Finish ion beam treatment of the longrange cylindrical products outer surface in automatic mode

B A Kalin[1], N V Volkov[1], R A Valikov[1], A S Yashin[1], V P Krivobokov[2], S N Yanin[2], O Kh Asainov[2] and Yu N Yurev[2]

[1]National Research Nuclear University MEPhI (Moscow Engineering Physics Institute), Kashirskoe highway 31, Moscow, 115409, Russia
[2] National Research Tomsk Polytechnic University, Lenin Avenue 30, Tomsk, 634050, Russia

E-mail: yashin_itf@mail.ru

Abstract. The results of using of ion-beam technologies methods for finish treatment of metal products are presented. The experiments were performed at the installation ILUR-03, which allows the operation of cleaning, polishing and surface layers doping of the material of unlimited length cylindrical samples by radial Ar^+ ions beam with energy up to 5 keV. The tubes from zirconium alloy E110 up to 500 mm length were used as samples for investigation. It is shown that selected automatic treatment modes reduce the surface roughness over the entire length of the samples and increase uniformity of the surface layer without observable effect on the bulk properties of material. Treatment promotes the formation of oxide films with improved defensive properties.

1. Introduction

Materials resistance to corrosion and wear largely depends on such characteristics of the surface layer of material, such as topography, elemental composition and the presence of impurities, grain size, etc. Wherein, state of the surface is often substantially different from the main volume material. For example, mechanical abrasive treatment, reducing roughness significantly deforms material barrier layer and the chemical etching contributes to the material saturation by difficult to remove impurities [1-3]. In connection with this the using of environmentally friendly and non-contact ion beam technologies for finishing of metal products is interesting.

2. Materials and tools

For the experiments carrying out was used the installation ILUR-03 [4], designed to clean and polish the outer of cylindrical samples surface with unlimited length by radial beam of Ar^+ ions with an energy of 5 keV. The design of installation also provides for local doping of the material product surface layerin the mode of ion mixing films, deposited built magnetrons. The appearance of the installation is shown in Figure 1.

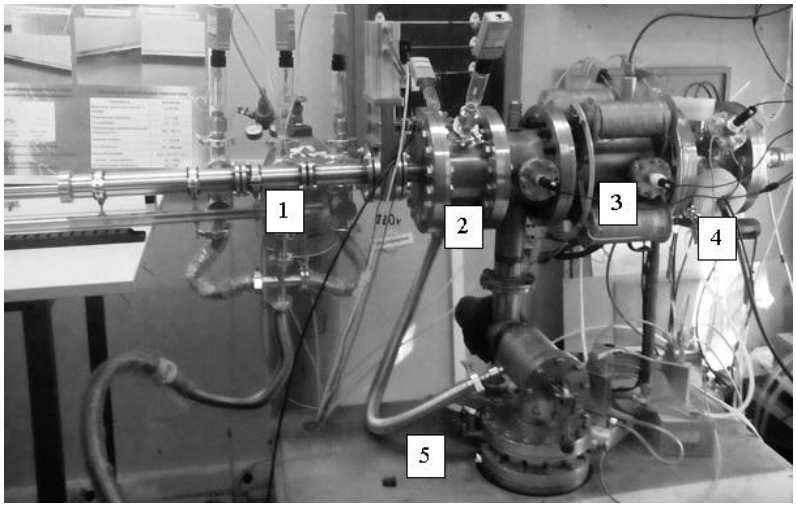

Figure1. Installation for ion-beam treatment of long range products ILUR-03:
1 – gateway chamber, 2 - preliminary pumping chamber, 3 – chamber with ion source, 4 – chamber with magnetrons, 5 – vacuum system desk.

Using an ion beam with a wide energy spectrum allows to increase the processing speed at a constant power load on the sample, as well as to increase the depth of the modified layer what can be seen in Figures 2 and 3, respectively.

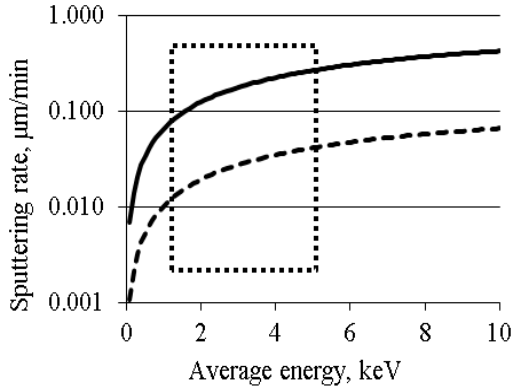

Figure 2. Dependence of sputtering rate of Zr sample on average energy of Ar^+ ion beam with narrow (dotted line) and wide (solid line) energy spectrum (calculationbySRIM-2012): area of ILUR-03 ion beam is shown by dotted rectangle.

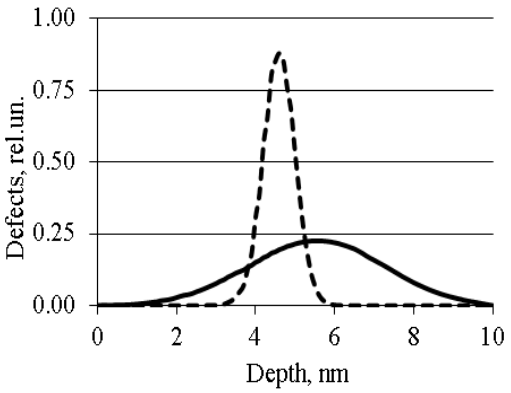

Figure 3. Distribution of defects generated by Ar^+ beam by depth of Zr sample for the ion beam with narrow (3.0-4.0 keV – dotted line) and wide (1.0-5.0 keV – solid line) energy spectrum (calculation by SRIM-2012).

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, wall thickness 0.65 mm, length 500 mm) from the factory mechanical polishing the outer surface.

For analysis of the material surface layers was measured roughness (roughness profiler, TR-200), and microhardness (PMT-3, indenter - diamond pyramid with an angle of 136 °) the surface of the samples at various points along the length of the membranes. Several tubes before and after ion treatment were subjected to autoclave tests in the steam-water medium at a temperature of 350 °C and a pressure of 16.5 MPa on a temporary base to 500 hours. The cross structure of the grown oxide are studied by scanning ion microscopy methods (SIM unit Helios-660, a beam of Ga^+, the accelerating voltage of 5 kV).

3. Results and discussion

Figures 4-6 show the SIM- images of samples outer surface in the initial state and after the treatment beam Ar^+ 3×10^{18} cm^{-2} and 9×10^{18} cm^{-2}, respectively. As can be seen, on the surface in the initial state is having a significant amount of both longitudinal and transverse crack with sharp edges (indicated by arrows in Figure 4), the value of the roughness of $R_a = 0.8 \pm 0.1$ μm. With increasing irradiation dose, the number of scratches is reduced, the relief surface becomes smoother character roughness decreases to $R_a = 0.5 \pm 0.1$ μm, while the crystallite boundaries are detected (indicated by arrows in Figure 5).

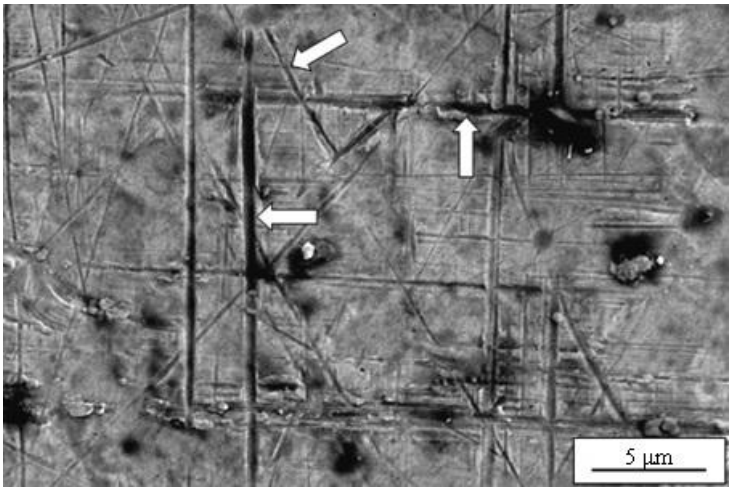

Figure4. SIM-image of the sample from E110 alloy outer surface in initial stage (arrows show the technological scratches).

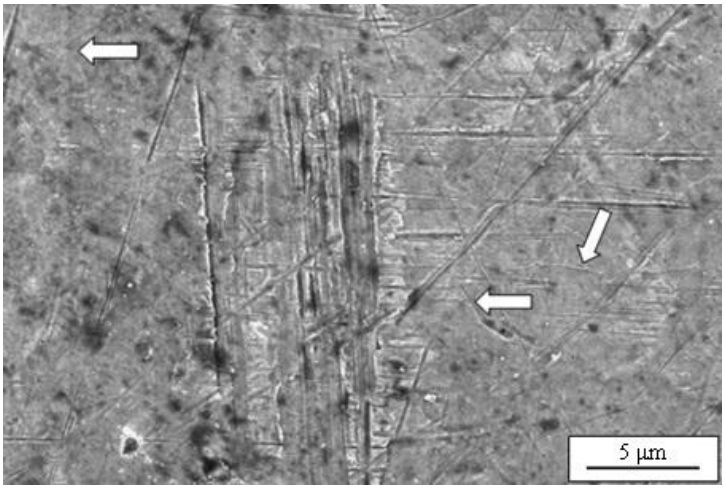

Figure5. SIM-image of the sample from E110 alloy outer surface after treatment by Ar^+ ion beam up to 3×10^{18} ions cm^{-2} (arrows show the crystallite boundaries).

For fluence 9×10^{18} cm^{-2} (Figure 6) vertical drop of neighboring grains, is measured by the photos, amounts to about 0.1 microns with roughness values $R_a = 0.3 \pm 0.1$μm. This value corresponds to the calculated value due to different speed spraying basal and prismatic planes face-centred close-packed lattice of α-Zr. Thus, for the polycrystalline samples surface polishing by ion beam is required to limit the maximum dose, depending of the required final roughness value.

Analysis of the measurement results of the microhardness showed that the matrix treatment of the zirconium alloy by Ar^+ ions beam at modes of cleaning and polishing does not change the average $H\mu$, which is 1.5 GPa, but significantly reduces the scatter in the values of microhardness for sample length, as can be seen from Table 1. The doping of the near-surface layer of the matrix by selected elements (Fe, Al, Ti) at ion mode mixing increases the magnitude of $H\mu$ to 1.6-1.8 GPa while maintaining reduced level of spread of values. The obtained results probably indicate the gradient structure formation on the material surface with high uniformity thickness of about 1 μm (a diamond indenter indentation depth of 4-5 μm).

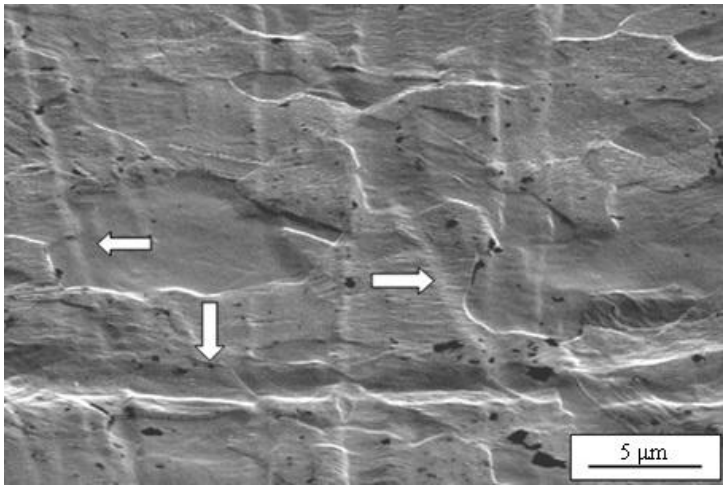

Figure 6. SIM-image of the sample from E110 alloy outer surface after treatment by Ar^+ ion beam up to 9×10^{18} cm^{-2} (shooting at angle 52°, arrows show the smoothed technological scratches edges).

Table 1. Microhardness of tubular samples outer surface.

№	Sample state	H_μ, GPa	Dispersion $\Delta H_\mu/H_\mu$, %
1	initial (mechanical abrasion)	1.5	18
2	Ar^+ treatment up to 5×10^{18} cm^{-2}	1.5	9
3	Ar^+ treatment up to 1×10^{19} cm^{-2}	1.5	15
4	Ar^+ 1×10^{18} cm^{-2} + doping Fe	1.6	11
5	Ar^+ 1×10^{18} cm^{-2} + doping Al	1.8	11
6	Ar^+ 1×10^{18} cm^{-2} + doping Ti	1.6	9

Table2. State of the oxide films on the cladding tubes from E110 alloy outer surface after autoclave tests up to 500 h.

№	Sample state	Oxide film thickness, μm	Pores size and density
1	initial (mechanical abrasion)	0.8±0.1	10±1 % 0.2±0.1 μm
2	Ar^+ treatment up to 5×10^{18} cm^{-2}	0.8±0.1	6±1 % 0.2±0.1 μm
3	Ar^+ treatment up to 1×10^{19} cm^{-2}	0.7±0.2	uniform film
4	Ar^+ 1×10^{18} cm^{-2} + doping Fe	0.4±0.1	uniform film
5	Ar^+ 1×10^{18} cm^{-2} + doping Al	0.5±0.1	uniform film

As can be seen from the results of the autoclave tests, presented in Table 2, the oxide film formed on the ion-modified surface, characterized by increased protective properties (data table obtained by analysing images of the transverse structure of oxide by SIM). On the shell in the initial state the film has a thickness of 0.8 ± 0.1 μm, its body contains pores (gaps) by 0.2 ± 0.1 μm and a bulk density of 10 ± 1%. On the time oxidation basis up to 500 hoursoxide at the samples after ion treatment usually has, as a rule, a smaller thickness and great homogeneity.The best result shows the sample doped with Fe - oxide thickness of 0.4 ± 0.1 μm, pores and discontinuities isn't observed in the body of the film.

4. Conclusion

The influence of the finish ion-beam treatment the external surface the shell pipe length of E110 alloy to 500 mm on the condition of the near-surface layer of material and corrosion productsis studied. Machining was carried out on the installation ILUR-03 in modes of cleaning, polishing and additional alloying of thesample material surface layer by ion mixing radialAr^+ions beam.

It is shown that the selected processing modes reduce the surface roughness of the pipe throughout its length with $R_a = 0.8 \pm 0.1$ μm to $R_a = 0.3 \pm 0.1$ μm.Wherein the homogeneity of the surface and the protective properties of the oxide formed in the autoclave conditions (350 °C, 16.5 MPa, 500 h) increase.

Acknowledgements

This work was performed within the framework of the Center of Nuclear Systems and Materials supported by MEPhI Academic Excellence Project (contract № 02.a03.21.0005, 27.08.2013).

References

[1] Nikulina A V and Malgyn A G 2008 *Atomic Energy* **105** 259
[2] Khusu A P, Vitenberg Yu R and Palmov V A 1975 *Surfaces roughness: theoretical-probability method* (Moscow: Science) p 344
[3] Zaymovsky A S, Nikulina T V and Reshetnikov N G 1994 *Zirconium alloys in nuclear power engineerinig* (Moscow: Energoatomizdat) p 256
[4] Volkov N V, Kalin B A, Valikov R A, Yashin A S and Yalcev V N 2012 *Russian Phys. J.* **55** 186