

Features of relief formation of the polycrystalline samples under the influence of a wide-aperture ion beam

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Abstract. The results of investigation of the cylindrical surface state after influence of wide-aperture ion beam are presented in this paper. Experiments were carried out on the parts of cladding tubes from E110 alloy (Zr-1%Nb) up to 500 mm length. The outer surface of the tubes was treated by Ar^+ ions with energy 0.1-1.0 keV up to doses $(1-10) \times 10^{18} \text{ cm}^{-2}$ on the installation KVK-10. Due to design features of the installation, the angle of incidence of particles on the surface varied from 0 to 90°. It is found that as a result of treatment made, smoothed relief of the surface forms and uniformity of near-surface layer structure improves. The statistical method suggested allowed to reveal features of the polycrystalline samples surface state with random and regular components, based on the results of profilograms analysis.

1. Introduction

As is known, many service properties of the products, such as corrosion resistance, friction, wear, etc. essentially depend on the condition of the surface layer material. In particular, the protective properties of the oxide films formed on the external surface shells of nuclear reactors fuel elements from zirconium alloys is largely determined by the state of the shells external surface the presence of stress and technological defects and impurities in the near-surface layer of the material etc. [1-2] One of the most effective surface modification methods is ion-beam processing. As a result of charged particle beams the material near-surface layer spraying is taking place, a special relief forms, and new non-equilibrium state can arise in the area of radiation exposure. Because the ion-induced processes are not thermo-activated, the limits of the material surface properties control significantly expanded in comparison with the traditional treatment methods. To increase the treatment homogeneity of polycrystalline materials is the use of a wide-aperture low-energy ion beam of inert gases is interesting.

2. Materials and tools

Formed as samples were used fragments of staff claddings pressurized water reactors of E110 alloy (Zr-1% Nb, an outer diameter of 9.15 mm, length 500 mm), in the state after mechanical factory polishing. The external surface the tubes treated with wide-aperture beam of Ar^+ ions with energies of 0.1-1.0 keV (average 500 eV) to doses $(1-10) \times 10^{18} \text{ cm}^{-2}$ at the installation KVK-10 [3]. The radiation dose was chosen on the material surface layer spraying calculation the thickness up to 2 μm , which was determined based on the microbalance 1 mg = 0.17 μm . The samples weight was carried out on an analytical balance GH-252 company A&D (sensitivity of 0.01 mg). The outer surface temperature of



the samples during the exposure does not exceed 150-200 °C. As can be seen from the circuit in Figure 1, the ions incidence angle at the sample during treatment varies from 0 to 90°, resulting in peak energy release shifts toward the material surface (see. Figure 2) decreases the thermal load on the sample and improves the treatment homogeneity. The using expose geometry allows to avoid the relief development due to the smoothing of the maximum in the angular distribution of the sputtering in this energy range (Figure 3).

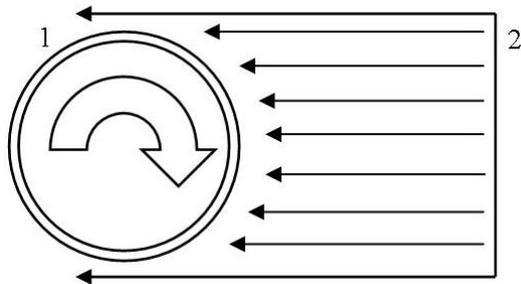


Figure 1. Scheme of treatment of the cylindrical samples on the installation KVK-10 (top view): 1 – sample, 2 – wide-aperture ion beam, arrow shows the direction of the sample rotation.

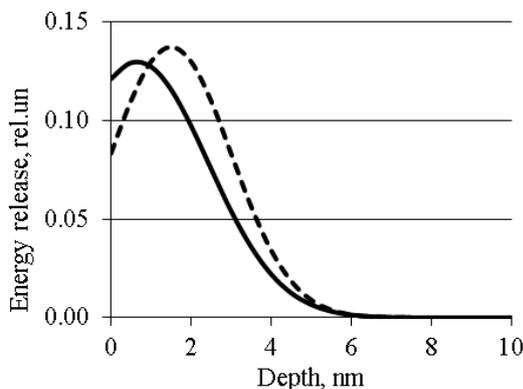


Figure 2. Distribution of energy release by depth of the Zr sample under the influence of focused (dotted line) and wide-aperture (solid line) Ar^+ ion beam (calculation by SRIM-2012).

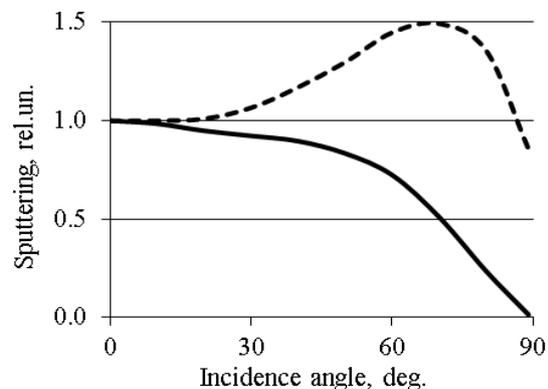


Figure 3. Dependence of the normalized Zr sputtering by wide-aperture Ar^+ 500 eV beam from its angle of incidence on the flat (dotted line) and cylindrical (solid line) surface (calculation by SRIM-2012).

Microhardness measurement of ion-treated surface was carried out on a PMT-3 (indenter - a diamond pyramid with an angle of 136°, load 70 g, loading time 30 seconds). State of the near-surface layer was studied by optical and scanning ion microscopy (SIM unit Helios-660, a beam of Ga^+ , the accelerating voltage of 5 kV). To reveal the material structure on the sample surface using the ion microscope in the milling mode are cut out the hole measuring $15 \times 15 \mu\text{m}$, after that obtained cross-section pictures are made. Profilograms ion-treated surface were filmed along a generator tubes at the contact profilograph TR-200 (scanning length of 1.0 mm, 0.5 mm step measurement) with subsequent transfer of data to a PC automatically. As a profile of the center line to a first approximation, the straight line was chosen whose coefficients were determined by the method of least squares (according to GOST 2789-73).

It is known that, for analysis topography convenience real surface profile of a material can be represented as a superposition of systematic and random irregularities [4]:

$$h(x) = h_{\text{sys}}(x) + h_{\text{rand}}(x), \quad (1)$$

where $h_{\text{sys}}(x)$ and $h_{\text{rand}}(x)$ – the systematic and random components, respectively.

To systematic component relate regularly arranged elements of the relief, which may occur in the process of permanent technological factors, such as, for example, the shape, size and vibration of the

cutting tool. Random component in its turn is formed from a large number of irregular effects on the surface of the material, such as, for example, treatment by the abrasive particles. As a first approximation we can assume that $h_{\text{sys}}(x)$ - there is some periodic function, and $h_{\text{rand}}(x)$ - random stationary process with zero mean. As is known, one of the most informative methods of stationary random processes description is the autocorrelation function $K(\tau)$, which characterizes the values dependence at the predetermined point x on the values of the same process at another point $x + \tau$ with displacement τ , and for a limited set of discrete values considering normalization can be evaluated by the formula [4]:

$$K(\tau) = \frac{\sum_{i=1}^{n-1} (h(x_i) - \langle h \rangle)(h(x_i + \tau) - \langle h \rangle)}{\sqrt{\sum_{i=1}^{n-1} (h(x_i) - \langle h \rangle)^2 \sum_{i=1}^{n-1} (h(x_i + \tau) - \langle h \rangle)^2}}, \quad (2)$$

where n - is number of measurements h_i , x_i - x coordinate of the measurement point, $\langle h \rangle$ - arithmetic average of the measured values of $h_i = h(x_i)$. Considering (1), the calculated autocorrelation function of the real profile allows to allocate the systematic and random components and obtain information both about the profile as a whole and its separate components. The solution to this problem seems relevant since identifies mechanisms of relief formation in the treatment process and to correct its parameters according to their stated objectives.

3. Results and discussion

Figure 4 shows distribution histograms values of the microhardness of the samples outer surface in the initial state (after mechanical treatment) and after ion treatment to $(3-10) \times 10^{18}$ ions cm^{-2} . Performed analysis shows that the ion treatment doesn't change the microhardness mean value of the outer surface, which is about 1.86 GPa. Where in the scatter of values significantly reduces - from 0.18 GPa for the sample in the initial state to 0.08 GPa for the sample after ion-beam processing, and distribution becomes more random. These changes evidence about the near-surface layer homogenization of the material thickness, commensurable with the depth indenter penetration, which is under the chosen conditions of loading of 3-4 μm .

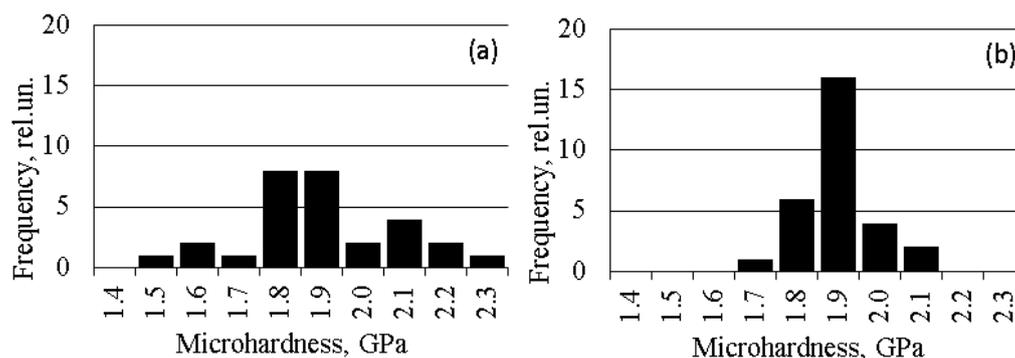


Figure 4. Histograms of microhardness distribution of the samples outer surface after mechanical abrasion (a) and ion treatment to $(3-10) \times 10^{18}$ cm^{-2} (b).

Figures 5 and 6 show cross-sectional SIM-photos on the samples surface in the initial state and after ion beam treatment to 9×10^{18} cm^{-2} , respectively. Visually, it is seen that in the considered scale of the original surface relief of the sample has a characteristic wavy shape which is almost completely is aligned after ion treatment. Near the untreated sample surface is observed strongly distorted structure (detected layer thickness is about 2 μm - shown by the arrows in Figure 5), which is probably formed as a result of material deformation during the surface mechanical treatment, possibly in combination with saturation of this layer by technological impurities. The structure of the sample near-surface layer

after ion beam treatment consists of well-defined grains with an average size of 3-5 μm (grain boundaries are shown by the arrows in Figure 6) that corresponds to the main state of material volume.

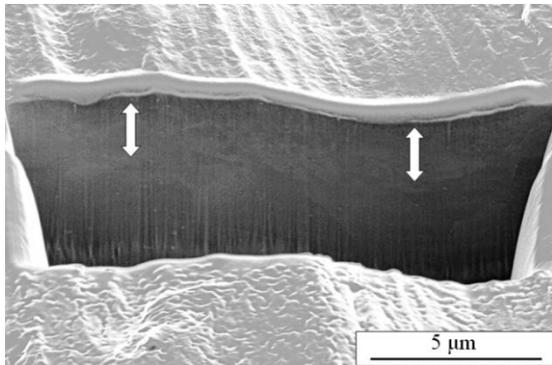


Figure 5. SIM-image of cross section on the outer surface of the sample from E110 alloy after mechanical abrasion (arrows show the boundaries of the defective surface layer).

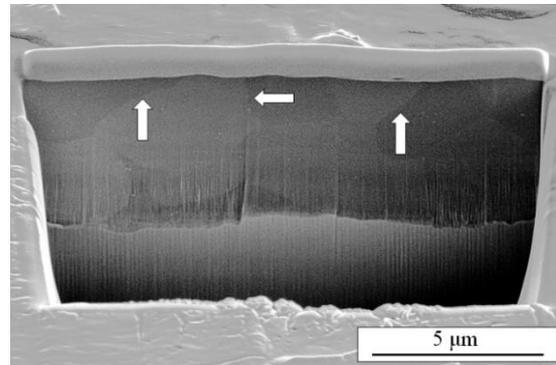


Figure 6. SIM-image of cross section on the outer surface of the sample from E110 alloy after the treatment by Ar^+ beam to $9 \times 10^{18} \text{ cm}^{-2}$ (arrows show the grain boundaries near the surface of the material).

Figure 7 shows the profiles (a) and charts of normalized autocorrelation functions (b) the external surface profiles of samples in different states. As can be seen, the autocorrelation function at the coordinate has a characteristic recession and then oscillates around zero. With increasing irradiation dose, the graph $K(\tau)$ becomes smoother, and the periodic component having a wavelength equal to about 0.1-0.2 mm detects at the oscillating portion. These changes point to a reduction in the share of casual and part - systematic profile roughness whose characteristic size amounts less than 100 μm , which confirms the results obtained by the SIM.

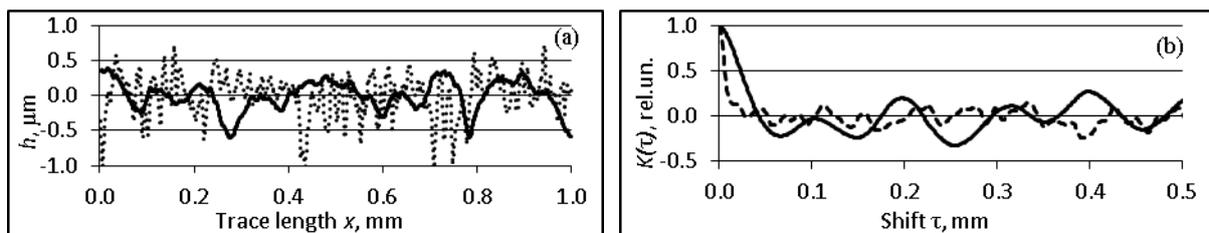


Figure 7. Profilograms (a) and autocorrelation functions (b) of the outer surface of the samples form E110 alloy after mechanical abrasion (dotted line) and ion-beam treatment to $10 \times 10^{18} \text{ cm}^{-2}$ (solid line).

4. Conclusion

Investigated the state of the external surface the tubular samples of zirconium alloy E110 (Zr-1\%Nb) after treatment with a wide-aperture beam of Ar^+ ions with energies 0.1-1.0 keV.

Analysis of the microhardness measurement results and SIM images showed that by the removal the material with the thickness of 1-2 μm by physical sputtering, the smoothed surface relief structure forms and the near-surface layer material homogenizes.

With the proposed statistical method of processing profilograms it found that the carried out treatment leads to a reduction in the contribution of the surface topography of the random component that occurs in the process of mechanical material grinding and accompanied by a significant distortion of the structure.

Acknowledgements

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