OBTAINING AND CHARACTERIZATION OF β TITANIUM ALLOYS FOR MEDICAL APPLICATIONS

Carla Dainese
Departamento de Engenharia de Materiais, Faculdade de Engenharia Mecânica, Universidade Estadual de Campinas, C.P. 6122, CEP:13083-970, Campinas, SP, Brasil
c023312@unicamp.com.br

Peterson Luiz Ferrandini
Departamento de Engenharia de Materiais, Faculdade de Engenharia Mecânica, Universidade Estadual de Campinas, C.P. 6122, CEP:13083-970, Campinas, SP, Brasil
peterson@fem.unicamp.br

Enrico José Giordani
Departamento de Engenharia de Materiais, Universidade Federal de São Carlos. Rodovia Washington Luís (SP-310), km 235, CEP:13565-905, São Carlos, SP, Brasil
enricogiordani@uol.com.br.

Rubens Caram
Departamento de Engenharia de Materiais, Faculdade de Engenharia Mecânica, Universidade Estadual de Campinas, C.P. 6122, CEP:13083-970, Campinas, SP, Brasil
rcaram@fem.unicamp.br

Abstract. *One of the most suitable metallic materials for orthopaedic applications is titanium and its alloys, due to the high biocompatibility, favourable weight/strength ratio and corrosion resistance. Nowadays, Ti-6Al-4V is the most used alloy for these applications despite the toxicity presented by V and Al. In order to replace these undesirable elements several alloys of much more biocompatible compositions are being developed, based on addictions of Nb, Ta and Zr, for example, that are β-stabilizers. The β Ti alloys present an important advantage over the α alloys that is the relatively low modulus. The purpose of this work was to present the characterization of a β Ti-25Nb alloy using scanning electron and optical microscopies, tensile test and Vickers hardness test. It was found that rapid cooled samples present completely martensitic structures while the slow cooled samples present β grains with grain boundary α precipitation. On mechanical properties, it was observed that , as expected, the martensitic structure presents lower strength and higher ductility. The water quenched sample presented σ_{UTS} = 635 MPa and total elongation 16.8 % while the furnace cooled sample presented σ_{UTS} = 770 MPa and total elongation 8.5 %. The hardness values, on the other hand, were higher for the martensitic structure.*

Keywords: *titanium alloys; mechanical properties, microstructure, heat treating , rolling.*

1. Introduction

While commercially pure titanium is currently used in dentistry, its relatively low strength limits its use in stress bearing orthopaedic applications (Lin, 2002), for what the most used Ti alloy is the well known Ti-6Al-4V. Titanium alloys can generally be divided into three major groups that are α, α + β and β. Unalloyed Ti is a good example of α structure. The α + β alloys, which is the case of Ti-6Al-4V, offer higher potential for strengthening by precipitation mechanisms, but on the other hand, it presents both relatively high stiffness (high modulus) and poor toughness. The β alloys present low modulus, which is remarkably important for orthopaedic applications, since it is closer to the bone modulus and hence does not prevent tissue stimulation, which is definitely necessary for the tissue soundness (Niinomi et al., 2002). Besides, the recent trend is to develop β alloys composed of non-toxic and non-allergic elements, like Ta, Nb and V.

The final microstructure of both α + β and β alloys are dependent not only on composition but also on thermo mechanical processing (Lütjering, 1999) and no doubt, it completely influences the mechanical properties shown by the alloy (Lindemann, 1999). Processing temperature, strain rate and cooling rate after processing are important parameters that determine the final microstructure, since the alloy may be processed at a temperature above, bellow or even at a temperature range through the β transus. Each of these situations may involve the presence of a certain amount of a primary phase, which may dictate a certain volume of β phase to be transformed into another depending on the cooling rate. On cooling rate, besides thermo mechanical processing, the Ti alloys can also be heat treated in order to obtain a microstructure formed by stable α and β phases or by the metastable α’, α” and ω phases.

The purpose of this work is to describe the obtaining of a β alloy of composition Ti-25Nb that was arc melted, hot rolled and heat treated, and its characterization regarding microstructure and mechanical properties.

2. Experimental procedures

High purity Nb and Ti were arc melted using a non-consumable tungsten electrode in a water-cooled copper hearth under an argon atmosphere. The ingots were flipped and remelted at least five times in order to improve composition homogeneity. Than the ingots were homogenized at 1000°C for 24 h and furnace cooled. Then the ingots were heated to 850°C and rolled, after what they were heated to the β field at 800°C and cooled under two different cooling rates. Figure 1 schematically shows the thermal and thermomechanical processing described. Samples were extracted from both the as cast and hot rolled specimens and prepared for metalographic characterization using the conventional methods and Kroll's etchant. The microstructural analysis was performed by optical and scanning electron microscopy (OM and SEM respectively). Energy diffraction spectroscopy (EDS) was used for semi-quantitative composition determination.

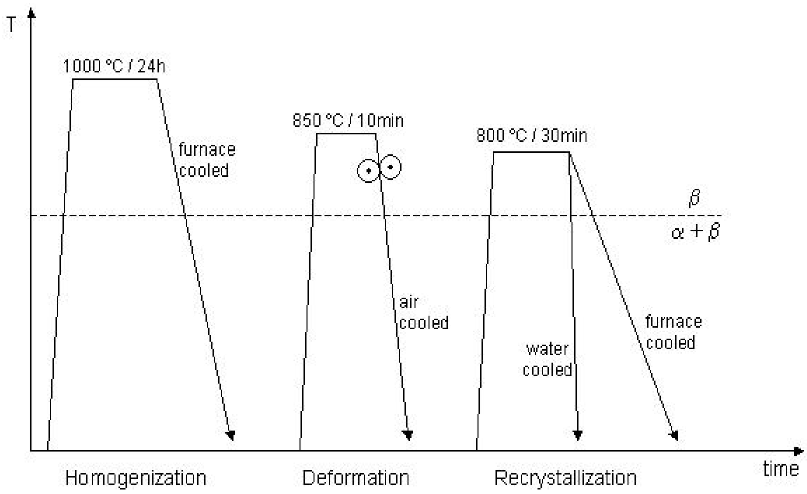


Figure 1. Thermal and thermomechanical processing of the Ti-25Nb alloy (schematically).

Mechanical properties of the alloy in the final conditions were evaluated by performing tensile and hardness tests. Tensile tests were performed using an MTS equipment, following the ASTM E 8 M standard, and the specimen is seen in Fig. 2. Micro-Vickers hardness tests were performed according ASTM E 92 using 200 gf load which was applied for 15 s.

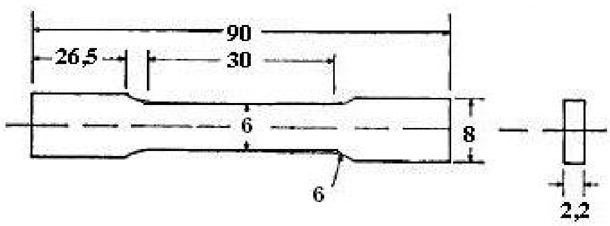
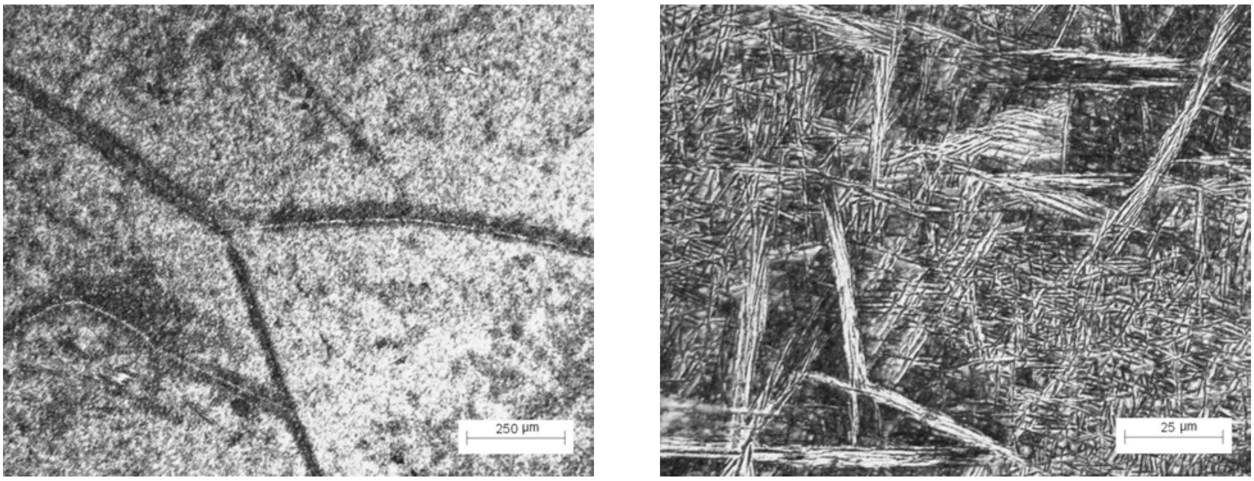


Figure 2. Dimensions of the tensile specimens (in mm).

3. Results and discussion

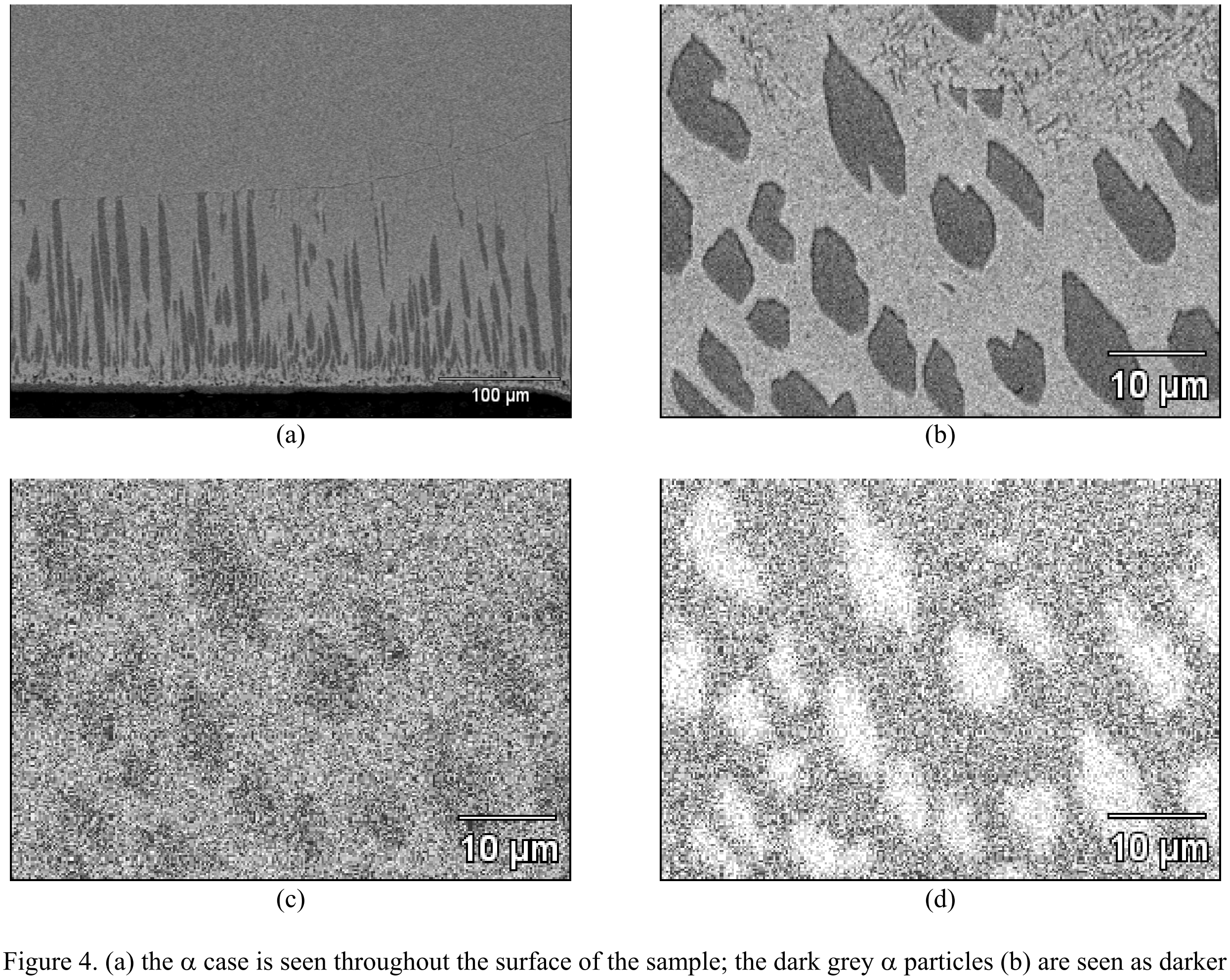
Figure 3 shows the microstructure obtained when the alloy was homogenized at 1000°C and furnace cooled. The first detail one notes is the coarse grained structure, Fig. 3(a), where the β grain boundary is marked by the presence of a continuous α-layer. Figure 3(b) shows the α + β structure found, composed of α plates within the β matrix. Using EDS analysis the ingot composition was determined, and results show that no significant difference exists through its entire length.

Intense α precipitation was noted near the specimens surface, as can be seen in Fig. 4(a), a phenomenon known as α case formation, attributed to oxygen contamination during the homogenization at 1000 °C. Oxygen is a powerful α stabilizer that may diffuse through the Ti array during high temperature treatments. Figure 4(b) shows back scattered electron image (BSE) of selected areas of the α case where one seems dark gray stabilized α particles surrounded by the transformed β matrix. The X-ray images shown in Fig. 4(c) and (d) show Ti and Nb distribution, respectively, which confirms that the dark gray α phase is titanium rich and niobium poor. The dark gray areas seen in Fig. 4(b) are slightly darker in Fig. 4(c), showing that those regions are Ti richer. On the other hand, those dark gray areas are lighter in Fig. 4(d), which in both cases indicates higher Nb content. One should agree that the transformed β phase must present higher content of the β-stabilizer Nb, while the α stabilized particles present lower Nb content and higher O and Ti contents.



(a) (b)

Figure 3. Microstructure of the sample homogenized and furnace cooled.



(a) (b) (c) (d)

Figure 4. (a) the α case is seen throughout the surface of the sample; the dark grey α particles (b) are seen as darker regions (c) in the X-ray image, which shows higher Ti content, and as lighter regions (e), which shows lower Ti content than the transformed β matrix.

The structure of the sample hot rolled from the β phase field (at 850°C) and air cooled presented much smaller β grain size than those of the homogenized one (Fig. 3), as can be seen in Fig. 5. Since the sample was a 2.2 mm thick sheet, one may consider that it was rapid cooled after rolling. Hence, its microstructure is considerably refined, presenting some α precipitated at the grain boundaries. Some precipitation is also seen within the grains. According to Lütjering (1999), the final microstructure depends heavily on the cooling rate after deformation, thus, one may suppose that despite the high cooling rate the sample was subjected, there was enough time to occur some precipitation. Besides, the strain imposed to the sample during the last rolling pass favours the α precipitation.

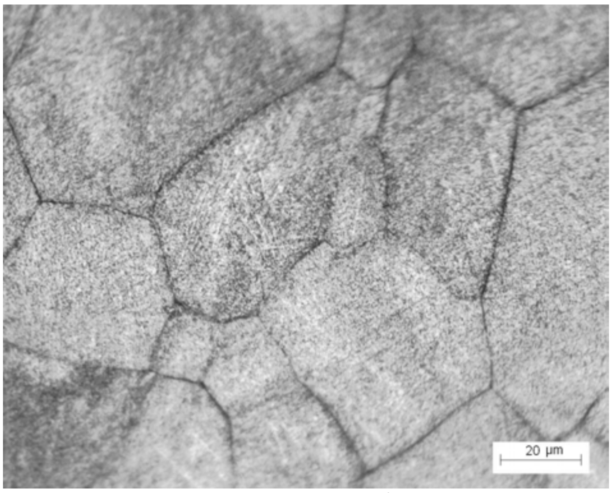


Figure 5. Microstructure of the Ti-25Nb alloy hot rolled from 850 °C and air cooled: refined microstructure presenting some α precipitation.

The microstructure of the samples heat treated at 800 °C after hot rolled and then furnace and water cooled are seen in Fig 6 and Fig 7, respectively. One can see that the structure of the furnace-cooled sample is formed by β grains presenting intense α precipitation at the grain boundaries. One should also notice the preferential direction of α precipitation. On the other hand, as expected, the structure of the water-cooled sample is completely martensítica, as can be seen in Fig. 7. The rapid cooling does not permit any diffusion and thus, only one phase is seen, with no boundary phase precipitation.

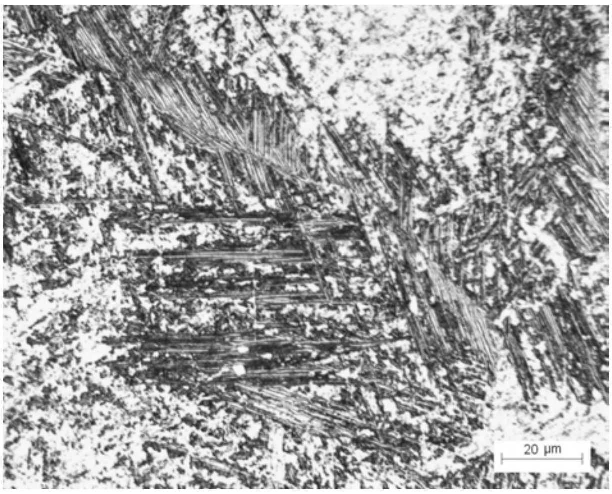


Figure 6. Microstructure of the Ti-25Nb alloy reheated and furnace cooled after hot rolled from 850 °C: α precipitation at the β grain boundaries.

Results obtained with tensile and hardness tests were apparently contradictory. While the results of tensile tests indicated that the martensitic structure presents lower strength, the micro-Vickers values found were considerably higher for the same structure as it can be seen in Tab 1. Not only the water-cooled structure presented lower tensile strength but also lower yield strength and higher total elongation than the furnace-cooled structure, which is completely coherent, since the water-cooled structure is completely martensitic (fig. 6). The furnace-cooled structure presents β grains with α precipitation at grain boundaries. The hexagonal close packed structure of the α phase presents higher strength as well as prevents any considerable deformation. The Vickers hardness values, on the other hand, does not follow the expected trend and are higher for the low resistant martensitic structure. The same unexpected property relationship was found by Aoki et al. (2004) and should be further investigated. Some hypotheses to be discussed are the influence of the α layer width, the α layer growth direction and the accommodation of the elastic deformation during the indentation process.

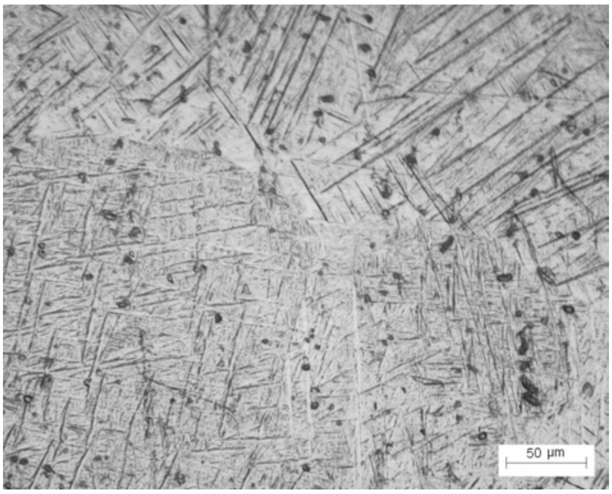


Figure 7. Microstructure of the Ti-25Nb alloy reheated and water cooled after hot rolled from 850 ^{o}C: structure presents only martensite

Table1: Mechanical properties of the conditions evaluate.

	σ_{UTS} (MPa)	σ_{YS} (MPa)	Elongation (%)	HV
As rolled	-	-	-	234
Water cooled	635	345	16.8	227
Furnace cooled	770	635	8.5	204

4. Conclusion

As a preliminary study, this work permits one to conclude that the microstructure of the Ti-25Nb alloy can be controlled, based on the thermal and thermomechanical processing imposed to it. After recrystallization, the higher cooling rate lead to completely martensitic structure, which are less resistant and more ductile than the slow cooled structure. The slow cooled structure presents β grain with grain boundary α precipitation, while the martensitic structure is more uniform.

5. Acknowledgements

The authors are grateful to CNPq (Conselho Nacional de Desenvolvimento Científico e Tecnológico) and to FAPESP (Fundação de Amparo a Pesquisa do Estado de São Paulo) for the financial support received during the period in which the research was carried out.

6. References

Aoki, T., Okafor, C.I., Watanabe, I., Hattori, M., Oda, Y., Okabe, T. “Mechanical properties of cast Ti-6Al-4V-Xcu alloys” Journal of Oral Rehabilitation, 31 (2004), p. 1109.

Lin, D.J., Chern Lin, J.H., Ju, C.P. “Structure and properties of Ti-7.5Mo-xFe alloys” Biomaterials, 23 (2002), p.1723.

Lindemann, J., Wagner, L. “Microtextural effects on mechanical properties of duplex microstructures in (α + β) titanium alloys” Materials Science and Engineering, A263 (1999), p. 137.

Lütjering, G. “Property optimisation through microstructural control in titanium and aluminium alloys” Materials Science and Engineering A263 (1999), p. 117.

Niinomi, M., Hattori, T., Morikawa, K., Kasuga, T., Suzuki, A.,Fukui, H., Niwa, S. “Development of low rigidity β-type titanium alloy for biomedical applications” Materials Transactions, v. 43, n^{o}12 (2002), p.2970.