

## INFLUENCE OF TITANIUM IMPLANTATION ON HYDROGEN SORPTION RATE OF Zr-2.5Nb

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## ВЛИЯНИЕ ИМПЛАНТАЦИИ ТИТАНА НА СКОРОСТЬ СОРБЦИИ ВОДОРОДА Zr-2.5Nb

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***Аннотация.** Представлены результаты исследования влияния плазменно-иммерсионной ионной имплантации титана на скорость сорбции Zr-2.5Nb. После имплантации титана снижается концентрация и скорость сорбции водорода, что связано с образованием диффузионного барьерного слоя, который снижает диффузию водорода в объем образца.*

Zirconium and its alloys are important constructional materials of light-water nuclear reactors. Hydrogen is released under the influence of radiation by water radiolysis, which leads to hydrogen embrittlement of zirconium cladding. Ion surface modification and coating deposition are a way to protect zirconium alloys from hydrogen embrittlement.

The plasma immersion ion implantation (PIII) is the process in which highly negative pulsed bias is applied to the target material which is immersed in plasma causing the positively charged ions to accelerate to the target surface at very high velocities. The accelerated ions penetrate into the lattice structure of the target material which resulted in a change in the chemistry and microstructure of the target [1].

Titanium implantation improves the oxidation and corrosion behaviors of zirconium [2-4]. However, the influence of titanium implantation on hydrogenation and mechanical properties has not been discussed. We choose titanium implantation since titanium has low neutron capture cross-section and we expect that titanium ion implantation can reduce hydrogen sorption rate. It also can improve the mechanical properties of zirconium alloys. In this paper, we described the influence of Ti ions implantation by PIII on the hydrogen sorption rate of Zr-2.5Nb alloy.

**Material and Methods.** Titanium implantation into zirconium alloy Zr-2.5Nb substrates were performed by PIII method using «Raduga Spectr» technique. The samples with fixed size of 1 mm thickness and 30 mm diameter were polished at first to the average roughness  $R_a$  of 0.045  $\mu\text{m}$ . The vacuum chamber was evacuated to the base pressure of lower than  $2.5 \times 10^{-3}$  Pa. Before deposition samples were subjected to ion bombardment in argon glow discharge at 1500 V for 3 min. Ti was used as the cathode material. Parameters of Ti implantation: total pressure 0.15 Pa, arc current 70 A, pulsed substrate bias 1500 V, implantation time 15 min.

Hydrogenation of the samples was performed using an automated complex Gas Reaction Controller LPB (USA) at 673 K constant temperature (6 K/min heating rate) and hydrogen pressure 2 atm. Hydrogenation time was 120 min. The surface morphology of the samples was studied using scanning electron microscope Hitachi S-3400N (Japan) and atomic force microscope Centaur UH R (Nano Scan Technology, Russia). Study of the elemental composition of samples was performed by high-frequency glow discharge optical emission spectroscopy (GD-OES) using GD-Profilер 2 technique (Horiba, Japan). Hydrogen concentration was measured according to the principle of melting in inert gas using hydrogen analyzer RHEN602 (LECO, USA).

**Results and Discussion.** Fig. 1 shows the surface morphologies of the samples after Ti implantation. The traces are observed on the surface after samples polishing. Microparticles are typical for the cathodic vacuum arc evaporation process. The plasma filtering reduces the quantity of microparticles on the surface. They have spherical shaped form in the size range of 0.1–1  $\mu\text{m}$  (Fig. 1).

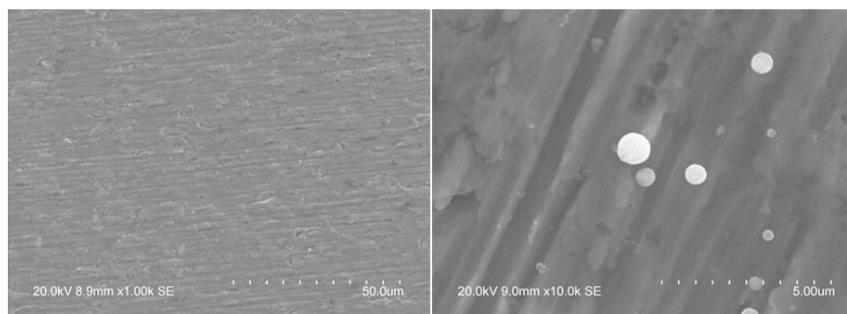


Fig. 1. SEM images of Zr–2.5Nb after Ti implantation

After titanium implantation and hydrogen saturation, a total hydrogen concentration in the implanted sample was 206 ppm and in the initial Zr–2.5Nb was 3920 ppm.

Fig. 2 shows the GD-OES depth distribution profiles of elements. The titanium signal was observed at the depth of ~250 nm (Fig. 2c).

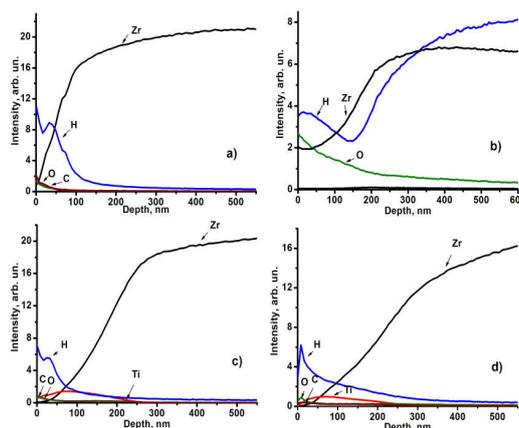


Fig. 2. The depth distribution of elements in Zr–2.5Nb: a) initial; b) initial after hydrogenation; c) after Ti implantation; d) after Ti implantation and hydrogenation

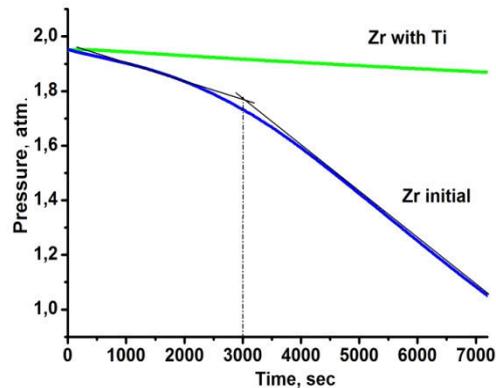


Fig. 3. Hydrogen pick-up curve of initial Zr–2.5Nb and after Ti implantation

The hydrogen sorption rate of the initial Zr–2.5Nb is significantly higher than the sorption rate of Zr–2.5Nb after Ti implantation. The hydrogen sorption rate of the initial Zr–2.5Nb is changed after 3000 sec from  $0.52 \cdot 10^{-3}$  to  $1.98 \cdot 10^{-3} \text{ cm}^3\text{H}_2/(\text{sec} \cdot \text{cm}^2)$ . This increasing of the initial Zr–2.5Nb sorption rate is caused by oxide film destruction and hydrogen embrittlement of zirconium alloy.

**Conclusion.** Titanium was implanted into Zr–2.5Nb alloy by PIII method. The modified surface layer reduces the hydrogen penetration depth of Zr–2.5Nb. Hydrogen sorption rate decreased after titanium implantation that is probably related to the defect structure formed during PIII.

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