Thin films deposition on 304L stainless steel using e-gun technology for medical applications

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Abstract. The purpose of this article is to characterize surface of 304L stainless steel coating by e-gun technology with thin films of Ta₂O₅ and ZnO. ZnO films are used in pharmaceutical industries, in cosmetics and in medicine, grace of their better anticorrosive, antibacterial and photo catalytic properties. Ta₂O₅ films have rapidly evolved in medicine research with the promise of development of new types of biomaterials used in medicine. Ta₂O₅ coatings present an excellent biocompatibility, good dielectric properties, and high corrosion resistance. The biomaterials are realized by novel and special methods to resist of the biological reactions that occur in medical implants (protein adsorption, cell adhesion, cell growth, blood compatibility, etc.). The most important surface properties are physical properties, durability and biocompatibility. The physical properties of a biomaterial that must to be measure, are mechanical strength, permeability and elasticity. Biocompatibility is the response of the biomaterial or of the device to the proteins, cells and organisms action, when the implants are in contact with the biological tissue. The common method to characterize biomaterial surfaces are: contact angles, ESCA (XPS), Auger Electron Spectroscopy, SIMS, FTIR-ATR, STM, SEM and EDAX.

1. Introduction
The 304L stainless steel is used in a large segment of the medical device industry, medical instruments and medical implants (orthopaedic hips and knees, maxillofacial implants, dental implants, etc.).

The major themes of metallic biomaterials are the metallurgical structure, physical and chemical properties, the design, the production and the proper utilization of medical devices [1, 2].

Implantable stainless steels must present a crystal structure as face centered cubic. The chemical composition of 304L stainless steel is: 0.03% max. C, 18-20% Cr, 8-12% Ni, 2% max. Mn, 0.75% Mo, 0.75% Cu, 0.10% max. N, 0.045% max. P, 0.75% max. Si, 0.03% max. S, and <0.01% Fe.

The mechanical properties of 304L stainless steel are: ultimate tensile strength 586MPa, yield strength 241MPa, 159 hardness Vickers, elongation at break 58%, melting point 1399-1421°C, Charpy impact 216 J, modulus of elasticity 193-200GPa.

Grace of lower carbon content of 304L stainless steel, allows minimize/eliminate carbide precipitation during welding process.

This alloy can be used in the “as-welded” state in severe corrosive environments. 316L stainless steel is considered as an alternative of 304L stainless steel in medical applications.

The physical vapours deposition permits to realize corrosion resistant and uniform thin coatings of angstrom order. The vacuum used a pressure of 10⁻⁵ to 10⁻⁶ torr. The most know technologies are: PVD and sputtering. Sputtering advantages are the possibility to sputter deposition of different metal,
alloys and compounds, obtaining various coating microstructures in function of variation pressure. But the disadvantage is the sputter deposition rates lower than thermal evaporation rates. The angle of incidence of particles flux influences the properties film, generally the optical properties.

In figure 1 is presented the physical vapours deposition chamber. The material is vaporized thermally using a heated electrically or an electron beam (e-beam). The vaporization process is realized in a good vacuum that reduces the samples contamination. The source material used for evaporation process is granulated solids at any desired purity. In figure 1, the evaporator source is an e-beam gun head. The quartz crystal microbalance controlled the coating growth, reporting the thickness and evaporation rate. The ion gun is used to increase the density of coating material [3].

![Physical vapours deposition chamber](image)

**Figure 1.** Physical vapours deposition chamber [3].

The thermal evaporation determines the transition from solid to vapour state, using a thermal heating or electron bombardment.

The important parameters of coating technology are: the average speed of the evaporated particles and the angular distribution [3].

The coating film by thermal deposition technology permits the metal with low melt-point, with low purity, the deposition rate is between 1-20 A/s and the temperature range is 1800°C.

The coating film by e-beam technology permits the metal and dielectrics deposition, with high purity, the deposition rate is between 10-100 A/s and the temperature range is 3000°C.

2. **E-gun technology used for oxides coatings**

For the experimental results were prepared 2 disk samples presented a diameter of 25 mm and with a thickness of 2 mm.

The disk samples were obtained by casting using 304L stainless steel. The disk samples are polished and cleaned with ethylic alcohol.

PVD coatings can be realized by evaporation or by sputtering. The physical vapour depositions are used in generally for the optical coatings in the field of ophthalmic and precisions optics coatings.

The PVD technologies are applied for aesthetic, electronic, anticorrosion and mechanical films [4-8].
In thermal evaporation (sublimation under vacuum), the deposition materials consist in the transition from solid to vapour state by means of thermal heating or electron bombardment.

The evaporated material is deposited to the substrate, where grow the thin film. The important parameters of e-gun coating technology are the average speed of the evaporated particles and their angular distribution [4-8].

The process must be realized to the base pressure in the high vacuum range to minimize the number of impact between the evaporate particles and the residual gases in the chamber.

High vacuum permits that the particles have a “mean free path” for the thin film deposition at the substrate.

For evaporation source is used an e-beam gun head, the coating growth controller is a quartz crystal microbalance, which determinates the thickness and evaporation rate.

The ion-gun permits to increase the density of the coating material and to prepare the substrates for deposition.
The 304L stainless steel substrate is cleaning in plasma glow discharges like in figure 2. In figure 3 is presented the PVD evaporation chamber illustration.

The PVD evaporation chamber is equipped with two e-guns, one for Ta$_2$O$_5$ and other for ZnO like in figure 4 and 5. The oxides present sputtering target forms.

**Figure 6.** Disk samples mounted on the dome chamber. **Figure 7.** Ta$_2$O$_5$ film deposition process by e-gun technology.

The disk samples are mounted on the dome chamber like in figure 6. In figure 7 is presented the Ta$_2$O$_5$ deposition process on 304L stainless steel, by e-gun technology, by electrons bombardments at 180°. Other disk sample supported a deposition process with Ta$_2$O$_5$ (50%) and ZnO (50%) by e-gun technology, by electron bombardment at 180°.

**3. SEM and EDAX analysis**

The samples were mounted on the platen of the JSM 7610F Schottky Field Emission scanning electrons microscope and introduced in microscope chamber, like in figure 8.

**Figure 8.** Disk samples introduced in the SEM microscope.
In figure 9 are presented the SEM analysis concerning the Ta$_2$O$_5$ film and can remarked the very grains fine structure of angstrom order (0.6 nm) obtained by e-gun technology.

The roughness obtained is excellent. The EDAX analysis showed the Tantalum oxides and the 304L chemical compounds (Cr, Ni, Fe).

The oxide coating layer obtained by e-gun technology has 5μm thickness. In figure 10 are presented the SEM analysis concerning the film of Ta$_2$O$_5$(50%) and ZnO(50%) and can remarked that the structure is less fine of nanometres order in rapport with tantalum oxide coating(100%) obtained by e-gun technology.

The roughness obtained is very well. The EDAX analysis show the presence of Ta and Zn oxides picks and the 304L chemical compounds (Cr, Ni, Fe).

Figure 9. a) SEM analysis (x 200 000) and b) EDAX analysis of 304L stainless steel disk sample coated with Ta$_2$O$_5$(100%) by e-gun technology.

Figure 10. a) SEM analysis (x 200 000) and b) EDAX analysis of 304L stainless steel disk sample coated with Ta$_2$O$_5$(50%) and ZnO(50%) by e-gun technology.

4. Tribological behavior of 304L stainless steel coating by Ta$_2$O$_5$

In the case of tribological behavior of the carbonitrided steels, don’t exist almost no radial or horizontal displacement in the contact zone. This means that the friction coefficient doesn’t influence the indentation of carbonitrided steels.
The wear test was realized in dry medium, with sapphire spherical indenter using a force F=1N, v=15 cm/s, d=400m, r=7mm. In figure 11, it shows a rapid increase in coefficient of friction values to the specific substrate and it is establishing at the value of 0.9.

In figure 12 shows typical profiles of the wear tracks for 304L stainless steel coating by Ta$_2$O$_5$ and the indication of wear has a depth of ~ 15 microns. Layer of Ta$_2$O$_5$ presents a good wear resistance, using a force of 1N.

5. Conclusions
Both oxides used for coating, Ta$_2$O$_5$ and ZnO have a positive effect results after surgical intervention and present a lower rates of post operator infections, inflammatory reactions or other surgical complications. The SEM analysis shows the ultrafine structure of nanometer order for the both oxides deposed on 304L using PVD technique. EDAX analysis evidences the presence of the oxides on the 304L stainless steel.

Using a force of 1N, can remark a good wear resistance for Ta$_2$O$_5$ layer deposed on 304L by PVD method. This behaviour of the 304L stainless steel coated by Ta$_2$O$_5$ using PVD technology, means that can be used safety in different medical and industrial applications, grace the material don’t present displacements in function of different friction conditions.

6. References
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