THE INVESTIGATION OF HYDRIDES DEPTH DISTRIBUTION IN ZIRCONIUM ALLOY ZR-1NB AFTER HYDROGENATION AT GAS ATMOSPHERE

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Zirconium and its alloys are widely used in nuclear power. They are subject to corrosion and hydrogenation during operation in the nuclear reactor core. Hydrogen penetration occurs in the zirconium fuel cladding mainly from the outside and as a result nonuniform hydrides distribution is observed after exploitation. Preparation of samples with the same hydrides distribution like after exploitation is required for mechanical testing of fuel cladding tubes. For this, zirconium alloy Zr-1Nb samples with the dimensions of 20×20×0.6 mm were prepared and grinded to remove the oxide film. Hydrogenation was carried out on the Gas Reaction Controller equipment at temperature 320 °C and pressure 2 atm. for 6 hours to a concentration of 2500 ppm. The analysis of hydrogen distribution in the prepared samples was carried out by using glow discharge optical emission spectrometer Profiler 2. Microhardness was measured with a Vickers hardness tester KB 30S. The results of investigation are presented on the Figure 1.

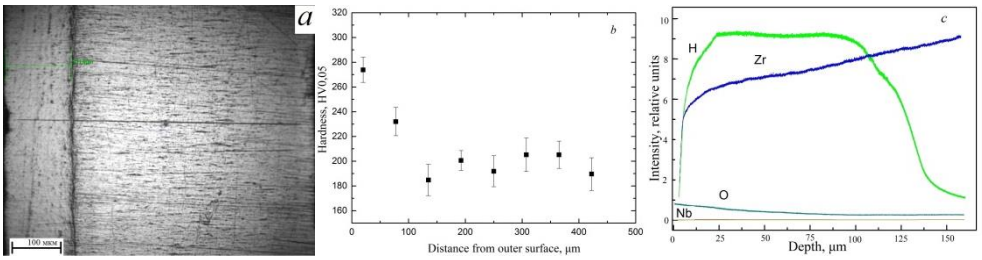

Figure 1. Image of transverse section of the sample of zirconium alloy E110 after hydrogenation (a), the results of microhardness transverse section (b), and the profile of the distribution of hydrogen (c)

Figure 1a shows an image of transverse section of zirconium alloy after hydrogenation. It is clear that on the outer surface of the samples there is a hydride layer with a thickness of ~130 microns. Figure 1b shows the results of microhardness measurements on the transverse section of the sample. Hydride layer has a greater hardness in comparison with the zirconium matrix. However, the hydride layer has a hardness distribution gradient. This effect is due to the fact that during the hydrogenation at low temperature and high pressure the low diffusion rate is provided at high hydrogen permeation rate. This leads to the hydrides formation directly after the penetration of hydrogen near the surface of the material. Subsequent penetration of the hydrogen occurs through the pre-formed hydride, and it leads to reducing of hydrogen amounts with increasing depth which is confirmed by the analysis of the distribution of hydrogen by glow discharge plasma spectrometry (Figure 1c).

Metallographic analysis and study of the hardness distribution of zirconium alloy hydrogenated to a concentration of 2500 ppm showed that the sample have the nonuniform distribution of the hydrides. During the hydrogenation at 320 ° C the hydride layer with the thickness of 130 micrometers is formed in the samples. At the same time the hydride layer has a hydrogen distribution gradient which is connected with the hydrogenation at low temperature and high pressure hydrogen when hydrides are formed directly at hydrogenation. Hydrogen distribution gradient by depth is confirmed by glow discharge plasma spectrometry.

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DETERMINATION OF THE NICKEL COATING ON THE Zr1%Nb ALLOY THICKNESS BY X-RAY METHOD

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Physical, chemical and mechanical properties of metals and alloys depend on the penetration and accumulation of hydrogen. This is acute for zirconium and titanium alloys, which are widely used in the nuclear industry [1]. For various research special preparation of samples is required with respect to hydrogen concentration and volume distribution of the material. With hydrogenation of alloys it is important to consider the possibility of oxide film formation on the sample surface which prevents penetration of hydrogen. Nickel coating on the surface increases the rate of hydrogen sorption. Furthermore, nickel is oxidized worse than titanium and zirconium, this property contributes to hydrogen absorption. Therefore, it is important to take into account the thickness of the nickel layer and its adhesive behavior.

During the work flat samples of Zr1%Nb alloy were prepared. Samples were subjected to mechanical polishing to remove surface dirt. Nickel layer was coated by magnetron sputtering method at different deposition time, which varied from 10 to 40 minutes.

The analysis of the thickness of the coating was carried out in three different ways: by the distribution profiles of nickel in depth, by spherical abrasion test method and by X-Ray diffraction method.

Calculation of thickness by XRD method was carried out on a Shimadzu XRD-7000S diffractometer in the grazing beam geometry. The penetration depth of the X-Ray beam varied by reducing the angle of incidence until the signal from the substrate is supressed [2].

Measurements of the adhesion strength were made by the Micro-Scratch Tester MST-S-AX-0000. The critical load was determined during the measurement by scratching with a diamond indenter. The critical load is the minimum load at which the coating starts to destroy.

Present study demonstrates the possibility of determining the thickness of the micronic coatings by X-Ray diffraction method. The correlation between the calculation results of the distribution of nickel in depth, by spherical abrasion test method and by X-Ray method is established. The appropriate thickness of the nickel layer with the best adhesive properties and acceptable rate of hydrogen sorption was determined.

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