



Full Length Article

High-intensity low energy titanium ion implantation into zirconium alloy

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ABSTRACT

This research describes the possibility of ultra-high dose deep titanium ion implantation for surface modification of zirconium alloy Zr-1Nb. The developed method based on repetitively pulsed high intensity low energy titanium ion implantation was used to modify the surface layer. The DC vacuum arc source was used to produce metal plasma. Plasma immersion titanium ions extraction and their ballistic focusing in equipotential space of biased electrode were used to produce high intensity titanium ion beam with the amplitude of 0.5 A at the ion current density 120 and 170 mA/cm². The solar eclipse effect was used to prevent vacuum arc titanium macroparticles from appearing in the implantation area of Zr sample. Titanium low energy (mean ion energy $E = 3$ keV) ions were implanted into zirconium alloy with the dose in the range of $(5.4\text{--}9.56) \times 10^{20}$ ion/cm². The effect of ion current density, implantation dose on the phase composition, microstructure and distribution of elements was studied by X-ray diffraction, scanning electron microscopy and glow-discharge optical emission spectroscopy, respectively. The results show the appearance of Zr-Ti intermetallic phases of different stoichiometry after Ti implantation. The intermetallic phases are transformed from both Zr_{0.7}Ti_{0.3} and Zr_{0.5}Ti_{0.5} to single Zr_{0.6}Ti_{0.4} phase with the increase in the implantation dose. The changes in phase composition are attributed to Ti dissolution in zirconium lattice accompanied by the lattice distortions and appearance of macrostrains in intermetallic phases. The depth of Ti penetration into the bulk of Zr increases from 6 to 13 μm with the implantation dose. The hardness and wear resistance of the Ti-implanted zirconium alloy were increased by 1.5 and 1.4 times, respectively. The higher current density (170 mA/cm²) leads to the increase in the grain size and surface roughness negatively affecting the tribological properties of the alloy.

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1. Introduction

Zirconium alloys (Zr-1Nb, Zr-2.5Nb, etc.) are widely used as fuel cladding material for Water-Water Energetic Reactor (WWER) due to low thermal neutron cross section, high corrosion resistance and required mechanical properties [1–3]. However, under normal operational conditions of WWER-type reactors, the fuel claddings are corroded in coolant stream [4]. Moreover, part of the hydrogen released during corrosion and water radiolysis is absorbed by the fuel cladding which causes hydrogen embrittlement of the material [5–7]. In case of a loss of coolant accident (LOCA) conditions, the temperature of fuel cladding rapidly increases up to 1200 °C causing high rate of corrosion and production of large amount of hydrogen [8]. Large amount of hydrogen released during the accident at the Fukushima-1 station in Japan in 2011 caused

explosions in the reactor building and seriously aggravated the accident progression [9]. In recent years intensive research has been carried out in the field of development new alloys also called accident tolerant fuel cladding materials as well as in the area of protection existing alloys from corrosion, hydrogenation and high-temperature oxidation by protective coatings deposition and surface modification [10–16]. The main advantage of ion implantation method of surface modification is the absence of problems with adhesion in comparison with deposited coatings. This advantage becomes one of the key factors that determines the prospects of this technology for fuel cladding processing. Significant reduction of hydrogenation of Zr-1Nb alloy after plasma-immersion titanium ion implantation was achieved due to the capture of hydrogen by the ion-modified layer [17,18]. It was also found that the thickness of the modified layer has significant influence on the diffusion rate and the amount of “trapped” hydrogen. Peng et al. showed the increase in the corrosion resistance of zirconium alloy as the function of the dose of implanted titanium [19].

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The depth of ion implantation is generally limited by the projected range of ions in the target material and usually does not exceed a micrometer [20]. The realization of the regimes of high-intensity repetitively pulsed ion implantation using filtered vacuum arc plasma makes it possible to increase the implantation depth to several micrometers and enlarge the concentration of the dopant due to compensation of ion sputtering by plasma deposition [21]. High-intensity low-energy implantation of metal ions has not been investigated yet due to the lack of appropriate equipment. In the case of gas ions in particular nitrogen ions, the method of high current implantation by ion beams with the current density up to 5 mA/cm^2 is widely studied and finds its practical application. Wei [22] showed that for deep ion implantation it is not necessary to use high energy ions. The ion energy should be sufficient for sputtering the surface oxide layer and overcoming the surface diffusion barrier. Deep nitriding of the near-surface layers of materials under low-energy ion implantation strongly depends on the ion current density while the ions diffuse under the concentration gradient according to the traditional diffusion theory [23]. Such implantation process is commonly called low-energy ion implantation (LEI) or high-current low-energy ion implantation using high-density ion beams. Recently, the possibility of low-energy metal and gas ion beams generation with the current density within the range from several tens of mA/cm^2 to 1 A/cm^2 was experimentally demonstrated in Ref. [24].

This work is devoted to the study of the principle possibility of high-intensity ($>100 \text{ mA/cm}^2$) implantation of low-energy titanium ions into zirconium alloy. The effect of Ti ion doping on the structure and mechanical properties of the alloy is also studied.

2. Materials and methods

The simplified schematic diagram of the experimental setup is shown in Fig. 1a. The DC vacuum arc discharge was used to form the dense metal plasma. The water-cooled titanium cathode (99.9% purity) was located within the modest axial magnetic field of 60 Gs. The arc discharge current was 100 A. The hemispherical metal grid was positioned on the central axis on the distance of 40 cm from the cathode working surface. The hemisphere radius was 7.5 cm (mesh size $1.4 \times 1.4 \text{ mm}$). The base pressure in the chamber was 10^{-3} Pa .

The circular open face of the mesh hemisphere was mounted on and electrically attached to the cylindrical section, providing the field-free drift space for ballistic focusing of the ion beam formed by the grid (Fig. 1b). The current collector plate, or sample holder

on which implantation targets might be placed, was positioned at the geometric focus and connected to the repetitively pulsed negative bias generator. In the experiment, the following parameters of the generator were used: amplitude of negative bias of 1.5 kV, pulse repetition rate of 10^5 pulses per second (p.p.s), pulse duration of 4 or 5 μs . A Rogowski coil was used to monitor the collector current. High intensity repetitively pulsed titanium ion beam was formed through the plasma-immersion ion extraction from the vacuum arc plasma with subsequent ballistic focusing of ions in the drift space. Compensation of the beam space-charge was provided by the injection of the plasma in the drift space in the pauses between bias pulses [24]. Macroparticles in the plasma flux incident upon the hemispherical grid were blocked from viewing the sample holder by the metal plate mounted on-axis as shown in the Fig. 1b.

The samples of zirconium alloy Zr-1Nb (0.9–1.1 Nb, 0.050, 0.015 Fe, 0.02C, Zr balanced, in wt%) with the size of $20 \times 20 \times 2 \text{ mm}$ were used as the target material. The samples were polished to the average roughness of $R_a = 80 \text{ nm}$ and cleaned with alcohol. The samples were mounted on the disc radiator allowing the sample temperature stabilization. The samples were located near the system focal plane (7.5 cm) or beside it at the distance of 8.5 cm. At the discharge current of 100 A, the pulsed density of an ion beam was 120 mA/cm^2 and 170 mA/cm^2 , respectively. The implantation was carried out at the temperatures in the range from 650 to $720 \text{ }^\circ\text{C}$. The sample temperature was controlled with thermocouple, mounted on the backside of a sample. The ion implantation dose was estimated as:

$$D = 6.25 \times 10^{18} \times j \times t \times f \times \tau / Z,$$

where j – ion current density, t – exposition time, f – pulse repetition rate, τ – pulse duration, Z – mean charge state of titanium ions.

The ion implantation dose, at the stabilized temperature mode, at current density of 120 mA/cm^2 was changing from $5.4 \times 10^{20} \text{ ion/cm}^2$ and pulse duration of 4 μs to $6.75 \times 10^{20} \text{ ion/cm}^2$ at pulse duration of 5 μs . At current density of 170 mA/cm^2 , the ion implantation dose reached $9.56 \times 10^{20} \text{ ion/cm}^2$. The total ion implantation dose was approximately 10% more through the implantation during the heating process.

Phase composition was analyzed by X-ray diffraction in $\theta - 2\theta$ geometry using Shimadzu XRD 7000S diffractometer equipped with high speed wide-range 1280 channels detector OneSighth. The diffraction data was analyzed using Sleve+ program. As a reference for the patterns of $\alpha\text{-Zr}$, $\text{Zr}_{0.7}\text{Ti}_{0.3}$ and $\text{Zr}_{0.5}\text{Ti}_{0.5}$ there were the ICDD PDF-4+ database: #04-004-8479, #01-072-3354 and

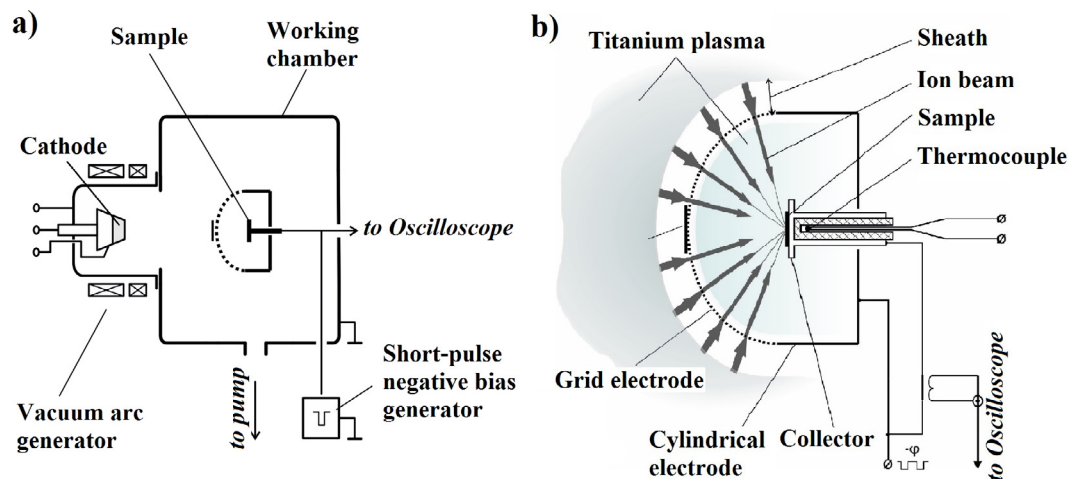


Fig. 1. Schematic diagram of the experimental setup (a) and ion implantation process (b).

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