Microstructural evolution of Ti-10Nb and Ti-15Nb alloys produced by the blended elemental technique

Martins, G.V.[1, a], Silva, C.R.M.[2, b], Henriques, V.A.R.[3, c],Borges Junior, L.A.[4, d], Souza, J.V.C.[1,e], Machado, J.P.B.[1, f]

[1]INPE, Av. dos Astronautas, 1.758, S. J. Campos - SP, CEP. 12245-970, Brazil

[2]UnB, Asa Norte, Brasília - DF, CEP. 70910-900, Brazil

[3]DCTA-IAE/AMR - Pça. Mar. do Ar E. Gomes, 50, S. J. C. - SP, CEP. 12228-904, Brazil

[4]UNIFOA, Av. Paulo E. A. Abrantes, 1.325, Volta Redonda – RJ, CEP. 27240-560,Brazil

[a]givmartins@yahoo.com.br, [b]cosmeroberto@gmail.com, [c]vinicius@iae.cta.br, [d]borges.jr@itelefonica.com.br, [e]vitor@las.inpe.br, [f]joaopaulo@las.inpe.br

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Abstract. Alfa/beta titanium alloys have been intensely used for aerospace and biomedical applications. Production of powder metallurgy titanium alloys components may lead to a reduction in the cost of parts, compared to those produced by conventional cast and wrought (ingot metallurgy) processes, because additional working operations (machining, turning, milling, etc.) and material waste can be avoided. In this work, samples of Ti- 10, 15Nb (weight%) alloys were obtained by the blended elemental technique using hydride-dehydride (HDH) powders as raw material, followed by uniaxial and cold isostatic pressing with subsequent densification by sintering carried out in the range 900–1500 ºC. These alloys were characterized by X-ray diffractometry for phase composition, scanning electron microscopy for microstructure, Vickers indentation for hardness, Archimedes method for specific mass and resonance ultrasound device for elastic modulus. For the samples sintered at 1500ºC it was identified α and β phases. It was observed the influence of the sintering temperatures on the final microstructure. With increasing sintering temperature, microstructure homogenization of the alloy takes place and at 1500 ºC this process is complete. The same behavior is observed for densification. Comparing to the Ti6Al4V alloy properties, these alloys hardness (sintered at 1500 ºC) are near and elastic modulus are 18% less.

Introduction

Titanium and its alloys have over the years proven themselves to be technically superior and cost effective materials for a wide range of applications spanning the industries of aerospace, biomedical, marine, and even commercial products [1]. This is because of their high strength to weight ratio, stiffness, immune to corrosion in sea water environment, good erosion resistance, and importantly their acceptable mechanical properties at elevated temperatures [2].

Cost reductions can be obtained by vacuum hot pressing (VHP) and powder metallurgy (P/M) techniques by producing near net shapes and consequently minimizing material waste and machining time [3].

Some titanium alloys present a very low modulus of elasticity which is roughly half that of steel and nickel alloys. Materials with high flexibility (i.e. low elastic modulus) present reduced bending and cyclic stresses in deflection-controlled applications, making it ideal for springs, bellows, body implants, dental fixtures, dynamic offshore risers, drill pipe and various sports [4].

In this paper it will be presented the development and characterization of both Ti- 10 wt.%Nb (Ti-10Nb) and Ti- 15 wt.% Nb (Ti-15Nb), using the blended elemental technique, showing mainly their microstructural evolution on different sintering temperature, and comparing their properties with Ti alloys characteristics.

Experimental Procedure

In this work, samples of Ti-10Nb and Ti-15Nb were obtained by the blended elemental technique. Both Niobium and Titanium powders (raw materials) were obtained by the hydride-dehydride (HDH) process. Niobium chips originated from a machining operation on a high purity Niobium ingot (Electron bean melted) were washed in water, acid pickled and dried in stove. These were submitted to hidrogenation treatment in electric resistance furnace at a temperature of 600 °C, under 0.15 MPa of pure H_2 and overcooled to room temperature. Titanium's sponges coming from the Kroll process were hydrogenized in vertical furnace, at 500 °C and 0.1 MPa of pure H_2. The hydrogenated powders were milled in a vibratory mill without protecting atmospheres. The dehydriding stage was carried out in dynamic vacuum conditions, at 800 °C for Nb and 550 °C for Ti. The starting powders were weighted with the desired composition and blended in a planetary mill, putting the mixture inside a closed plastic recipient with balls inside.

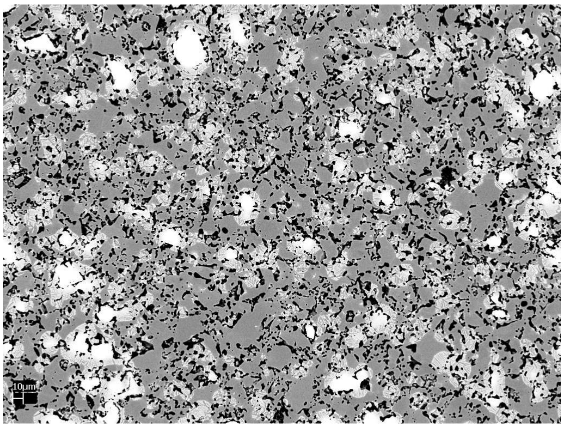
After that, the powders were: cold uniaxially pressed, under pressure of 10 MPa, in cylindrical 15 mm dia-dies and encapsulated under vacuum in flexible rubber moulds for cold isostatically presseing (CIP) at 350 MPa for 30 s in an isostatic press. The specimens were sintered in the range of 900–1500 ºC, in high vacuum, with a heating rate of 20 °C/min. On the nominal temperature, the sample was held at the chosen temperature for 2 h and then the furnace was cooled to room temperature. Microstructural analysis of the materials was performed in a JEOL model JSM-5310 scanning electron microscope (SEM); and a Philips PW-1380/80 X-ray diffractometer (XRD). The sample were hot mounted in resin, ground from #180 to #4000 SiC sandpaper and polished using OPS (colloidal silica – Struers) with oxalic acid. Etching was done by immersing the samples in a modified Kroll solution (3ml HF + 6 ml HNO3 + 100 ml H2O) for 10 seconds to reveal its microstructures. Microhardness tests were carried out in Micromet 2004 equipment, Buehler, with a load of 0.2 kgf. The density of the sintered sample was determined by the Archimedes method and the elastic modulus by resonance ultrasound.

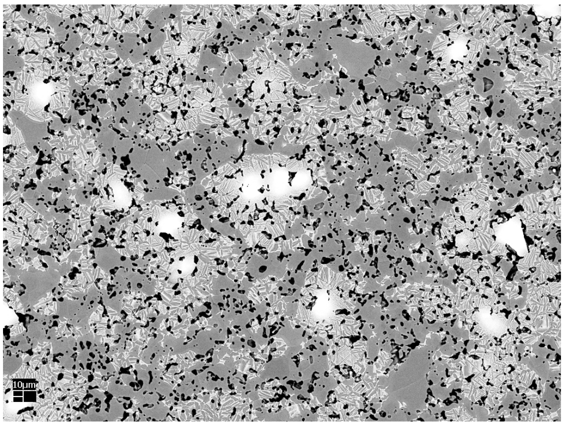
Results and Discussion

Fig. 4 and 5 show the microstructural evolution for the Ti-10Nb and Ti-15Nb alloys respectively, in the sintering temperatures of 900, 1000, 1100, 1200, 1300 and 1500 ºC. The microstructural analysis showed an acicular two-phase (α + β) microstructure growing with the dissolution of the niobium particles that act as β-phase nucleation agent, by increase of the sintering temperature. Nb particles are white color and Ti particles are dark gray. Nb rich regions are present in both samples until 1100 ºC and they go from an irregular shape (900 ºC) until a round shape (1100 ºC). Exclusively for Ti-15Nb it could be seen that at 1200 ºC there were still a round soft gray colored regions, indicating the presence of a remainder Nb rich region. The first two-phase areas resembling a Widmanstätten (α + β) structure become distinguishable at 1000 ◦C. These areas consist of a pure niobium core region (a strong β -

stabilizer in titanium alloys) surrounded by a two-phase microstructure. With increasing sintering temperature, the dissolution of the niobium particles continues with consequent increase in the volume fraction of the two-phase structure, since the Nb dissolution reaches the more distant titanium areas.

At 1500 ◦C, the Widmanstätten like (α + β) structure is distributed all over the alloys resulting in a homogeneous microstructure. In addition, the porous volume fraction decreases with increasing sintering temperature.


(900 ºC)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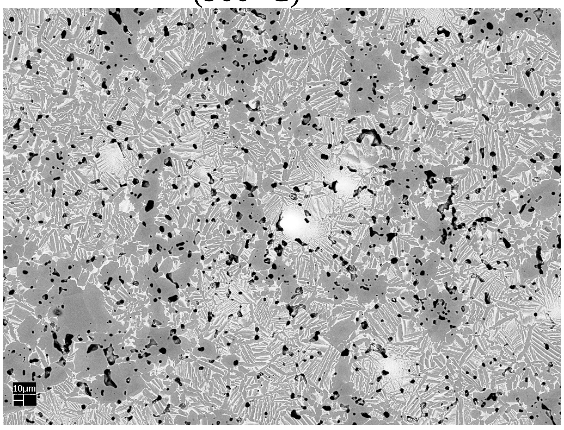
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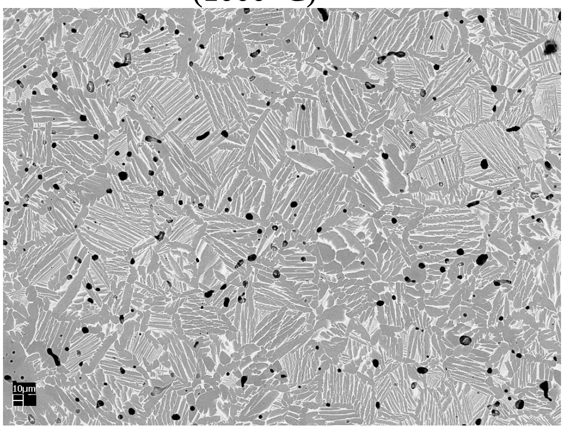
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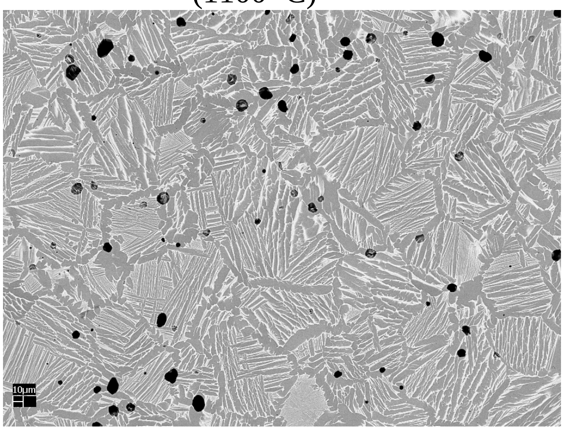
(1300 ºC)

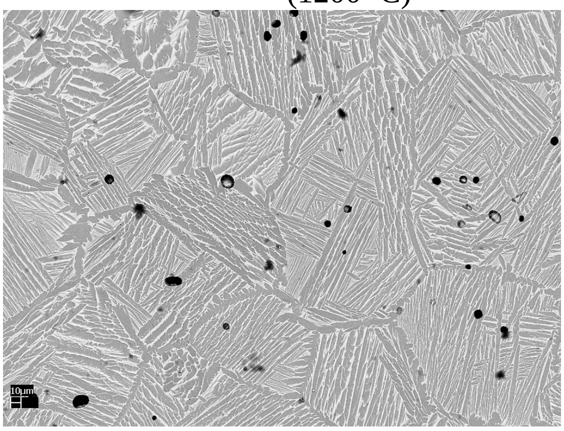
(1500 ºC)

Fig. 1. Microstructural evolution of Ti-10Nb alloy in the range from 900 to 1500 °C.

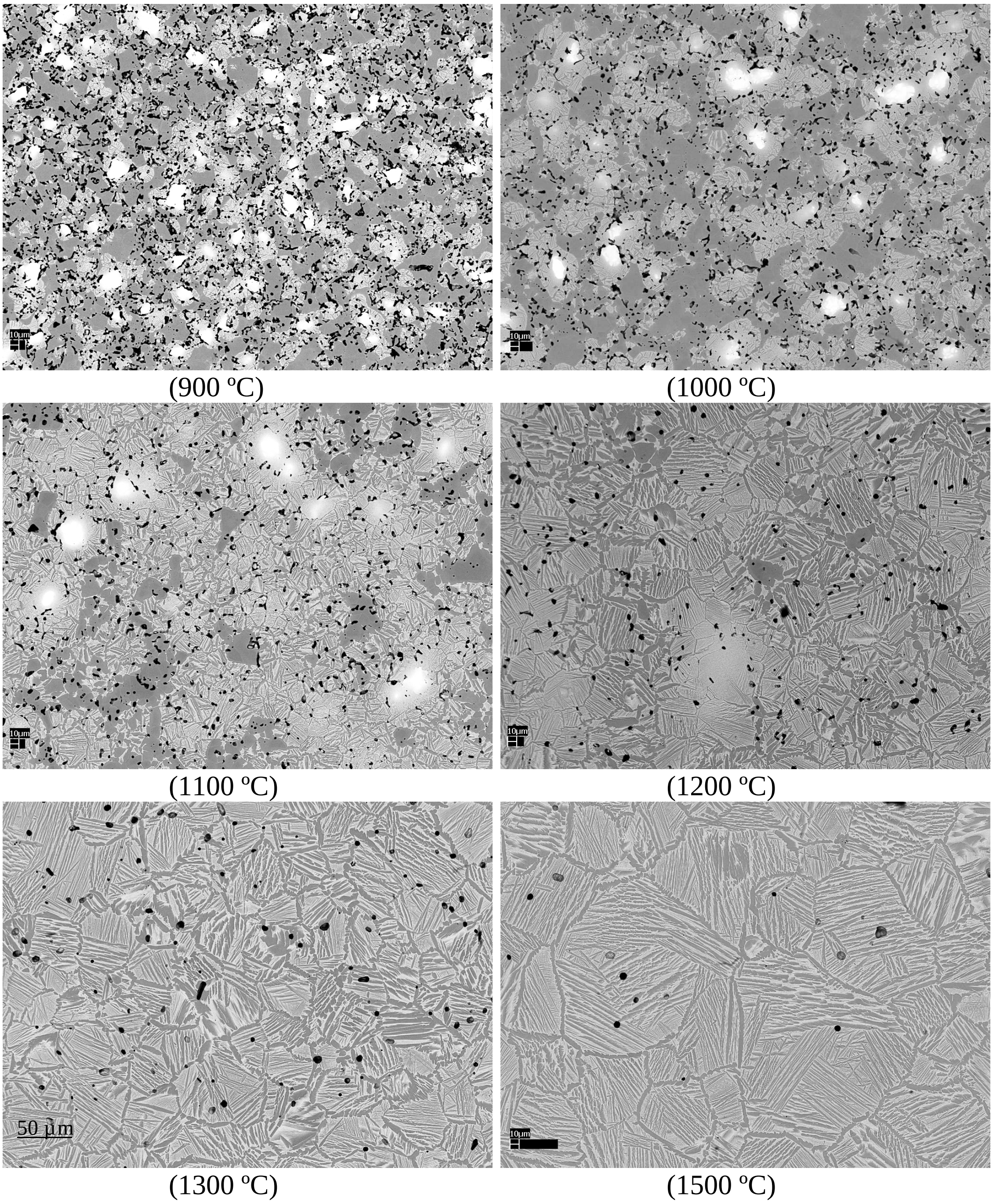

Fig. 2. Microstructural evolution of Ti-15Nb alloy in the range from 900 to 1500 °C.

Fig. 3 presents the XRD patterns for Ti-10Nb (a) and Ti-15Nb (b) alloys sintered in the range from 800 ºC to 1500 ºC. It was identified peaks from α and β Ti phases. However, it is important to elucidate that Nb and β Ti phases present peaks in the same 2θ (38,4º, 55,5º and 69,6º). The others XRD peaks are related to the α Ti phase. It was not observed the presence of intermetallics or impurities.

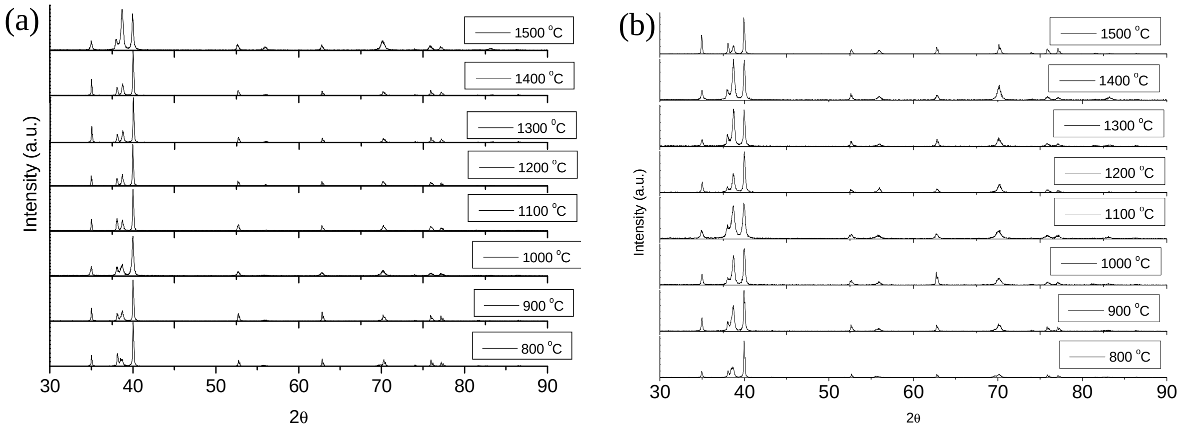

Fig. 3. XRD patterns for Ti-10Nb (a) and Ti-15Nb (b) alloys sintered in the range from 800 ºC to 1500 ºC.

The variation of density on different sintering temperatures is shown on Fig.4 for Ti-10Nb (a) and Ti-15Nb (b) alloys. One can observe the higher densification level from 800 to 1100ºC, possibly related to the expected β transus range. For both samples the maximum densification is reached at 1500 ºC, with values of 99,70 % (of the theoretical density) for Ti-10Nb and 99,73 % for Ti-15Nb.

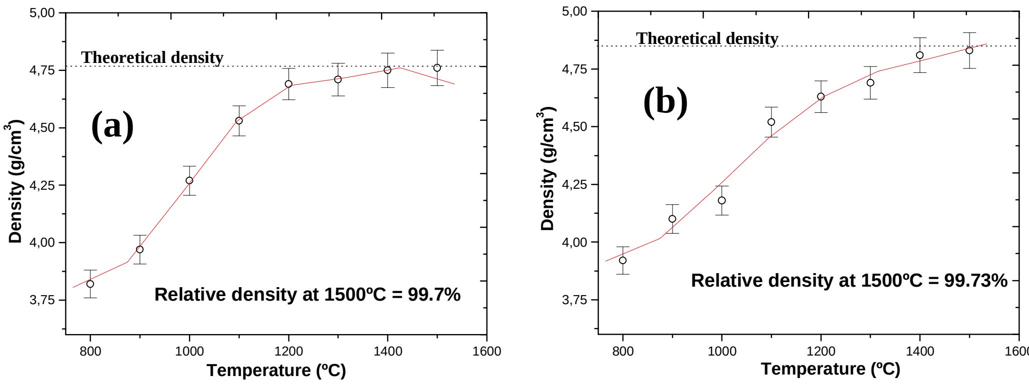

Fig. 4. Density vs. sintering temperature for Ti-10Nb (a) and Ti-15Nb (b). Dotted line indicates the alloy theoretical density.

Fig. 5 shows the Vickers hardness values for Ti-10Nb, Ti-15Nb, pure Ti, Ti6Al4V and INOX F138. It can be seen that both alloys, Ti-10Nb and Ti-15Nb, presented hardness values near Ti-6Al-4V alloy value.

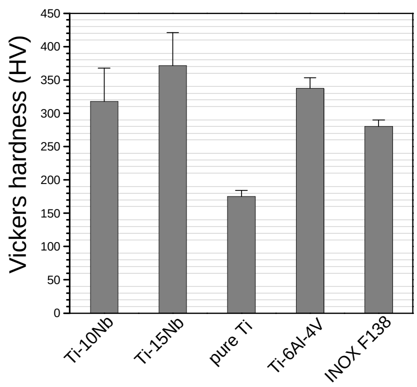

Fig. 5. Vickers hardness values for Ti-10Nb, Ti-15Nb, pure Ti, Ti6Al4V and INOX F138.

There are lots of applications, in the aerospace and biomedical field, that require flexible metallic materials. Fig. 6 presents the elasticity modulus for Ti-10Nb, Ti-15Nb, pure Ti, Ti6Al4V and INOX F138. One can observe that both Ti-Nb alloys exhibited the lower modulus. Their Young modulus are approximately 1.3 times lower compared to Ti6Al4V modulus.

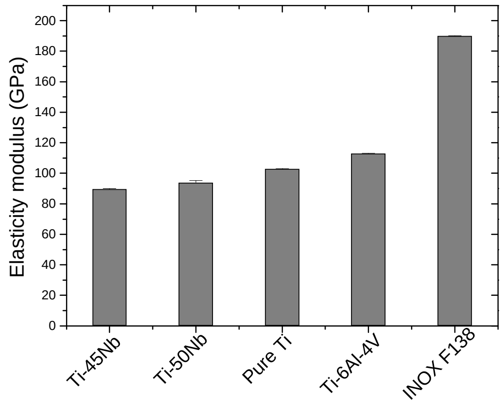

Fig. 6. Elasticity modulus for Ti-10Nb, Ti-15Nb, pure Ti, Ti6Al4V and INOX F138.

Conclusions

From this work it can be concluded that the applied experimental methodology made possible to observe the diffusion and reaction of Nb particles in the Ti matrix, according to the different sintering temperatures used. It could be determined that, for both Ti-10Nb and Ti-15Nb alloys, the temperature of 1500 ºC is adequate to obtain a homogeneous material with Widmanstätten-like microstructure (α + β phases) and high relative density. Comparing the physical / mechanical properties, of the developed alloys, with the aerospace and biomedical alloys presented here, one can conclude that these alloys present a high potential to be applied in this fields.

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