

BIOMATERIAL AISI 316L SS SURFACE FILM ANALYSES AFTER ELECTROPOLISHING TREATMENTS UNDER VARIED MAGNETIC FIELDS

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Introduction

Type 316L stainless steel is one of the most commonly used medical grade materials because of its good corrosion resistance and cleanability [1, 2]. Our up-to-date studies have revealed that its surface properties, specifically corrosion resistance to pitting, may be significantly enhanced after electrolytic polishing in a magnetic field [2-5]. It is the surface oxide film formed after electropolishing which makes the difference in the behaviour in corrosion media. Many surface analytical techniques have been used to determine the composition of surface film formed [2-6]. Specifically, Auger Electron Spectroscopy (AES) is one of the most appropriate to investigate the elemental composition [4, 5].

The studies of surface film formed on AISI 316L SS biomaterial after its electropolishing (EP) under varied conditions of the magnetic field intensity (magneto-electropolishing MEP) [2, 3, 7] and the process current density applied, have been the main aim of the work. The results of the AES analyses of surfaces are compared with those obtained on the samples after a conventional electropolishing (EP).

Experimental

Materials and set-up

Two sets of austenitic stainless steel samples of dimensions 25×5×1 mm have been prepared for the investigation. The first one was electropolished (EP) in the transpassive region at the anodic current density of 500 A/dm² (EP500) and at about 1000 A/dm² (EP1000). The second set of samples was treated by electropolishing using a magnetic field (magneto-electropolishing MEP) of intensity 66 mT up to 450 mT, and current density from 50 A/dm² up to 800 A/dm², e.g. MEP66/190 up to MEP450/800, leaving all other conditions unchanged.

Apparatus and procedures

The set up for electropolishing consisted of a potentiostat and a controller. The studies were carried out for a broad range of current densities (up to 1200 A/dm²), potential to about 10 V, vs. MSE (mercury-sulphate electrode), and electrolyte temperature of

60 °C at the temperature control equaled ±1 °C. For the studies a proprietary sulphuric/orthophosphoric acids mixture electrolyte was used [2-4, 7].

Auger measurements were performed in a PHI model 4200 Thin Film Analyzer, equipped with a variable resolution cylindrical Mirror Analyzer (CMA) and a coaxial electron gun. Quantitative analysis is obtained using reference materials and the elemental sensitivity factors (SF). The system base pressure was 1.6×10⁻⁹ Torr. As a rule, the interface time *t* was determined between 84 and 16% of the intensity change at the interface [5]. A 2.44 nm/min erosion rate was obtained for the silicon dioxide.

Depth profiles were obtained by alternating sputtering and acquisition cycles. A 2 keV Ar⁺ beam, rastered over an area of 2 x 2 mm, sputtered the samples while a 3 keV, 450 nA electron beam excited the Auger electron.

Results and discussion

Scans of the whole range of Auger electron energies (20-1020 eV) were carried out by detecting and counting the number of Auger electrons. The analysis gives information about the presence of elements and contaminants on the sample surface region (2-20 atomic layers). By taking into account the SF's of the elements detected, quantification is possible. This method is useful in identification the unknown elements and estimating their concentration on the sample surface.

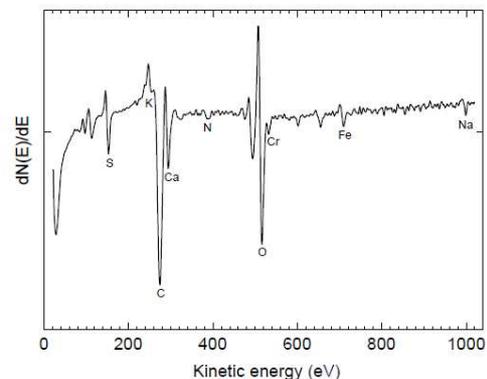


Fig. 1. AES Survey on the sample EP500

The AES depth profile measurements were performed for all studied samples. **Figures 1, 2** show the AES selected spectra acquired on the surface of EP500, and MEP225/525, respectively.

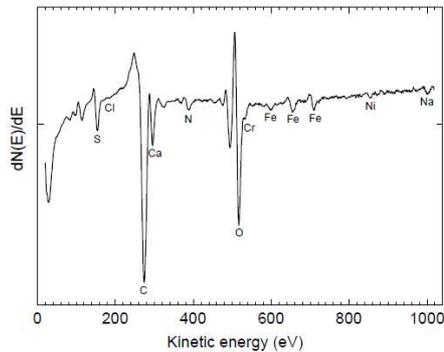


Fig. 2. AES survey on the sample MEP225/525

Excepting for the EP1000 sample (not presented here), where a thicker oxidation layer was detected (above 10 nm), no remarkable differences can be observed (mostly 4-7 nm) when comparing the elemental composition as a function of depth.

Oxygen, iron, nickel and chromium regions have been individually reported. The pictures obtained allow to appreciate the small differences in the depth compositional profiles, i.e. thickness of the outermost oxide layer of iron, nickel, and chromium (**Fig. 3**) as well as oxygen (**Fig. 4**) surface enrichment phenomena.

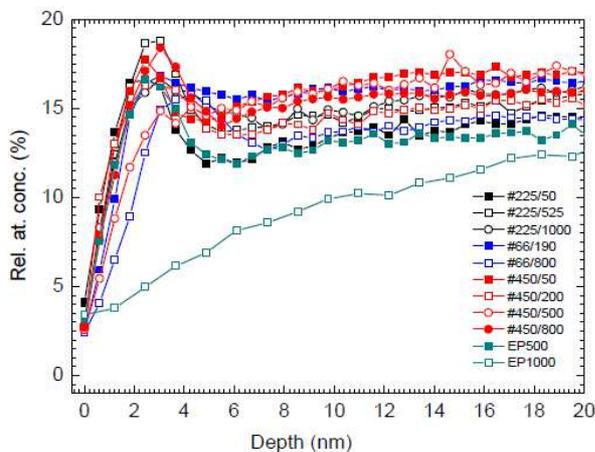


Fig. 3. Chromium depth profile of the studied samples in the depth range 0-20 nm

Conclusion

The Auger spectra acquired on the received surfaces reveal a remarkable contamination of carbon. Its high concentration (>50%) has a “shadow” effect on the elements composing the sample matrix (Fe, Ni, Cr, O). Surface is also affected by trace amounts of a variety of elements (S, Cl, Ca, K, N and Na) due to contamination or process residue.

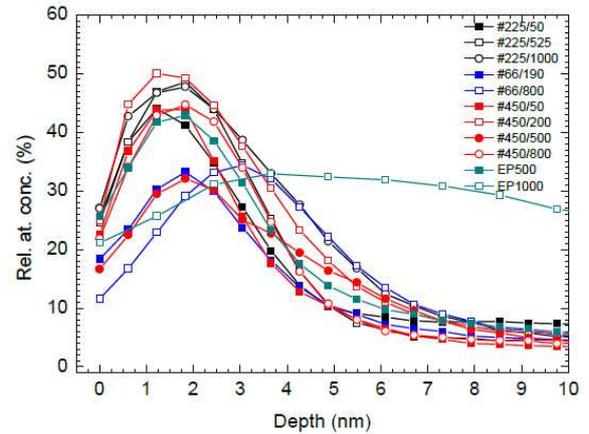


Fig. 4. Oxygen depth profile of the studied samples in the depth range 0-10 nm

Except for the EP1000 sample, depth profile analyses show on outermost oxide layer whose thickness does not exceed the 5 nm for all samples. Carbon and other contaminants (not reported here) rapidly decrease with the erosion time. A sub-surface enrichment layer can be observed for both chromium and nickel. To conclude, the EP1000 sample clearly shows remarkable discrepancies in the depth compositional analysis compared to the others. As for the small differences (oxide thickness, C contamination, Ni and Cr behaviour) in the other samples; some of the can be well observed in the expanded pictures (Figs. 3, 4).

References

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