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TI30TA ALLOY FATIGUE STRENGTH AFTER ALKALINE AND HEAT TREATMENT – BIOMEDICAL APPLICATIONS

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Abstract. *In the present study the fatigue strength of the experimental alloy Ti30Ta was evaluated after the surface modification using alkaline and heat treatment. The alloy was obtained from the fusion of the elements in a voltaic arc furnace with inert atmosphere (argon gas), forged and heat treated. The specimens were machined in accordance with ASTM E466 for the fatigue test. Surface modification was performed by alkaline treatment in 1.5 M NaOH solution and heat treatment. The fatigue test was performed under modified surface conditions and substrate. The characterization of the alloy used techniques of optical microscopy, scanning electron microscopy, X-ray diffraction, roughness and contact angle. The results suggest that the surface modification did not influence the mechanical performance of the Ti30Ta alloy.*

Keywords: *fatigue, titanium alloys, alkaline treatment, mechanical properties, biomaterials*

1. INTRODUCTION

One of the alternatives to improve the properties of titanium and its alloys in the use in implants has been the superficial modification through the alkaline and heat treatment (Capellato et al., 2013). This surface treatment is a chemical method that results in the formation of porous layers bioactive and nanostructured that, when in contact with body fluids, is able to stimulate the formation of biologically active apatite (Escada et al., 2010).

Although the benefits are large from a biological point of view, the surface modification can generate various imperfections, such as notches and pores that consequently can lead to premature failure of the implant under cyclic loading (Oliveira et al., 2017). Thus, the fatigue behavior of a metal material can be influenced by surface modification. The increase of roughness on the surface can modify mechanical performance, reducing structural integrity and fatigue strength, accelerating the initial crack growth stage (Claros et al., 2016).

Fatigue is an important cause of material failure and can be very observed in titanium and its alloys. Several studies have been carried out with new titanium alloys in order to develop new materials for implantation with better mechanical properties (Hussein et al., 2016). In particular, the Ti30Ta alloy presents mechanical properties closer to living tissue than commercially used metal materials such as low modulus of elasticity, higher mechanical strength and superior biocompatibility (Capellato et al., 2012). Given the above, the objective of this study is to verify the influence of the surface modification through alkaline and heat treatment in the fatigue strength of experimental Ti30Ta alloy.

2. EXPERIMENTAL PROCEDURE

2.1 Processing of the alloy

The Ti30Ta alloy was obtained from commercially pure titanium (grade 2) and tantalum with 99.9% purity. These elements were melting in an arc voltaic furnace with argon atmosphere. The ingots were melted at least ten times to ensure homogeneity of the composition. Heat treatment of homogenization was carried out at 1000 ° C for 24 hours, followed by heat treatment of solubilization at 950 ° C for 2 hours. They were cold worked by the rotary swage and solubilized again at 950 ° C for 2 hours.

Ti30Ta alloy microstructure was evaluated in an optical microscope (Axio Imager Z2m Zeiss). For the metallography analysis, the samples were grinded with silicon carbide sandpaper #100 to #1500, polished with colloidal silica and 5% oxalic acid and etching was carried out in 5 ml HF solution, 30 ml HNO_3 and 65 ml H_2O.

2.2 Alkaline and heat treatment

Before surface treatment, the surfaces were previously grinded (#100) and cleaned in ultrasonic bath with distilled water and acetone for 30 minutes. For the alkaline treatment, the specimens were immersed in 1.5M NaOH solution at 60 ° C for 24 hours. Then, the specimens were washed in distilled water and oven dried at 40 °C for 24 hours. The heat treatment was carried out at 300 °C for 1 hour, followed by slow cooling to room temperature.

2.3 Fatigue strength test

Fatigue test specimens were machined according to ASTM E466, as shown in Fig. 1.

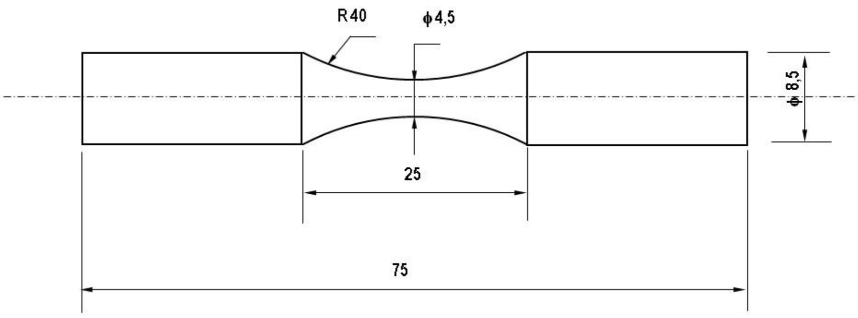


Figure 1. Geometry and dimensions (mm) of the fatigue test specimens

Axial fatigue tests were conducted according to ASTM E466 using a servo-hydraulic equipment (Instron). The tests were performed at room temperature using a frequency of 10 Hz and loading ratio R = 0.1. In the present study, the stress was established from previous alloy characterization works carried out by Bortolini Júnior (2016). Table 1 shows the main mechanical tensile properties of the Ti30Ta alloy.

Table 1. Ti30Ta alloy mechanical properties

	Tensile strength limit (MPa)	Yield strength (MPa)	Modulus of elasticity (GPa)	Strain (%)
Ti30Ta	528	357	48	13,2

Source: Bortolini Júnior (2016)

The value of the stress used in the tests was based on the yield strength. Fatigue strength was defined as the maximum stress at which the specimen doesn't fail in 10^6 cycles.

2.4 Surface Characterization

The X-rays diffraction analysis was performed using a PANalytical diffractometer (Empyrean) operating at 40 kV and 25 mA using CuKα radiation.

The wettability was evaluated by contact angle measurement using the sessile drop method. Measurements were done depositing a drop of 10 µl of deionized water on the surface of the samples using an automated goniometer (Kruss). During the process, the droplet shape was monitored by a digital camera and the contact angle measurements were obtained from the images.

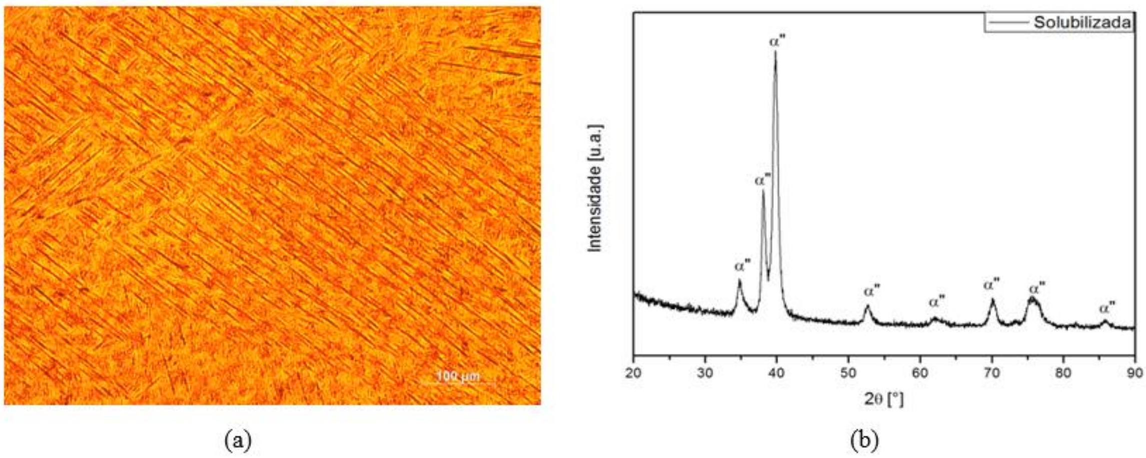
To estimate the superficial changes of the Ti30Ta alloy induced by the surface modification with the alkaline and heat treatment, roughness measurements were carried out. All the specimens were evaluated using surface roughmeter (Mahr - Marsurf M300). The mean roughness (Ra) were recorded at three different points and determined by the simple arithmetic average of the values.

The surface morphology before and after surface treatment was examined using scanning electron microscope Zeiss - EVO LS 15 and FEI - Magellan 400 L.

3. RESULTS AND DISCUSSIONS

3.1 Alloy characterization

The sequence used for processing of the experimental alloy Ti30Ta involves the follow steps: melting, homogenization heat treatment, cold forging and solubilization heat treatment. The final microstructure of the alloy can be observed in the Fig. 2 (a). The image reveals the presence of orthorhombic α" phase, evidenced by thin, parallel structures, similar to a needle. Zhou et al. (2004) verified the formation of martensite or α”-phase in Ti30Ta alloys as a result of the decomposition of the β phase when they were cooled rapidly or underwent mechanical deformation.



Source: Author

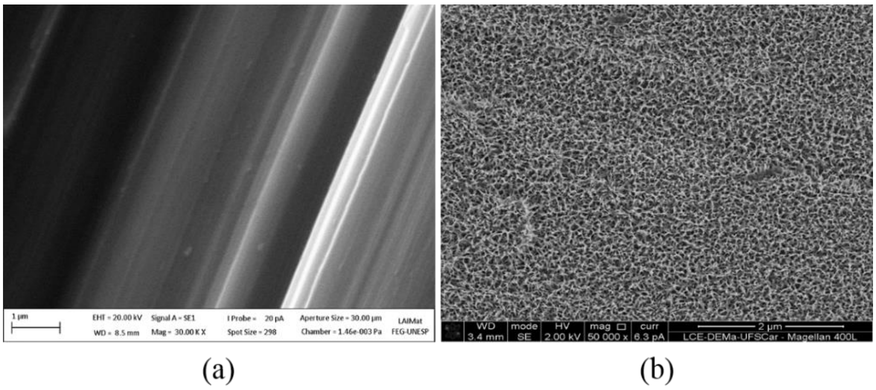
Figure 2. (a) Ti30Ta alloy optical micrograph and (b) X-rays diffraction spectrum after processing

The X-rays diffraction of the Ti30Ta alloy confirmed the results observed in the microscopy analysis. The spectrum of the Figure 2 (b) showed characteristic α”-phase peaks formed during solubilization at 950 °C for 2 hours. According to Zhou et al. (2007), the TiTa system alloys with tantalum content of 30%w lead to the retention of α-phase at room temperature. These results were also verified in studies of the Ti30Ta alloy performed by Konatu et al. (2016).

3.2 Surface modification characterization

The surface properties exhibit significant influence in determining the success or failure of an implant, since they interfere in the cell proliferation and in the formation of biologically active apatite (Cordeiro and Barão, 2017).

In Figure 4, micrographs obtained by Scanning Electron Microscopy show the surface characteristics of the Ti30Ta alloy before and after the surface. The images indicate the formation of a porous film on the alloy surface after alkaline and heat treatment with a small change in topography, Figure 3 (b). For the substrate, Figure 3 (a), only grooves resulting from grinding can be noted.



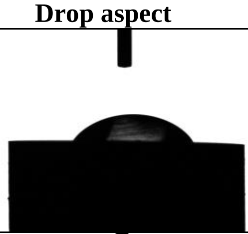
Source: Author

Figure 3. Micrographs of the SEM (a) substrate and (b) after alkaline and heat treatment

According to Asri et al. (2017), the appearance of a porous layer of sodium tantalate after chemical treatment with NaOH increases the surface roughness and benefits its adhesion to the bone tissue.

Table 2 shows the roughness measurements verified on the Ti30Ta alloy surface before and after alkaline and heat treatment. The substrate (control group) exhibited mean roughness measurements of 1.31 μm. The modified surface showed more roughness than initially, with a measurement of 1.44 μm. Or and Wang (2016) reports that increase of the surface roughness stimulates cellular activity and favors apatite nucleation, which in greater quantity provides rapid biological fixation of the implants.

Table 2. Roughness and contact angle measurements of the Ti30Ta alloy

Sample	**Roughness (μm)**	**Contact angle (°)**	**Drop aspect**
Substrate (control)	1,31	61,0 ± 0,97	
Alkaline and heat treatment	1,44	21,1 ± 0,68	

Source: Author

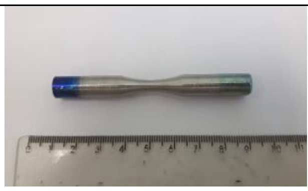
Contact angle measurement is used to determine the wettability. A surface characterized by an angle of less than 90° is denomined hydrophilic, and those with an angle greater than 90°, hydrophobic. For biomedical application where cell growth is desired, the materials should preferably be more hydrophilic which this favors the interaction between the tissue and the implant (Escada et al., 2010)

As indicated in Table 2, the alkaline and thermal treatment decreases the contact angle value making the surface of the Ti30Ta alloy more hydrophilic. The contact angle measurements after alkaline and thermal treatment were similar to those found by Carvalho (2013).

3.3 Fatigue strength analysis

With the fatigue test performance, it was possible to evaluate the interference of the surface modification in the resistance of the Ti30Ta alloy. The results obtained are shown in Table 3.

Table 3. Ti30Ta experimental alloy fatigue characterization

Specimen	Stress [MPa]	Number of cycles
 Substrate	340	10^6
 Alkaline and heat treatmet	340	10^6

Source: Author

The equivalence of the obtained data indicates that the fatigue life at this stress level is identical within the surface conditions analyzed. These results suggest that the surface layer modified by alkaline and heat treatment with the previously specified parameters has little influence on the fatigue behavior of the Ti30Ta alloy, especially in the high cycle fatigue regime where the crack nucleation is very sensitive to surface condition (Campanelli et al., 2017).

4. CONCLUSION

From this study, it was possible to conclude that fatigue test results suggests that the surface modification has not affected fatigue life. The same behavior was observed in both conditions analyzed. Thus, Ti30Ta alloy exhibited potential for biomedical applications.

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6. RESPONSIBILITY NOTICE

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