



TU Clausthal

**Coating and Surface Treatment for Novel Biomedical
Implants and their Corrosion Resistance**

Doctoral Thesis

(Dissertation)

To be awarded the degree

Doctor of Engineering (Dr.-Ing.)

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Approved by the Faculty of Natural and Materials Science

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Date of oral examination

31. March 2017

Abstract

Metallic biomaterials continue to be used extensively for the fabrication of surgical implants due to their excellent mechanical, physical and biological properties. This work aims to improve the performance of the materials that used as biomedical implants. Enhancement the mechanical properties, corrosion resistance, and biocompatibility, are the main factors that help to success the implant materials in the aggressive environment for a long time. Two types of stainless steel (AISI 316L and AISI 316Ti) and Ti-6AL-4V alloys were used as biomaterials in this study. The surface modification and bulk plastic deformation are the main techniques to maintain a relatively good mechanical properties and biocompatibility within this study. Bulk deformation was processed through rotary swaging (RS) and cold rolling while surface deformation was induced through shot peening (SP) at different parameters. Different heat treatments were done on the swaged bar Ti-6Al-4V to get on duplex and globular microstructure. The correlation between these microstructures and corrosion behavior was studied.

SP was carried out using three sizes of ceramic shots (125– 250, 450 and 850 μm), two Almen intensities (0.22 and 0.28 mmA) and two coverage degrees (100 and 200%). Moreover, hydroxyapatite coating (HA) was applied on the deformed material to improve their corrosion resistance. HA coating was sintering at 500, 600 and 700°C to improvement the adhesive properties.

The properties of HA coating were studied by X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX), scanning electron microscopy (SEM) and standard tensile adhesion test. The phase transformation of the stainless steel due to bulk and surface plastic deformation were recorded by X-ray diffraction spectra. The effect of these treatments namely, cold rolling, rotary swaging and shot peening on the surface roughness, microhardness, induced residual stresses, wettability, corrosion and corrosion fatigue were investigated. The corrosion behavior was studied using potentiodynamic polarization and electrochemical impedance spectroscopy. Ringer's solution was used as an electrolyte for the corrosion and corrosion fatigue tests at 37°C.

The bulk plastic deformation regarding cold rolling and rotary swaging enhanced the mechanical properties. The results showed marked improvement of the fatigue life, of rotary swaged 316L AISI materials, tested in air and Ringer solution. However, the electrochemical behavior after these treatments are not the same for the deformed materials (AISI 316Ti, AISI 316L, and Ti-6Al-4V). Appearing the martensitic phase after cold rolling led to a lower

corrosion resistance of AISI 316Ti while the electrochemical tests showed slightly increase the corrosion resistance of the AISI 316L and Ti-6Al-4V after rotary swaging compared with the as received materials.

On the shot peening, the results showed an increased surface microhardness and induced compressive stresses by increasing the coverage degree and the Almen intensity. The rougher surface after SP improved the wettability in terms of reduced contact angle. Increasing the shot size led to a lower surface roughness and an improved corrosion resistance. However, SP reduces the corrosion resistance compared with the untreated materials. The applied HA coating on the shot-peened surfaces led to a further improvement of the wettability. Applying of HA coating led not only to marked corrosion resistance but also to further improvement of the corrosion fatigue life. The sintering of HA coating refers to a good enhancement of the crystallization of the HA coating sintered at 700°C. The adhesive strength of as-coated (AC) material increased from 8.3 MPa to 12.2, 16.8 and 19.8 MPa after sintering at 500, 600 and 700°C, respectively. The corrosion rate of the as-coated material reduced sharply from 0.405 to 0.094 $\mu\text{A}\cdot\text{cm}^{-2}$ after sintering at 700°C.

The potentiodynamic polarization and electro-impedance spectroscopy techniques showed that the microstructure has significantly affected the corrosion behavior of Ti-6AL-4V alloy. The results proved that the globular microstructure shows high corrosion rate compared to duplex and nanostructured materials.