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BEHAVIOR OF Ti-30Nb-5Mo ALLOY AFTER COLD ROLLING AND AGING

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Abstract. *This work present result of mechanical and microstructural characterization of the Ti-30Nb-5Mo alloy deformed by cold rolling and submitted to aging treatment. The β -type titanium alloys are composed of non-toxic and biocompatible elements and present high hardness, good corrosion resistance and low Young's modulus, close to the human bone. Addition of β -stabilizing elements, such Nb and Mo, allows stabilization of the β -phase in titanium alloys. Samples containing 50 g of the Ti-30Nb-5Mo alloy (wt%) were obtained by electric arc melting, solutionized at 1000°C for 8h in controlled atmosphere followed by quenching. In sequence samples were aged in different temperatures and times to study behavior of this alloy in different conditions. Characterization of the samples was carried out using light optical microscopy, measurements of Vickers hardness and measurements of Young's modulus by ultrasonic method. Main results indicate a variation in Young's modulus and in the Vickers hardness in the aged condition that is related to the phases precipitation during aging heat treatment.*

Keywords: *Cold rolling, Young's modulus, aging treatment.*

1. INTRODUCTION

Titanium alloys has a variety of application in the aerospace and biomedical areas, present a good combination of mechanical and physical properties, including high strength-weight ratio and excellent corrosion resistance.

Titanium alloys can be divided into three groups: α , $\alpha+\beta$ and β alloys. The Ti-6Al-4V alloy is the main representative of the $\alpha+\beta$ alloys and for a long time it has being extensively used in the production of orthopedic implants, replacing commercially pure titanium. This alloy has interesting set of properties, including high mechanical strength. New alloys are being developed for the substitution of vanadium in the Ti-6Al-4V alloy, because it is considered a toxic element and can cause damage to the health of the implanted person (Eisenbarth *et al.*, 2004). Aluminum can be harmful, causing local inflammations (Zaff *et al.*, 2001) and may also be related to disorders in the neurological system (Silva *et al.*, 2004).

β -type alloys are composed of non-toxic and biocompatible materials such as Nb, also the modulus of elasticity is close to the human bone which range from 20 to 40 GPa (Tane *et al.*, 2008). Materials as the stainless steels and the Cr-Co-Mo alloys are also used in the production of orthopedic implant and may present Young's modulus close to 200 GPa and superior values to 240 GPa, respectively. When are used materials with Young's modulus superior to the human bone, it happens insufficient transfer of the tension could generate degradation of the bone and consequently osteoporosis (Tarr *et al.*, 1983).

One way to optimize the mechanical properties of the titanium alloys is to perform the aging thermal treatment which promotes the precipitation of reinforcement phases, causing an increase in the mechanical strength of the material.

The focus of this study is β titanium alloy which have applications in biomedical industry. In this context, this work presents the main results of the mechanical and microstructural characterization of the Ti-30Nb-5Mo alloy (wt%), deformed by cold rolling and then aged.

2. EXPERIMENTAL PROCEDURE

Starting material consists of 50g ingots each and was obtained by electric arc melting furnace with controlled atmosphere. After melting, the ingots were encapsulated in quartz tubes under vacuum and then submitted to the solutioning heat treatment, which consists of heating the material at a temperature of 1,000°C for 8 hours, followed by quenching in water and simultaneously breaking the quartz. After this heat treatment, samples of Ti-30Nb-5Mo alloy (wt%) were deformed by cold rolling up to 86% of thickness reduction, without intermediary annealing.

Samples cold-rolled were sealed in quartz tubes under vacuum are aged in temperatures of 260 and 400°C, the times considered were 1, 15, 30, 60, 120 and 240 min, samples were cooled inside the quartz tubes in the air.

Microstructural analysis was performed using light optical microscopy. The mechanical behavior was evaluated by the Vickers hardness test and the resulting values represent the average of 10 measurements, using a 98 gf applied for 20s, and measurements of Young's modulus using ultrasonic method.

3. RESULTS AND DISCUSSION

In this section are presented the main results of characterization of Ti-30Nb-5Mo alloy.

3.1 Starting condition

In the solutionized condition it was verified that the material presented a grain size of 390 μm , and the elements, Nb and Mo, contributed to the stabilization of β -phase. According to Lu and co-workers (2013), Mo is a strong β -phase stabilizer, in their study they found that in titanium alloys with 10 wt% Mo that are solutionized, β -phase only when the identified by X-ray diffraction technique, being possible to observe a presence of small peaks of the α " phase.

Young's modulus obtained by ultrasonic method was equal to 69 ± 2 GPa and Vickers hardness was de 182 ± 5 HV. Figure 1 show the microstructure of a sample after solutioning heat treatment.

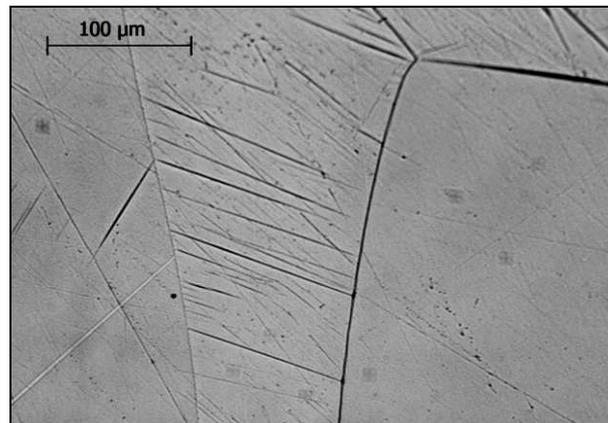


Figure 1 – Microstructure of a sample of the Ti-30Nb-5Mo alloy in solutioning condition.

3.2 Deformed condition

The studied alloy contains β -stabilizing elements (Nb and Mo), and these elements contributed to the stabilization of the β -phase, which resulted in ductility of the material and in a plastic deformation up to 86% of reduction in thickness by cold rolling, without intermediary annealing. This result was obtained in multiple passes, being the maximum possible reduction for this alloy.

According to Padilha and Siciliano Jr. (2005), during the cold rolling process, the grains that are initially equiaxial, become more elongated in relation to the rolling direction, this behavior can be observed in the Figure 2.

Cold-rolled sample presented shear bands that form approximately 40° with the rolling direction (Figure 2). Humphreys and Hatherly (2004), affirm that these bands are a result of plastic instability during deformation.

In deformed condition, the Young's modulus increased to 79 ± 4 GPa. This fact, in according with Matsumoto and co-workers (2007), can be a consequence of crystallographic anisotropy of Young's modulus of α' phase, followed by texture development.

It was observed also an increase in the Vickers hardness in deformed samples, being equal to 239 ± 4 HV. This behavior is related with increase of dislocations during the plastic deformation, because only a small amount of energy used during plastic deformation is stored in material under the form of dislocations and point defects (Humphreys and Hatherly, 2004). With increase of dislocations amount in material, there is an increase in the mechanical resistance and hardness with a decrease of ductility.

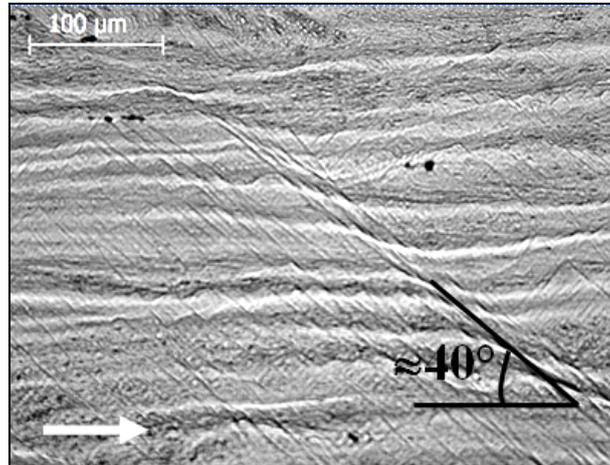


Figure 2 – Microstructure of the Ti-30Nb-5Mo alloy cold-rolled showing shear bands with approximate angle of 40° with the rolling direction indicates by the arrow.

3.3 Aged condition

Samples were aged after cold rolling process and some specimens presented small recrystallized grains, being observed only in longer times of aging. This behavior was observed for two temperatures of aging studied (Figure 3). It is also noticed in the Figure 3a the presence of several shear bands.

The objective of cold plastic deformation was to obtain a metastable hardened microstructure to accelerate the response to the aging heat treatment. Cold-deformed and aged samples contain a higher volumetric fraction of the ω phase when compared to samples that were solutionized and later aged (Froes *et al.*, 1980).

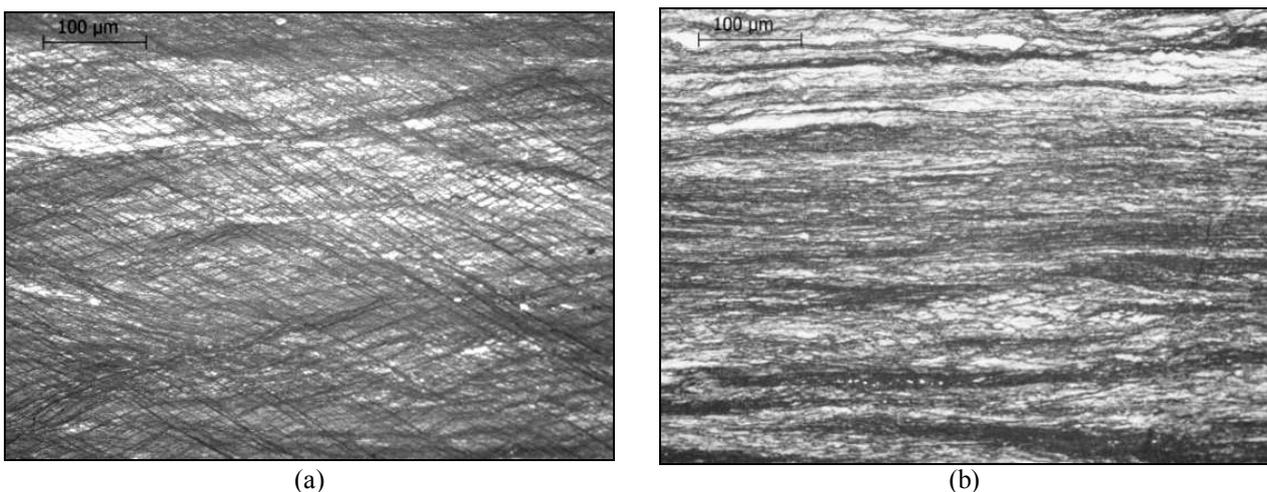


Figure 3 – Microstructure of the Ti-30Nb-5Mo alloy deformed and then aged at: (a) $260^\circ\text{C}/120$ min; (b) $400^\circ\text{C}/120$ min.

In agreement with the established theory on the precipitation of the ω phase in β titanium alloys, the quenching of the starting material from the β field leads to the precipitation of athermal ω phase, with very fine precipitates of a few nanometers in diameter. Aging of these alloys allows for the formation of isothermal ω phase precipitates, with sizes of

even hundreds of nanometers. Isothermal ω phase can be formed at low and intermediates temperatures, and its presence is related to the loss of ductility and increase of hardness (Kim *et al.*, 2006).

Young's modulus in an alloy is mainly a result of the volumetric fraction and Young's modulus of constituent phases and it is not sensitive to the size and morphology of grains. However, tensile strength is sensitive to microstructure. Young's modulus is an intrinsic material property and is a function of inter-atomic forces. Several microstructures can be obtained by different aging treatments, and this is a factor that influences various properties of the alloys. An appropriate aging treatment can promote a great possibility of improving the balance between low Young's modulus and high mechanical strength (Zhou *et al.*, 2004).

While α and ω phase precipitation increases mechanical strength, it is also increases the Young's modulus. Although ω phase leads to a loss of ductility, studies show that the control of the volume fraction of this phase can increase the mechanical strength without considerable loss in ductility (Froes *et al.*, 1980). This metastable phase can act as sites for α phase nucleation during aging heat treatments. As a result, α phase distribution in the microstructure is homogeneous and finely dispersed (Anken and Greene, 1999).

Was verified an increase in Young's modulus and in Vickers hardness in samples of the Ti-30Nb-5Mo after aging heat treatment. This fact can be related to precipitation of ω and α phase that can occur during aging of this alloy. It is also verified that occur stabilization in the values of Vickers hardness and of Young's modulus for superior times to 120 min, for the two aging temperatures studied. Figure 4 shows variation of Young's modulus and Vickers hardness during aging of Ti-30Nb-5Mo alloy.

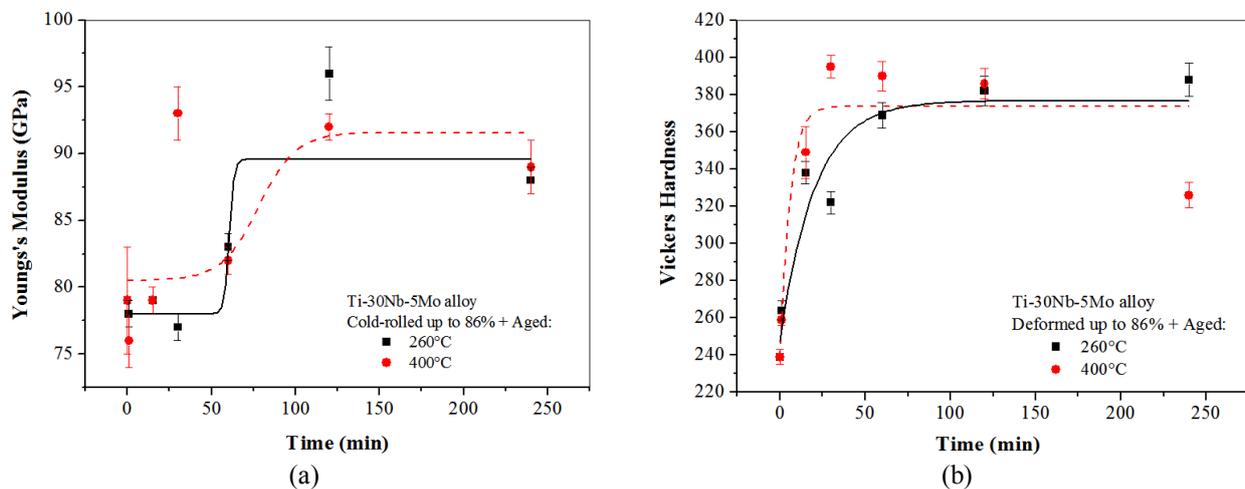


Figure 4 - Variation of mechanical properties during aged of the Ti-30Nb-5Mo alloy: (a) Young's modulus; (b) Vickers hardness.

4. CONCLUSIONS

Main results of this work indicate that after the solutioning heat treatment the material presents a grain size of 390 μm , and the elements, Nb and Mo, contributed to the stabilization of β -phase. After cold rolling the grains of the sample are aligned to the rolling direction and also there is the presence of deformation heterogeneities, as shear bands. In deformed condition was verified an increase in the hardness values, this fact is related to accumulation of defects, mainly dislocations, that occur during the process of cold plastic deformation. In the aged condition was verified a variation in Young's modulus and in the Vickers hardness, that is related to precipitation of different phases during aging heat treatment.

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