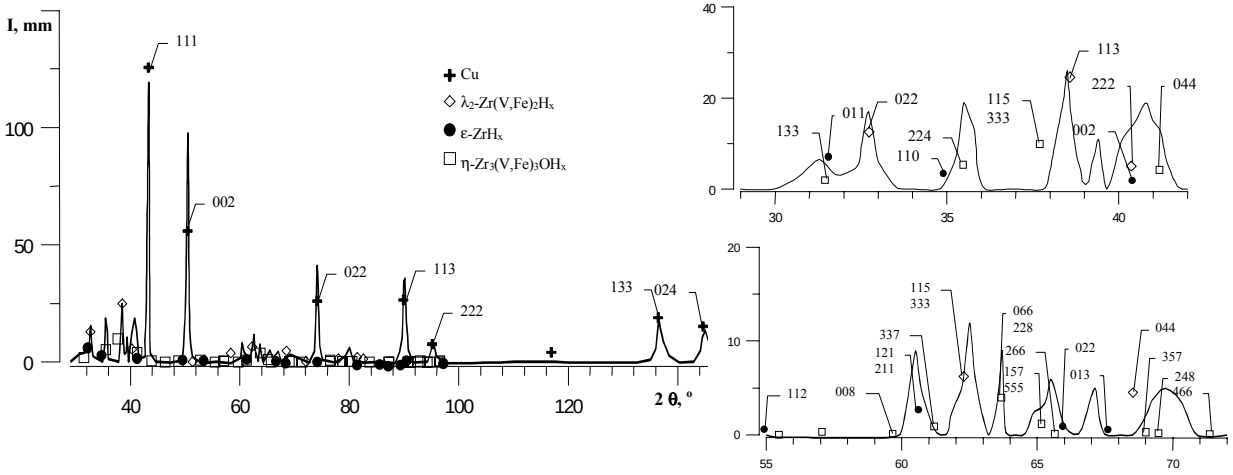

Fig. 2 The difractogram of the non-treated sample and its fragments

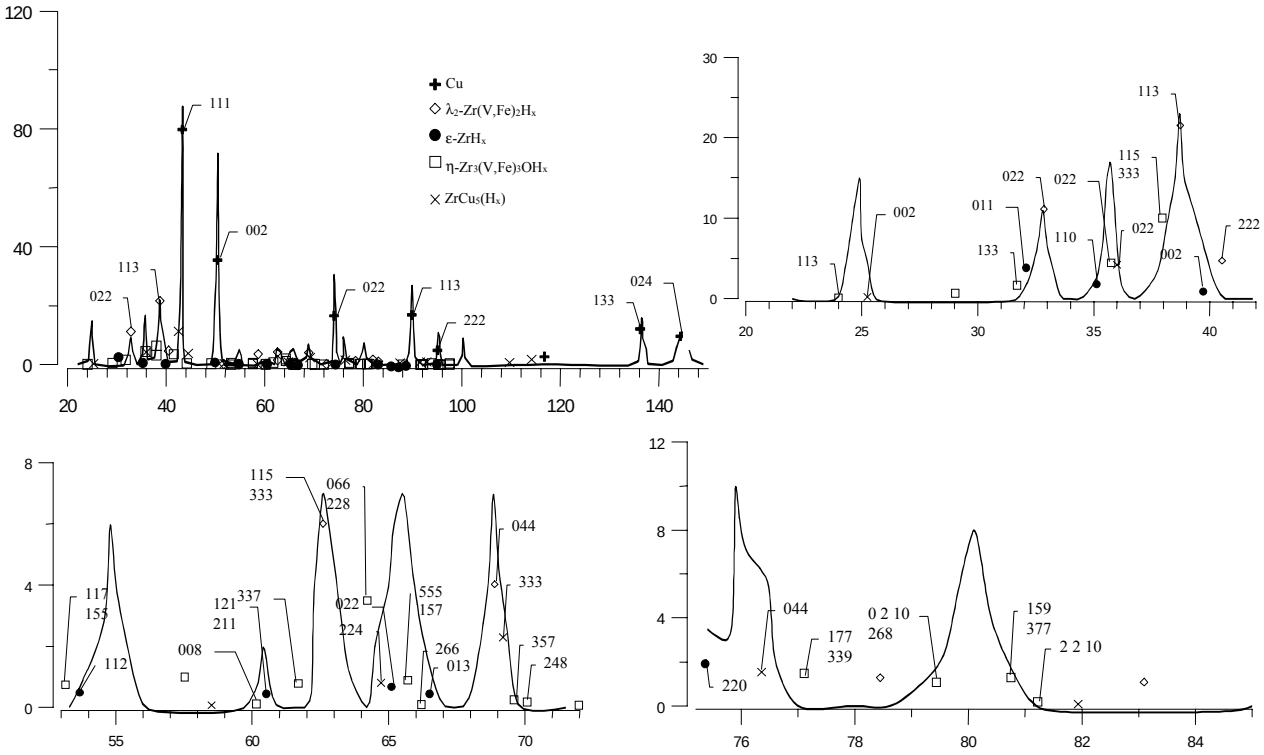

Fig. 3 The difractogram of the treated sample and its fragments

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