MECHANICAL PROPERTIES OF Ti-13Nb-13Zr ALLOY COLD WORKED BY SWAGING AND ROLLING

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Abstract *- Recent studies have been done to achieve biomedical alloys containing non-toxic elements and presenting low elastic moduli. It has been reported that Ti-Nb-Zr alloys rich in* β *phase, specially Ti-13Nb-13Zr, have potential characteristics for substituting conventional materials such as Ti-6Al-4V, stainless steel and Co alloys. The aim of this work was to evaluate the mechanical properties of Ti-13Nb-13Zr alloy submitted to mechanical working processes, swaging and rolling. This alloy was produced from an alternative route of thermomechanical processing which consists in arc melting, solution heat treatment at 1000°C/1h and water quenching, cold rolling or cold swaging (80% of area reduction in both) and in sequence other heat treatment at 900°C/30min and water quenching. Microstructural analyzes of the alloy were performed using Light Microscopy and Scanning Electron Microscopy (SEM). After the last heat treatment the alloy presented an homogeneous martensitic* α' *microstructure. Furthermore, the mechanical properties were evaluated based on tensile and hardness tests. Ultimate tensile strength, yield strength and elastic moduli present no significant difference. The difference observed for the elongation (about 30%) was associated to the anisotropic nature of the process route.*

Keywords: *Ti-Nb-Zr alloys, thermomechanical processing, mechanical properties, biomedical application*

1. Introduction

Titanium and its alloys are still attracting much attention for application as biomedical metallic materials. Their properties, such as relatively low density, high corrosion resistance and satisfactory biocompatibility, are desirable in this respect. One of the most important biomedical titanium alloy has been the Ti-6Al-4V ($\alpha+\beta$) (Geetha *et al,* 2001). However, recently the researches have demonstrated that vanadium causes cytotoxic effects and adverse tissue reactions (Davidson *et al*, 1994), while aluminum is associated with potential neurological disorders (Okazaki, 2001 and Khan *et al*, 1999). In addition, long term experience indicates that the high moduli $\alpha+\beta$ Ti implants transfer insufficient load to adjacent remodeling bone and this results in bone resorption and eventual loosing of prothetic devices (Geetha *et al*, 2004).

From these reasons, attempts were made to develop Ti alloys of different composition to achieve better performance. It has been reported that β rich Ti-Nb-Zr alloys are better substitutes, as these materials possess low modulus and consists of non-toxic elements in it (Niinomi, 1998). According to Davidson *et al* (1992) the Nb present in these alloys, an known β stabilizer, reduce their modulus and enhances the ability of the alloys to harden on subsequent aging. Geetha *et al* (2004) have reported recently about the effect of thermomechanical processing on microstructure evolution of the some Ti-Nb-Zr alloys in order to understand the influence of alloying elements on phase transformations.

Thermomechanical processing is often performed on titanium alloys to attain a desired combination of mechanical properties. According to Sauer and Luetjering (2001), the mechanical properties of high strength β-Ti alloys are very sensitive to variations in processing parameters. Therefore, careful process control and profound knowledge of the influence of processing on microstructure and properties are of significant importance for the manufacturing of β-Ti alloy products of high quality.

One example of β-Ti alloy is Ti-13Nb-13Zr, that was originally developed for medical implant applications, its properties make it attractive option for total hip replacements, orthopedic fixation devices and dental components. Research on this alloy has shown that the mechanical properties can be controlled over a significant range through hot working, heat treatment and cold working (Robare *et al*, 1997). Moreover, this alloy exhibits a low elastic modulus, which is much closer to that of the bone (40 GPa). Hence, it is considered to be a better alternate when compared to the conventional Ti-6Al-4V alloy with modulus of 120 GPa (Geetha *et al*, 2004).

The aim of this work was to evaluate the mechanical properties of Ti-13Nb-13Zr alloy submitted to mechanical working processes, swaging and rolling. Studying the metal forming is important to obtain the material in different forms with possibility of different biomedical applications. Ti-13Nb-13Zr alloy was prepared based on prior studies realized by Schneider (2001), consisting of argon arc melting followed by cold working and heat treatment.

2. Experimental procedure

Ti-13Nb-13Zr (%wt) alloy was produced from commercially pure materials (Ti, Nb and Zr) by arc melting under argon atmosphere in a water–cooled copper hearth, and the melting was repeated eight times to ensure chemical homogeneity. The obtained ingots were solution treated to eliminate the influence of the initial microstructure and to guarantee their homogeneity. For this purpose, they were solution treated for 1h in the β-field at 1000°C (β ST), followed by water quenching. Next stage was the cold-working by swaging and rolling (80% plastic deformation), and in sequence another heat treatment for 30min at 900ºC followed by water quenching (Silva et al, 2004). From these heat-treated materials standard samples for tensile tests, were produced.

Microstructural analyses of the alloy in the as-cast and heat-treated conditions were performed using light microscopy and scanning electron microscopy (SEM). Samples were prepared by standard metallography procedures used for Ti and its alloys and etched with modified Kroll's reagent. A Leica DMIRM light microscope and LEO-ZEISS 1450 VP SEM with OXFORD-LYNK energy dispersive spectrometry (EDS) system were used for the analyses.

Mechanical characterization of the alloy was based on tensile and microhardness tests at room temperature. Tensile tests were carried out in a servo-hydraulic MTS machine to determine the ultimate tensile strength, 0,2% offset yield strength, elongation, and elastic modulus. The tensile specimens were manufactured according to ASTM E8. The dimensions of swaged and rolled specimens are shown in Figs. 1 and 2, respectively. Microhardness tests were carried out in Micromet 2004 equipment, with a load of 100kgf and time of 20 seconds.

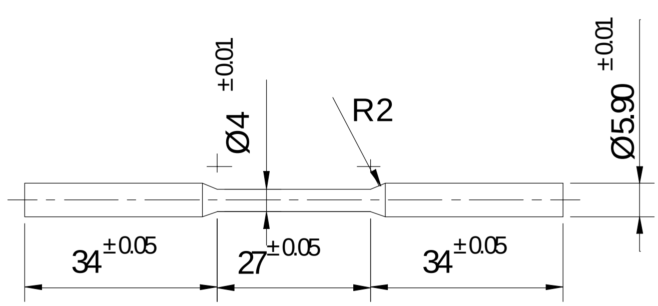


Figure 1 – Geometry and dimensions of the specimens used in this work for swaged alloy.

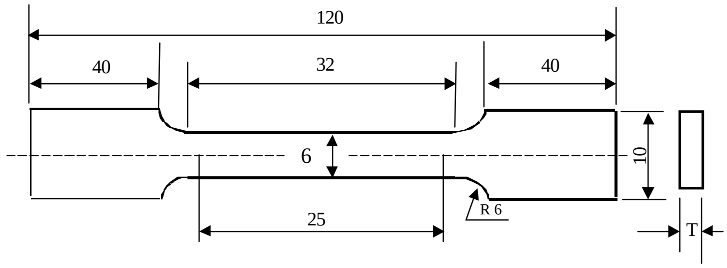


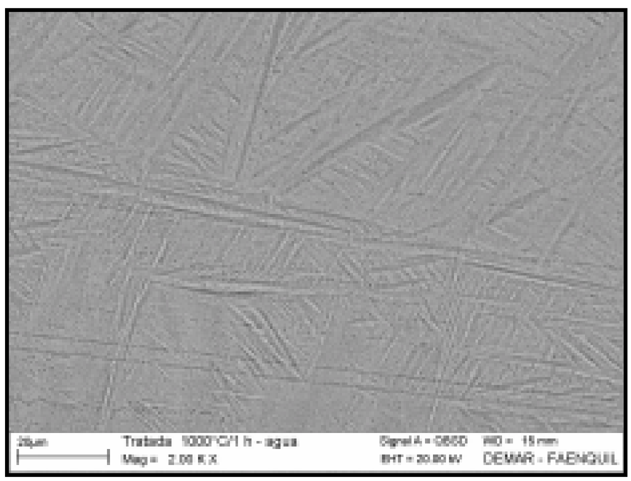
Figure 2 – Geometry and dimensions of the specimens used in this work for rolled alloy.

3. Results and Discussion

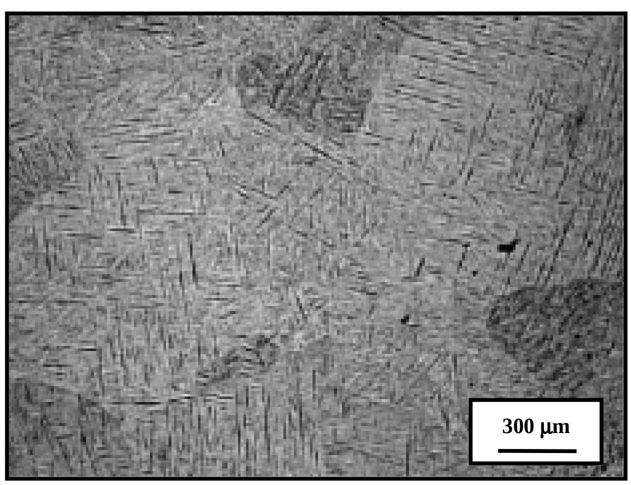
Thermomechanical processing is often performed on biomedical alloys to attain a desired combination of mechanical properties. Therefore, the principal results of this study pertained to the evaluation of microstructure and mechanical behavior of Ti-13Nb-13Zr alloy under cold working by swaging and rolling.

3.1 – Microstructural characterization

The obtained ingots of Ti-13Nb-13Zr alloy were solution treated in the β-field (β ST-1000°C/60min), followed by water quenching (WQ). The micrograph of the β ST alloy shows a single phase, martensitic α′ microstructure (Fig. 3), which allowed the cold working by swaging and rolling. Results of EDS analyses are close to the nominal compositions of the alloy, indicating a microstructure throughout homogenized.



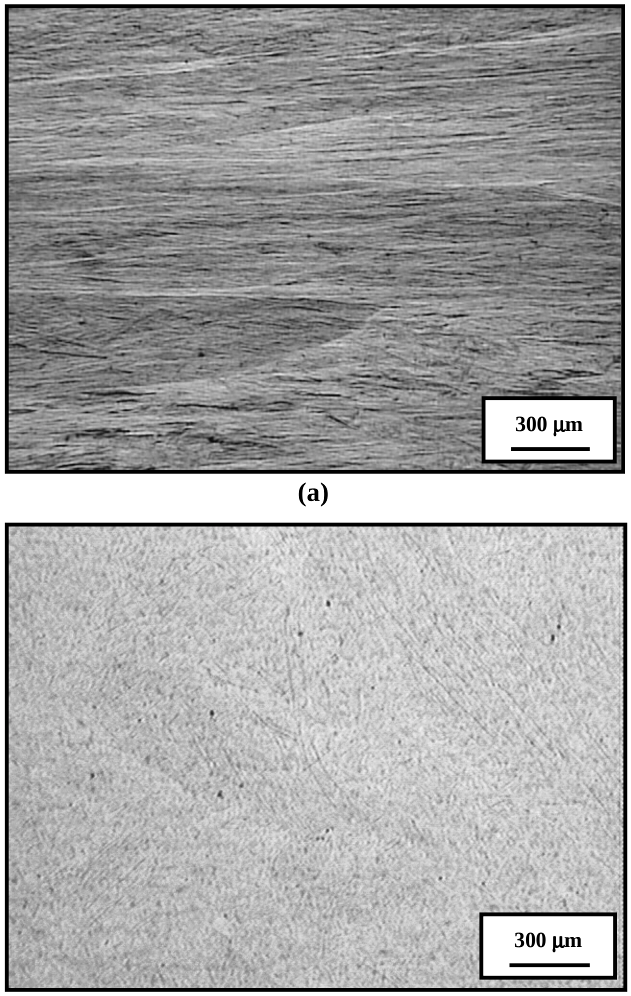
(a)



(b)

Figure 3 –Micrographes of Ti-13Nb-13Zr alloy after solution heat treatment at 1000ºC/60min and water quenched, obtained by: **(a)** SEM and **(b)** light microscopic.

The microstructures of cold-swaged alloy are shown in Fig.4. Elongated grains are observed in the longitudinal section (Fig.4a), which is a typical of a swaged structure. Grain boundaries are not clearly observed in the transversal section while random strain markings can be noted in Fig.4b.



(b)

Figure 4 - Optical micrographs of Ti-13Nb-13Zr alloy after conformation processing by swaging: **(a)** longitudinal section and **(b)** transversal section.

A representative optical micrograph of the microstructures cold-rolled alloy is shown in Fig.5, which shows the anisotropic nature of the uncrystallized grains, with “packets” of grains elongated along the rolling direction. Figure 4a is a representative of ST plane (rolling plane) and Figure 5d is a representative of SL plane, in which grain boundaries and strain markings are orientated perpendicularly and parallel to the rolling direction, respectively. Figures 5b is a representative of LT plane (parallel to the rolling direction).

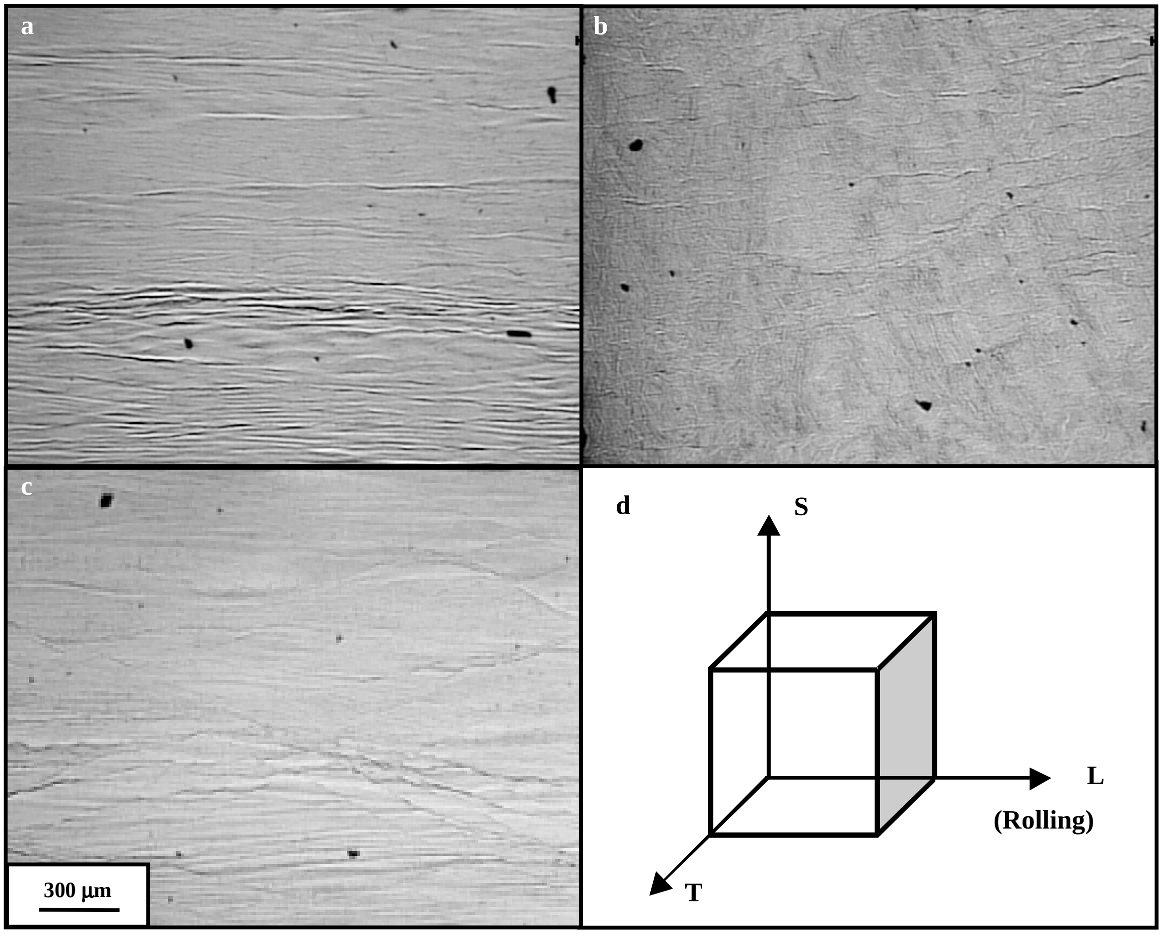
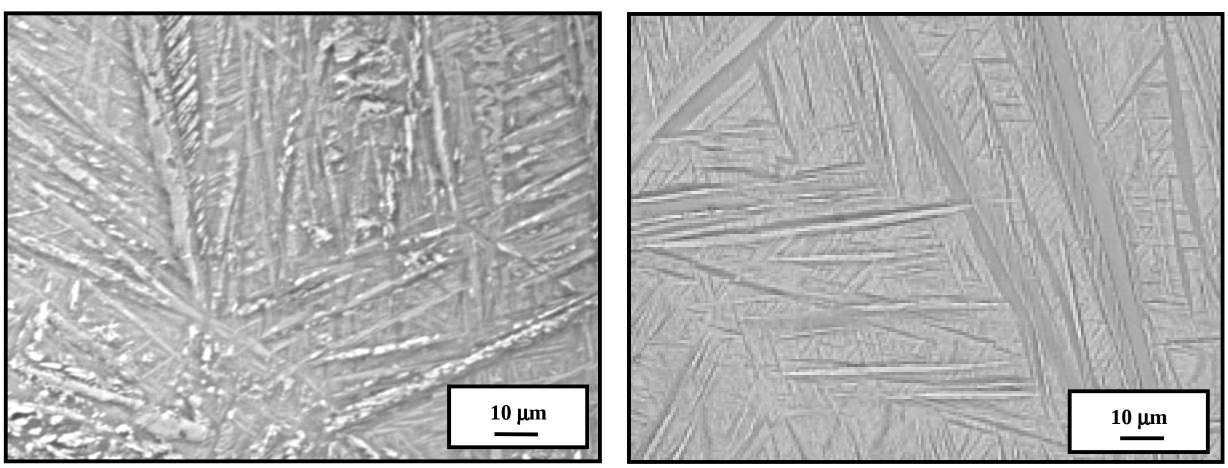


Figure 5 - Optical micrographs of Ti-13Nb-13Zr alloy after conformation processing by rolling in the following planes: **(a)** ST; **(b)** LT; **(c)** SL and **(d)** scheme of planes and directions.

After cold working, the alloy was heat-treated at 900°C/30 min and followed by water quenching to relieve induced stress by cold deformation process. It is noted a complete recrystallization microstructure of the Ti-13Nb-13Zr alloy for both processes. Very fine platelet-type features, that are indicative of martensitic microstructure, are seen inside the recristallized grains for the alloy obtained by both processes (Fig. 6).



(a) **(b)**

Figure 6 – SEM micrographs of Ti-13Nb-13Zr alloy heat treated at 900°C/30min and water quenched after different conformation processing: **(a)** swaging and **(b)** rolling.

3.2 – Mechanical characterization

Tensile stress-strain curves of Ti-13Nb-13Zr alloy swaged and rolled, that were annealed at 900ºC/30min and water quenched (WQ), are shown in Fig. 7. Mechanical properties values from this work are summarized in Tab. 1. It is noticeable that mechanical properties of the alloy processed by swaging and rolling do not present a significant difference, with exception to elongation.

The values of elastic moduli (about 60GPa) are higher than that of the bone (max. 40GPa), they are still smaller compared to the conventional Ti-6Al-4V (about 120GPa). The values of ultimate tensile and yield strengths are in required range for orthopedic applications, about 650 and 500MPa, respectively (Callister, 2002). This is also observed for the elongation, because the minimum value of elongation required for this kind of application is approximately 8%. Moreover, the rolled alloy presents a lower elongation (16%) than the swaged one (21%), and it can be associated to the anisotropic nature of the process route.

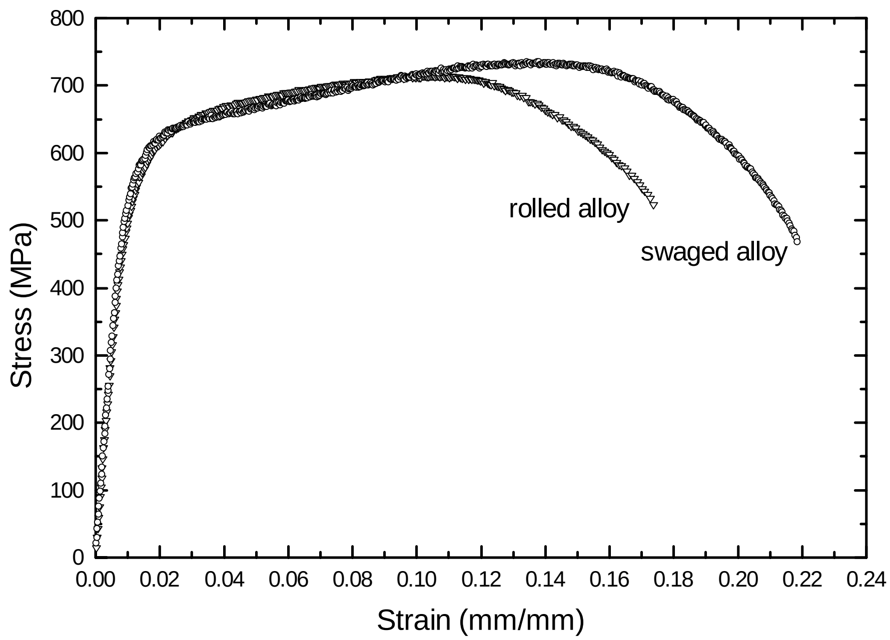


Figure 7 – Tensile stress-strain curves of swaged and rolled Ti-13Nb-13Zr alloy annealed at 900°C/30min WQ.

Table 1 – Mechanical properties of swaged and rolled Ti-13Nb-13Zr alloy at 900°C/30min WQ.

Mechanical Properties	Ti-13Nb-13Zr Swaged	Ti-13Nb-13Zr Rolled
Ultimate Tensile Strength (MPa)	733 ± 7	724 ± 13
Yield Strength (MPa)	510 ± 25	509 ± 23
Elastic Modulus (GPa)	64 ± 2	59 ± 5
Elongation (%)	21 ± 1	16 ± 1
Microhardeness (HV)	241 ± 12	256 ± 11

4. Conclusions

The present study investigated the microstructures and the mechanical properties of Ti-13Nb-13Zr alloy, which was produced by different routes of mechanical processing. In one route, the alloy was cold worked by swaging, in another, it was cold worked by rolling. With the analysis of the obtained results, it can be concluded:

(1) The heat treatment at 900°C/30min and water quenched after both mechanical working processes was sufficient to promote an homogeneous martensitic α' microstructure.

(2) The mechanical properties of Ti-13Nb-13Zr alloy are not dependent on the mechanical working process and its properties present adequate values for orthopedic applications.

(3) The different elongation values can be associated with the anisotropic nature of the cold rolled process.

(4) The similar mechanical behavior for both conditions allows the making of biomedical components of different shapes

5. Acknowledgements

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7. Responsibility notice

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