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Short Communication

Characterization of Thermally Oxidized Ti6Al4V Alloys for Dental Application

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In this work, thermal oxidation processes in the temperature range of 500-800 °C in air for 4 hours were performed on Ti6Al4V medical grade alloys to modify their surface structure and morphology for better wear and corrosion resistance, osseointegration and biocompatibility. Different type and amount of nanostructured phases were obtained as revealed by the X-ray diffration (XRD) technique such as: alumina, anatase and rutile. X'pert high score plus software was used for the calculation of the percentage and crystallite sizes of these phases. Alumina phase exhibits the greater amount of the oxide layers when Ti6Al4V alloys annealed at 500 °C, while rutile was found to be the predominant phase at 800 °C.

Keywords: Titanium alloys, Dental implants, Thermal oxidation, X-ray diffraction, Anatase phase.

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1. INTRODUCTION

Titanium and its alloys like Ti6Al4V are widely used in orthopedic and dental fields as implants due to its good corrosion resistance, biocompatibility and excellent osseointegration [1, 4]. However, the use of these materials was found to be limited by the fact that they exhibit a poor tribological behavior after a long term use as implants [5]. Therefore, several surface modifications were proposed in order to improve the wear and friction properties of Titanium and Ti-alloys, these modifications were carried out by various coating methods such as: thermal oxidation [6], electrochemical anodization [7], hydroxyapatite coating [8] and alkaline treatment with NaOH [9], etc.

Thermal oxidation treatment was performed on pure titanium and Ti6Al4V alloys by several authors at different annealing temperatures and different durations with focus to get only TiO_2 on Ti substrates and a mixture of TiO_2 with Al_2O_3 on Ti6Al4V alloys [6]. They suggest that oxide films formed on the surface of Ti and Ti6Al4V alloys can effectively enhance the wear and corrosion resistance behavior of Ti-based implants.

In this work, we focus our study on the X-ray diffraction investigation of the oxides formed on Ti6Al4V substrates by thermal oxidation at 500, 600, 700 and 800 °C for 4h in air.

2. EXPERIMENTAL PROCEDURE

The samples used in this work are medical grade titanium alloys, grade 5 (Ti6AL4V alloys) with the following chemical composition (wt. %) of 6.3 Al, 3.9 V, 0.185 O, 0.019 C, 0.18 Fe, 0.011 N, Ti balance as evaluated with energy dispersive X-ray spectroscopy (EDS) device attached to the SEM. Prior to the thermal oxidation, the samples provided as rods of 1 cm in diameter and 3 cm in length, were firstly cut into 2 mm thick disks and then mechanically grinded using SiC emery paper (800-2400 grit), followed by wet polishing using diamond slurry. This was followed by sonication in acetone, ethanol and then in deionized water for 5 minutes for each one. Finally, the samples were dried with hot air and nitrogen stream. Thermal oxidation was performed in a muffle furnace at 500, 600, 700 and 800 °C for 4 hours in air, the rate of heating was kept constant at 5°/min in all processes. Philips microscope was used in this work for the optical observations. Oxygen diffusion zone was evaluated by Secondary ion mass spectrometry (SIMS) measurements were carried out by means of an IMS 4F-E7 mass spectrometer (cameca, CRTSE- ALGERIA). Crystallographic structure of the oxide was investigated by X-ray diffraction (XRD,Philips X'PERT PRO) with Cu-Kα radiation (0.15406 nm), with a scan rate of 2°/min. The XRD profiles were compared to standards compiled by the Joint Committee on Powder Diffraction and Standards (JCPDS), with card JCPDS N°: 00-044-1294 for Ti, JCPDS N°: 00-021-1272 for TiO2 (Anatase), JCPDS N°: 01-076-0325 for TiO2 (Rutile) and JCPDS N°: 01-077-2135 for Al2O3 (Alumina).

3. RESULTS AND DISCUSSION

3.1 Optical Obsevations

The optical observations on the surface of thermally oxidized alloys at different temperatures were illustrated in figure 1.

It is clear to note that the samples treated at different temperatures exhibit different coloration on their surfaces: blue for the sample annealed at 500 °C,

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clear brown for the sample treated at 600 °C, brown for the sample treated at 700 °C and dark brown for the sample treated at 800 °C, this can be explained by the difference in the thicknesses and the structures of the

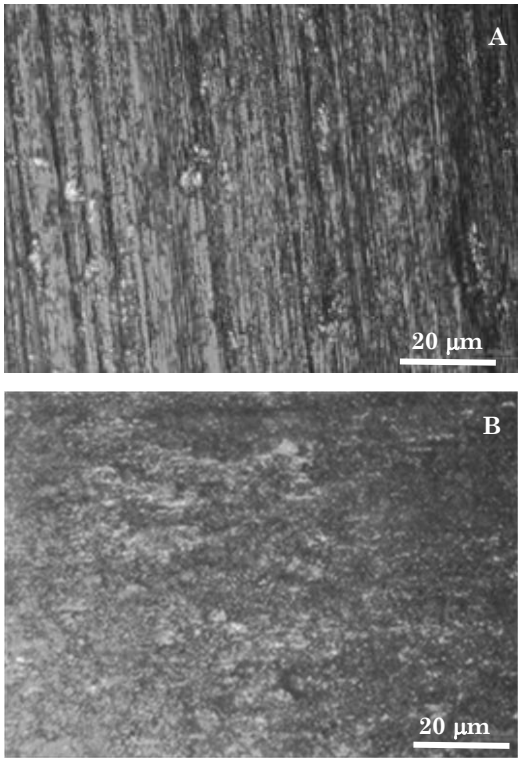

Fig. 1 – Optical micrographs of thermally oxidized Ti6Al4V alloys at A) 500 °C, B) 800 °C

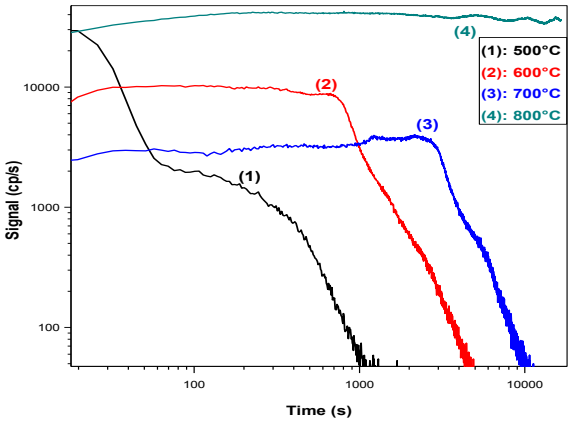

Fig. 2 – SIMS profiles of oxygen (^{16}O) in the oxide layers formed on Ti6Al4V alloy after different thermal treatment for 4h at: (1) 500 °C, (2)600 °C, (3) 700 °C and (4) 800 °C

3.2 Oxygen SIMS Profiles

SIMS profiles of oxygen depicted in figure 3 confirm diffusion of oxygen in Ti6Al4V substrates for all treated samples with different depths depending on the temperature. We can also assess and compare qualitatively the thikness of each oxide layer formed on the surface of treated samples with each other by measuring the length of time of the different plateaus. These plateaus shown in figure 3 correspond to the depths of oxigen diffusion zone in Ti6Al4V substrates so the thicknesses of formed oxide layers. We can observe clearly that the thikness of the oxide layer increases with the temperature.

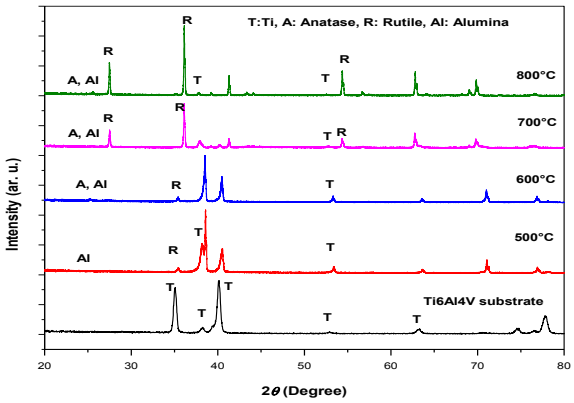

Fig. 3 – XRD patterns of untreated and thermally oxidized Ti6Al4V alloys at different temperatures for 4h in air

oxides formed on the surface of the samples after treatment as will be shown later by oxygen SIMS profiles and XRD in figure 2 and 3 respectively.

3.3 XRD Investigation

The X-ray diffraction patterns of untreated and thermally oxidized Ti6Al4V alloys at 500, 600, 700 and 800 °C for 4h in air were reported in figure 3. From the XRD patterns presented in figure 3, we can clearly note the presence of α/β titanium, anatase, rutile and alumina phases for all thermally treated Ti6Al4V alloys from 500 °C to 800 °C without exception but with different amounts as shown in figure 4.

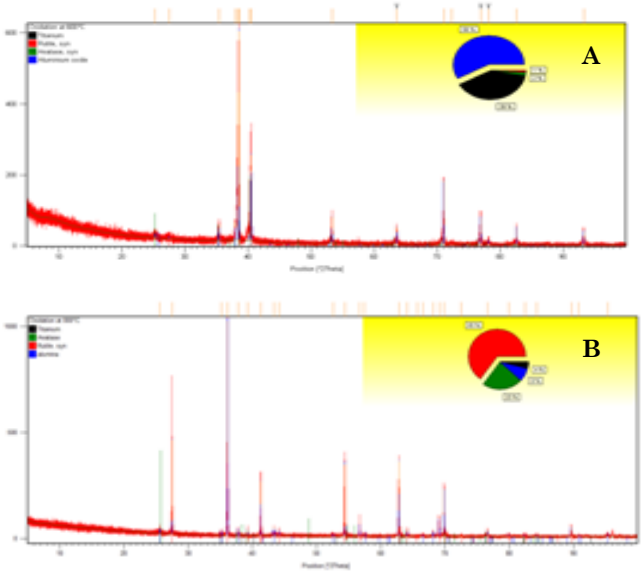

Fig. 4 – Rietveld analysis of the present phases (%) in thermally treated Ti6Al4V alloys at: A) 600 °C and B) 800 °C

The XRD peaks at 35.41°, 38.58 and 40.52° were corresponding to $\alpha + \beta$ titanium phase. Since the depth of penetration of Cu-kα radiation is in the range of 10-20 µm, the presence of α/β-Ti peaks in the XRD patterns of treated Ti6Al4V alloys at the different

annealing temperatures with thinner oxide layers is quite evident as reported by Kumar et al. [10]. Alumina is the predominent phase with 97 % in the oxide layer formed at 500 °C as revealed by Rietveld analysis illustrated in figue 4, followed by titanium and rutile with 2 and 1 % respectively. At 600 °C, the oxide layer contains 58 % of alumina, 39% of titanium, 2 % of anatase and 1 % of rutile. Anatase phase reaches 29 % in the oxide layer formed at 700 °C with 25 % for rutile. At 800 °C the rutile becomes the predominent phase with 65 %.

4. CONCLUSION

Thermal oxidation treatment of Ti6Al4V alloys at 500, 600, 700 and 800 °C for 4h in air for dental application was investigated by the x-ray diffraction method using PANalytical X'pert high score plus software. The study leads to the following conclusions:

– The thickness of the oxide layers increases with the increasing of the treatment temperature.

– The oxide layers were containing a mixture of oxides like alumina (Al_2O_3), anatase TiO_2 and rutile TiO_2 with different amounts depending on treatment temperature.

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