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Effects of Cold Deformation and Electron Irradiations on Deuterium Desorption Temperature Range from Zr – 1 %Nb Alloy

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The explored warm-up ranges of desorption ion-implanted deuterium from Zr – 1 %Nb alloy, with different structure: after crystallization from melt, plastic deformation and irradiations electron. Cool deformation under 300 K Zr – 1 %Nb alloy with degree $\varepsilon = 3.9$ has allowed to reach the nanostructure conditions in alloy with average size crystallite $d = 61$ nm, high three-dimensional concentration of the borders (~ 3,4 %) and significant level microstrain that in spectrum of thermodesorption ion-implanted deuterium was shown as additional low temperature area thermodesorption deuterium within the range of the temperature 770-1000 K. Irradiation electron to energy 10 MeV by fluence ~ 6×10^{17} cm^{-2} has brought about active development of the revocable processes in nanostructure alloy: reduction of the average grain size $d \approx 58$ nm, increase to concentrations of the borders (~ 8,8 %) and appearance additional peaks in spectrum of thermodesorption deuterium with the temperature 700 and 800 K and, accordingly, increase the warm-up range desorption deuterium toward reduction of the temperature.

Keywords: Zr – 1 %Nb alloy, Cold deformation, Electron irradiations, Nanostructure, TDS, TEM.

PACS numbers: 62.25 + g; 61.80.Fe; 61.82.Rx

1. INTRODUCTION

Zirconium alloys possess the unique combination nucleus-physical, mechanical and corrosive characteristic such as very low absorption neutron, high corrosive stability, high toughness and high plasticity. The Combination these characteristic conditions all increasing use zirconium and its alloy in creation of reactor. Preliminary introduction distributions in crystalline lattice reduces efficiency of radiation influences upon metals and alloys [1-3]. Introduces that one of the main trends in decision of the problem of increasing radiation stability is a making the firm fault structures. The most wide-spread way of the entering defect in crystalline lattice of the metal is a plastic deformation. Intensive plastic deformation under low homologous temperature ($T < 0.2\ T_m$, where T_m – temperature of the melting) allows to form the broad spectrum of the distorted structures. Scientific and practical interest presents the study penetrating irradiations on nanostructure condition reactor material [4, 5]. In process of the usages zirconium products are subjected to introduction of hydrogen that brings about reduction plasticity and stability to the cracks alloy [5]. For reduction of the influence of the hydrogen on characteristic zirconium alloy follows to consider such approaches, as creation covering or structured conditions of the metal, which delay the penetration of the hydrogen inside metal or stimulates its desorption from metal. In persisting work are presented results of the study warm-up interval desorption ion-implanted deuterium from Zr-1%Nb alloy in different structured conditions: after crystallization from melt, plastic deformation and electron irradiations.

2. THE METHODICAL ASPECTS

Warm-up ranges of desorption ion implanted deuterium from sample of the Zr – 1 %Nb alloy passed different changing a structure processing were researched by method thermodesorption spectroscopy on installation "SKIF" [6]. They were researched sample of the Zr – 1 %Nb alloy: (a) in cast condition (source); (b) passed plastic deformation under room temperature on $\varepsilon = 3.9$; (c) passed plastic deformation under room temperature on $\varepsilon = 3.9$ and irradiation electron to energy 10 MeV by fluence ~ 6×10^{17} cm^{-2}.

The sample by size $10 \times 5 \times 0.4$ mm^3 bore up on heaters from tantalum foil by size $40 \times 5 \times 0.1$ mm^3. The temperature was measured by tungsten-rhenium thermocouple WRe 5/20, attached to sample. For the reason reduction of the influence of the background hydrogen, available in sample and camera of the dartboards, in experiment was used isotope of the hydrogen – a deuterium. After the specimen was exposed to a prescribed dose, the beam was cut off, and the temperature of liquid nitrogen-cooled specimen was lowered to ~ 80 K; then the specimen heating was started, in the course of which the specimen temperature showed a rise up to ~ 1600 K at an average rate of ~ 3.5 K/s by an approximately linear law of time. The release of deuterium in the measurement chamber was measured with a mass spectrometer set to a mass $m = 4$ amu (D_2^+).

The structure sample researched by means of electronic microscope EMV-100BR at accelerating voltage 100 kV

3. THE RESULTS OF THE STUDY AND THEIR ANALYSIS

On Fig. 1 are brought typical spectrums thermodesorption of deuterium, implanted in source sample of

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the Zr – 1 %Nb alloy. The spectrums thermodesorption of deuterium display the structural conditions in him and their changes to dependencies from dose implanted deuterium. Under low dose implanted deuterium is formed phase condition of the hard solution deuterium in alloy zirconium that in spectrum thermodesorption reveals itself in the manner of two peaks with the temperature of the maximum ~ 1080 K and ~ 1400 K (refer to Fig. 1 curve a).

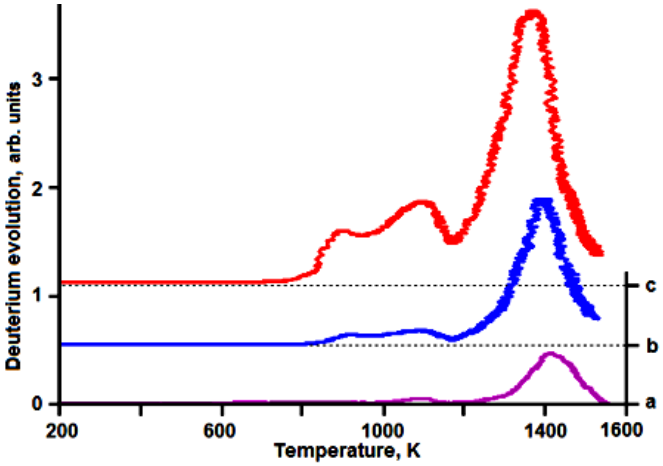

Fig. 1 – Thermal desorption spectra of deuterium implanted in Zr – 1 %Nb alloy doses: (a) ~ 1×10^{16} D/cm^2; (b) ~ 6×10^{16} D/cm^2; (c) ~ 2.5×10^{17} D/cm^2

Such type of the spectrum is conditioned presence in zirconium of the phase transition α-Zr $\Rightarrow$ β-Zr. The peak with the temperature of the maximum ~ 1080 K is conditioned by decay of the phase condition of the hard solution deuterium in α-Zr. Nearly simultaneously, with beginning of the decay this phase condition phase transition Zr $\Rightarrow$ β-Zr begins in zirconium, which in spectrum reveals itself in reduction of the velocities desorption of deuterium and forming the phase state of the hard solution deuterium in β-Zr. At the temperature ~ 1400 K occurs the decay of the phase condition of the hard solution deuterium in β-Zr. Increase the dose implanted deuterium stimulates formation zirconium hydride, about presence which witnesses the peak in spectrum thermodesorption deuterium with the temperature of the maximum ~ 850 K (refer to Fig. 1 curves b and c). For the further studies sample, passed the process changing a structures chose testing dose ~ 1.3×10^{17} D/cm^2 [7, 8].

Cool deforming the Zr – 1 %Nb alloy on greater degree ($\varepsilon = 3.9$) has allowed to reach the nanostructure state in alloy with average grain size $d = 61$ nm. For such nanostructure typical high concentration of the borders (~ 3,4 %) and significant level of microstresses (refer to Fig. 2b). The high degree of imperfectness and spottinesses of the sharing the internal voltages shows the change the warm-up range of deuterium desorption that in spectrum of ion-implanted deuterium thermodesorption reveal itself as additional low temperature area of deuterium desorption within the range of the temperature 770-1000 K (refer to Fig. 3, curve b). Herewith, deuterium desorption begins from room temperature. The irradiation electron to energy 10 MeV by fluence ~ 6×10^{17} cm^{-2} brings about active development of the revocable processes in nanostructure alloy. Distribution density falls into grains. The most intensive displacement of distribution occurs in top of the ragged borders and border-line zone – α places to maximum concentration of the voltages. Microsliding and crawling across of distributions is caused interaction them with flow point defect of radiation origins. As a result occurs shaping new polygonal borders (refer to Fig. 2c).

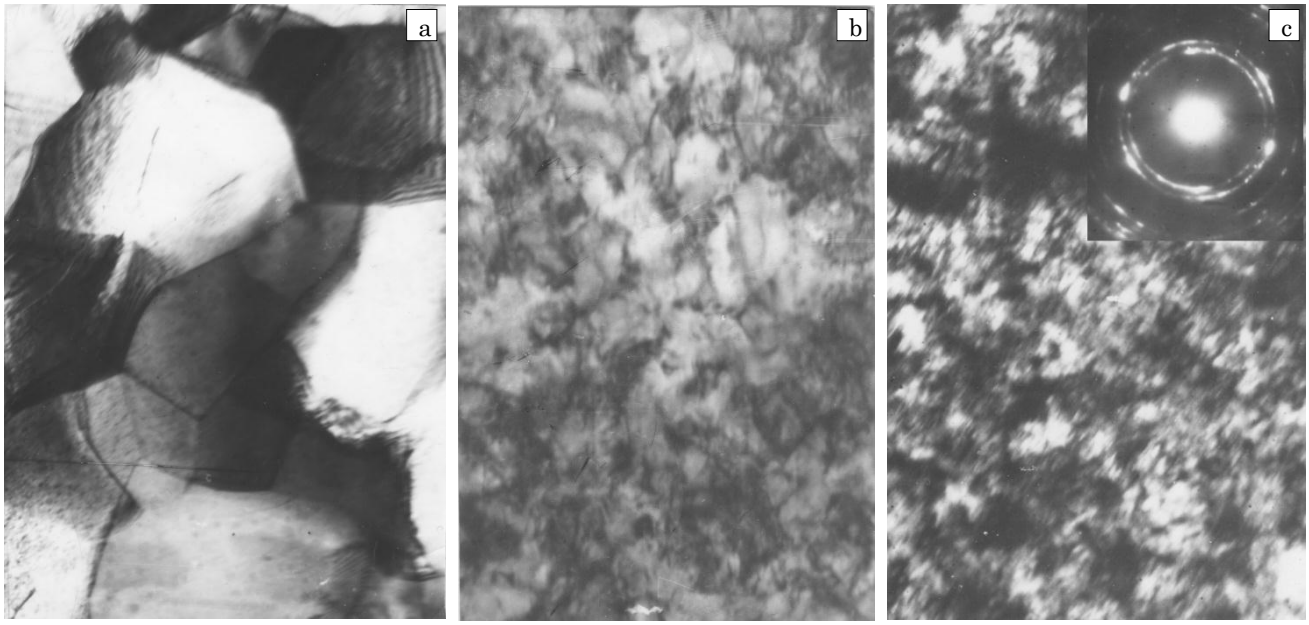

Fig. 2 – Structure Zr – 1 %Nb alloy: (a) source (× 80000); (b) deformed by cool rolling RT under on $\varepsilon = 96$ % (× 200000); (c) deformed by cool rolling RT under on $\varepsilon = 3.9$ and irradiated by electron beam fluence ~ 6×10^{17} cm^{-2} (× 200000)

The transformation of nanostructure is reduced to reduction of the average grain size (d = 58 nm) and increase to concentrations of the borders (~ 8,8 %). This in thermodesorption spectrum reveals itself in increase the warm-up range of deuterium desorption toward reduction of the temperature and appearance additional peak with the temperature ~ 700 and ~ 800 K (refer to Fig. 3, curve c), being indicative of structured changes to Zr – 1 %Nb alloy.

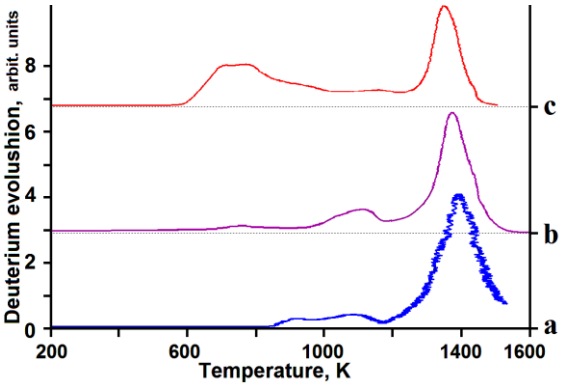

Fig. 3 – Thermal desorption spectra of deuterium (dose ~ 1.3×10^{17} D/cm^2) implanted in Zr – 1 %Nb alloy: (a) – source; (b) – deformed by cool rolling RT under on $\varepsilon = 3.9$; (c) – deformed by cool rolling RT under on $\varepsilon = 3.9$ and irradiated by electron beam fluence ~ 6×10^{17} cm^{-2}

We shall note that for austenitic stainless become Cr18Ni10T, passed get cold deformation, is discovered shift of the warm-up desorption area ion-implanted deuterium on 150-170 K in area of the more high temperature [9]. Herewith range temperature deuterium desorption from Cr18Ni10T and Zr – 1 %Nb, passed get cold deformation, very close on warm-up scale.

4. CONCLUSIONS

Cold deformation of Zr – 1 %Nb alloy on the big degrees has allowed to reach nanostructure state and has caused high degree of deficiency, heterogeneity of distribution of internal pressure. It has led to expansion of a temperature range deuterium desorption.

The electron irradiation Zr – 1 %Nb alloy which has preliminary passed cold deformation leads to active development of returnable processes in nanostructure an alloy: to reduction of the average size of grains, increase in concentration of borders and occurrence of additional peaks in a spectrum deuterium desorption with temperatures ~ 700 and ~ 800 K and, accordingly, to increase in a temperature range deuterium desorption in a direction of fall of temperature.

Possibly, intensity diffusions deuterium, caused level and in size of a gradient of internal pressure, supervises an exit deuterium on internal drains most powerful of which are borders. The increase in factor of ascending diffusion in the deformed and irradiated samples in connection with the increased concentration of borders facilitates moving introduced deuterium to external interfaces.

For reduction of influence of hydrogen by properties zirconium alloys it is necessary to consider the approaches providing creation submicrocrystalline grain of coverings or structural conditions of metal which stimulate decrease in its temperature desorption from metal.

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