FORMATION OF ON APATITE LAYER ON Ti-30Ta AFTER CHEMICAL TREATMENT

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Abstract. *The purpose of this work was evalutes bioactivity of Ti-30Ta after processing and chemical treatment surface. Ingots were obtained from titanium and tantalum by using an arc-melting furnace. They were submitted to heat treatment at 1100ºC for one hour, cooled in water, cold worked by swaging and turning on a CNC lathe in order to achieve microstructure and morphology next to dental implants. Samples were ultrasonically cleaned with distilled water, acetone and air dried prior being treatment surface. They were immersed in NaOH aqueous solution with 0.2 – 0.5M at 60ºC for 24 hourss, washed with distilled water and dried at 40ºC for 24h. Then, Ti-30Ta discs were immersed in SBF (Simulated Body Fluid) for 7 days to form an apatite layer on the surface.The surfaces were characterized by scanning electron microscope. Contact angle measurements were carried out in order to evaluate the wettability of surface allo. Analysis indicates that the surface was covered with a thin oxide layer after NaOH treatment. Results showed that a bioactive Ti-30Ta alloy can be obtained by chemical treatment.*

Keywords*: titanium alloys, roughness, chemical treatments*

1. INTRODUCTION

Tantalum metal is known to be excellent in fracture toughness and workability, this metal exhibited high malleability, high ductility and high corrosion resistance. It is clinically used as sutures, plates, and a component of titanium alloys in orthopedic and dental fields (Miyazaki *et al*, 2000).

Bioactivity of an artificial material can be assessed in vitro by examining its apatite-forming ability in SBF. The formation of the apatite layer can be reproduced on the surface of bioactive materials in an acellular simulated body fluid (SBF) with ion concentrations nearly equal to those of the human blood plasma (Kokubo *et al*, 1990).

Miyazaki *et al*, (1997) showed that commercially available pure tantalum metal formed apatite on its surface in SBF, but soaking period was as long as 4 weeks. Apatite formation could be observed within 1 week on the tantalum metal which was previously subjected in 0.5 M aqueous solution NaOH at 60°C for 24 h. This proposed that the treatment gave higher potential on bioactivity to pure tantalum metal (Miyazaki *et al*, 2001).

Zhou et al. (2004) evaluated mechanical properties of the binary Ti-Ta alloys with Ta contents varied from 10 to 80% w.. Ti-30%Ta and 70%Ta alloys have much lower modulus and a good combination of high strength and low modulus among all the studied Ti-Ta alloys.

In the present work, bioactivity of experimental alloy Ti-30Ta was evaluated after alkaline treatment and soaking in SBF to form bonelike apatite. Two types NaOH aqueous solutions were used and their influence on CaP coating formation was evaluated.

2. MATERIALS AND METHODS

2.1 Materials

The Ti-30Ta alloy was produced from sheets of commercially pure titanium (99.9%) and tantalum (99.9%). Melting was realized in an arc melting furnace under an argon atmosphere (Fig.1). The ingots were then homogenized under vacuum at 1100ºC for 86.4 ks to eliminate chemical segregation. The resulting samples were finally cold-worked by swaging, producing a 13 mm rod.

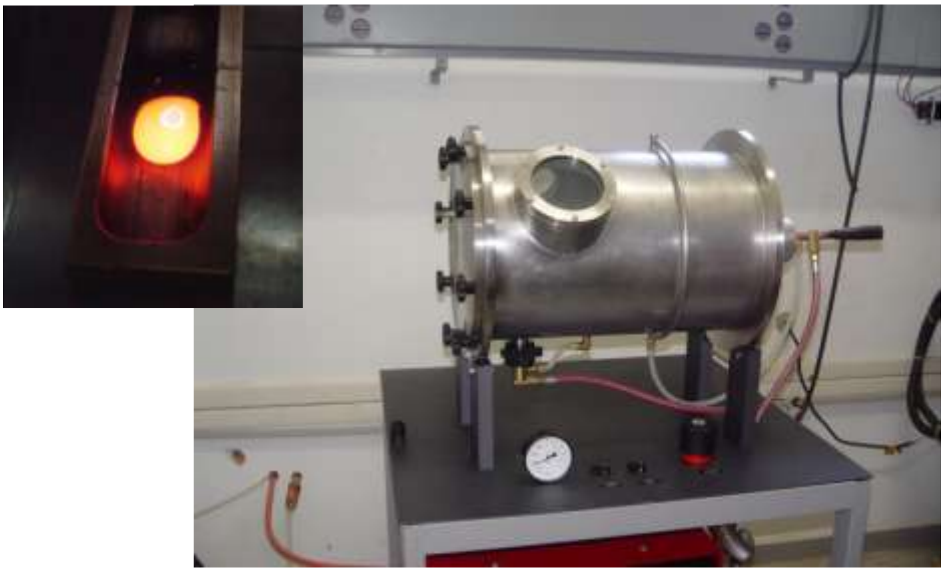

Figure 1 – Arc melting furnace (argon atmosphere)

Bars of this alloy were machined using a CNC lathe ZIL(CENTUR 30S, ROMY, Brazil) with a rotation speed of 1000 rpm to obtain grooved surfaces. Samples were prepared by cutting out discs (10 mm in diameter and 4 mm in thickness). Media roughness (Ra) was measured with a roughness meter (2.5 µm). These samples were ultrasonically cleaned with distilled water and acetone and air-dried prior to surface treatment. Machined samples were used as control group and were not subjected to further surface treatment. Samples were then divided in two groups according to alkaline surface treatment: Group I (0.2 M NaOH) and Group II (0.5 M NaOH).

They were immersed in NaOH aqueous solution with 0.2 – 0.5M at 60ºC for 24 hours at 60ºC for 24 hours , washed with distilled water, and dried at 40ºC for 24 h using a methodology proposed by Wei et al. (2002).

2.2 Biomimetic coating

Ti-30Ta discs were immersed in SBF (Simulated Body Fluid) for 7 days to form an apatite layer on the sample surface. The SBF solution composition proposed by Barrère et al. (2002) was used in this study. SBF with an ionic concentration approximately 5.0 times greater than blood plasma was prepared by dissolving reagent grade NaCl, $MgCl_2.6H_2O$, $CaCl_2.2H_2O$, Na_2HPO_4, and $NaHCO_3$ in distilled water and buffering to pH 7.4 at 36.5°C. The solution was refreshed every second day.

The sample surfaces (after NaOH treatments and soaking in SBF) were examined using a scanning electron microscope (SEM, LEO 1450 VP, Zeiss, Germany). The hydrophilicity of the surfaces was evaluated by contact angle analysis using the sessile drop method.

3. RESULTS AND DISCUSSION

Figure 2 shows scanning electron micrographs of the surfaces of samples after alkaline treatment (Fig 2a and Fig 2c) for two NaOH aqueous solutions (0.2M and 0.5M) and after immersion in SBF for one week posterior these treatments (Fig. 2b and 2d).

It can be observed that no film was formed for 0.2M NaOH (Fig. 2a) and thick layer of Ca-P with poor adhesion was obtained. Also, it can be observed cracks and discontinuous layer in micrograph (Fig.2b). Chen et al. (2003) observed that cracks occurs due differences in thermal coefficient of expansion between the substrate and surface layer. However, Jalota et al. (2006) reported that drying cracks are expected when coating cover the entire substrate surface.

For other solution a discontinuous film was formed after alkaline treatment. After SBF soaking for 7 days (Fig. 1d), surface was covered by a apatite thickness layer

According to Feng *et al*. (1999), the desired composition and thickness of the coatings can be controlled by adjusting the conditions of NaOH-treatment and immersion. An increase of the NaOH concentration, NaOH-treatment time and NaOH solution temperature is favorable for Ca-P crystals to grow on Ti.

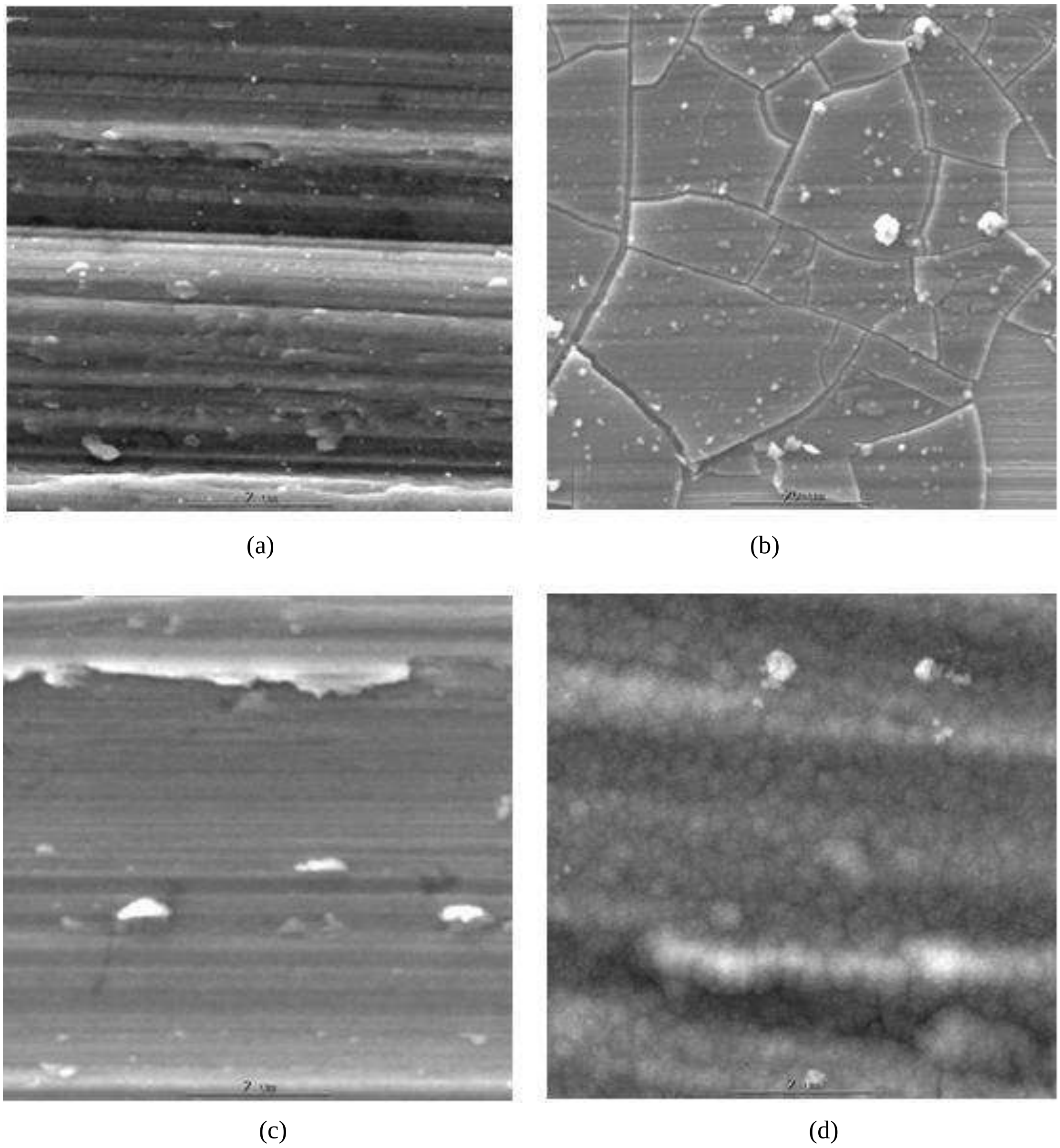

Figure 2 – SEM photographs of the surfaces for Ti-30Ta alloy : (a) treated with NaOH aqueous solutions at 60 °C for 24 h (0.2 M); and (b) after imersion in SBF for 7 days; (c) treated with NaOH aqueous solutions at 60 °C for 24 h (0.5 M) and (c) after immersion in SBF for 7 days

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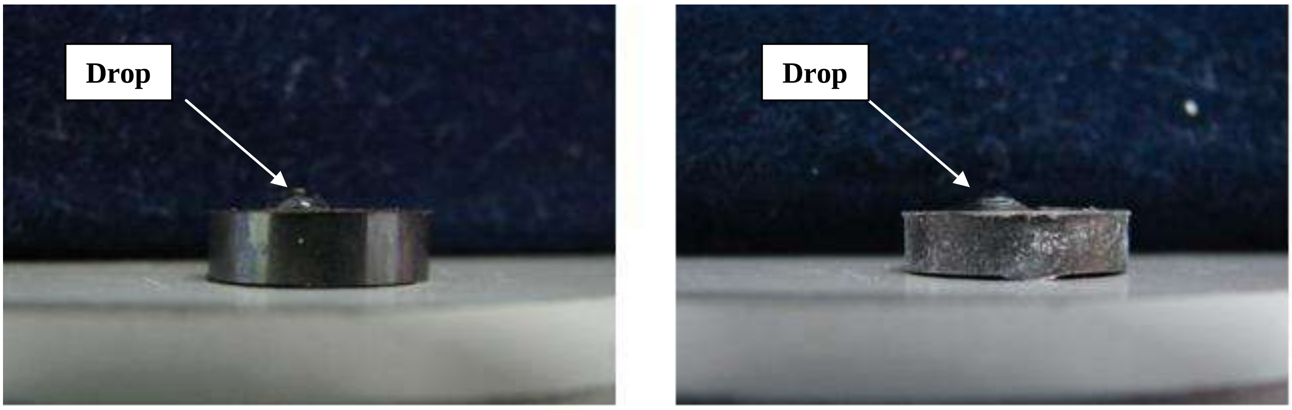
Contact angle analysis was very important to confirm our findings (Table 1). Figure 3 shows image of the water drop on the sample of Ti-30Ta after alkaline treatment and image of the water drop after growth of apatite on surfaces.

It was concluded that a greater concentration of NaOH solution results in formation of apatite and turned possible a smaller contact angle. Surfaces with small contact angle have greater hydrophilicity, which is important for the process of osseointegration.

Table 1 - Measures of contact angle

Treatments	Contact Angle	
	Group I	Group II
NaOH	60.07	54.64
NaOH + SBF (7 days)	22.01	13.32



(a) (b)

Figure 3 - Water drops on different surfaces: (a) after alkaline treatment; (b) after SBF soaking (Group II)

4. CONCLUSIONS

In this work was possible concluded that hydrophilic surface was obtained after surface treatment due apatite growth. However, new researches have been realized with new NaOH aqueous solutions (5M and 10M) to increased hydrophilicity.

5. ACKNOWLEDGEMENTS

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