Modification of the cylindrical products outer surface influenced by radial beam of argon ions at automatic mode

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Abstract. Obtaining surface with high purity and good roughness is important for increasing the corrosion resistance and wear resistance of products working in corrosion-active environment. Installation ILUR-03 with the coaxial ion beam wide energy spectrum source for cleaning, polishing and surface doping of long cylindrical items has been developed. Upgraded installation ILUR-03 provides effective technological defects cleaning (abrasives after mechanical polishing, acid residues after chemical etching, adsorbed gases), surface polishing, film deposition by using magnetrons and surface doping by ion mixing method in one technological cycle.

1. Introduction

Such surface's characteristics as topography, elemental composition, grain size of the surface layer have a significant effect on the wear resistance and corrosion resistance of the final products. Surface impurities and bad roughness parameter reduces resistance of the material to nodular corrosion. In this regard the development of cleaning and the polishing surface technology by ion beams is promising because during mechanical or chemical polishing product's surface is contaminated by abrasive particles or acid.

Materials and tools 2.

Installation ILUR-03 (fig. 1) has been developed in our university. It allows cleaning and polishing cylindrical items outer surface by ion beam with wide energy spectrum [1]. The ion source with wide energy spectrum increases speed of the process and also increases thickness of the modified layer (fig. 2). The chamber with magnetron source allows sputtering the metal films and ion source allows doping surface by ion mixing method after that.

Fragments of zirconium alloy E110 tubes (diameter - 9.15 mm, wall thickness - 0.65 mm, length - 30 mm and 500 mm) were used as samples.



Figure 1. Scheme of the installation ILUR-03: 1 -gateway, 2 -transition chamber, 3 -ion cleaning chamber, 4 -ion polishing chamber, 5 -magnetron chamber, 6 -exit items chamber, 7 -manipulator with sample holder, 8 -sample, 9 -vacuum pump connection



Figure 2. Calculated sputtering rate of zirconium: 1 - monoenergetic ion beam, the ion current 8 mA, 2 - monoenergetic ion beam, the ion current 16 mA, 3 - ion beam with the wide energy spectrum, the ion current 8 mA, 4 - ion beam with the wide energy spectrum, the ion current 16 mA

To determine the limit processing mode the thermal distribution along the length of the tube during process and the maximum of the temperature for different thermal load were simulated (Fig. 3). Experimental measurements of sample heating in the processing zone at different power beam were conducted (Fig. 4). Ion cleaning and polishing modes were determined by using these data and with calculations of zirconium sputtering rate (the average ion energy in the beam 3-4 kV, the ion current of $0.4-0.7 \text{ mA/cm}^2$, the radiation dose to $5 \times 10^{18} \text{ ions/cm}^2$). The maximum speed of the surface cleaning is 4 m/min. Polishing rate depends on the initial state of the surface and can reach 0.2-0.3 m/min.

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Figure 3. Calculated graph of processing item maximum temperature



Figure 4. Graph of the sample temperature rise in the treatment zone

3. Experimental results and discussion

After the treatment samples microhardness and roughness were measured. Some partil of the samples were tested in autoclave in condition simulating a nuclear reactor operating parameters (environment - water, temperature -350°C, pressure - 17 MPa). The condition of the surface and oxide films was studied using scanning ion microscopy (SIM).

Microhardness measurements along the length of the sample before and after treatment showed that the ion polishing doesn't change microhardness value. At the same time the variation of values after treatment is considerably reduces [2]. This changes indicates great homogeneity of the surface layer commensurate with the depth of indenter penetration which is equal 3-4 microns at selected load conditions. This increase of the homogeneity is probably happened due to removal of the material surface layer enriched by different type of technological defects [3].

Studying the surface roughness before and after ion treatment was carried out on profilometer TR-200 (f. A&D, Japan). The initial roughness R_a of the samples (after mechanical polishing) was 1.0 microns. Ion polishing provides great scratch smoothing. It reduces average surface roughness of up to $R_a = 0,3-0,5 \mu m$ (fig. 5, 6).



Figure 5. Samples before ion polishing (Ø9.15 mm)



Figure 6. Samples after ion polishing (Ø9.15 mm)

Estimation of the surface layer condition by the SIM has showed that ion treatment removes deformed layer that occurs at the mechanical polishing. The amorphous material layer after ion polishing is forms by the accumulation of defects on the surface. This layer helps to reduce stress in the oxide film at the first stages of oxidation and also reduces the oxygen penetration rate through the 'metal-oxide' border.

To examine the oxide films after the autoclave test ion scanning microscopy was used. To study the internal structure of the oxide (estimated thickness, the presence of pores and cracks) the holes were cut by using gallium ion beam. Surface alignment and amorphous layer formation on the surface by ion treatment promotes formation of the oxide film with a monoclinic crystal lattice. This lattice has protective properties and strongly adheres to the substrate. It also reduces the oxide growth rate. The slow down growth rate tendency of the oxide after ion polishing occurs after 500 hours of the oxidation (fig. 7, 8). After 3000 hours autoclave tests in the initial samples occurs a significant number of pores and cracks near the boundary 'metal-oxide'. That increases the migration of the oxygen to the metal and as a result increases oxide film growth rate (fig. 9, 10). At the same time concentration and the pore's size in the bulk of the sample's oxide after ion polishing and further corrosion test for 3000 hours is much lower (the difference is 30-40%).



Figure 7. Cross structure photo of the oxide film after 500 hours autoclave test obtained by SIM (the samples in the initial state)



Figure 8. Cross structure photo of the oxide film after 500 hours autoclave test obtained by SIM (the samples after ion polishing)



Figure 9. Cross structure photo of the oxide film after 3000 hours autoclave test obtained by SIM (the samples in the initial state)



Beam Note Constant Figure 10. Cross structure photo of the oxide film after 3000 hours autoclave test obtained by SIM (the samples after ion polishing)

4. Conclusions

- The possibility of using ion polishing as a finish operation of manufacturing long cylindrical items that requires high surface quality (surface roughness achieved 0.3 ± 0.1 mm) is shown.
- The experimental automatic ion cleaning and polishing installation ILUR-03 was created. The installation provides cleaning and polishing of the long products and doping the surface layers by the ion-mixing method.
- The experimental modes that allow cleaning of the surface at a speed up to 4 meters per minute were received.
- An experimental result shows that ion treatment improves corrosion resistance by 30-40%.

References

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