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STUDY OF Ti-35Nb ALLOY PROCESSED BY POWDER METALLURGY USING CONVENTIONAL PRESSURES

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Abstract. *Titanium alloys have a good combination of mechanical and physical properties, including a high strength-to-weight ratio and excellent corrosion resistance. Beta titanium alloys are composed of non-toxic and biocompatible elements, such as niobium, and have a low modulus of elasticity when compared to other materials used in the manufacture of implants, such as stainless steels. Such alloys can be obtained by melting processes in an inert atmosphere or by powder metallurgy processes. Within the presented, the objective of this work is to evaluate the microstructural and mechanical behavior of the beta-type titanium alloy, Ti-35Nb (% by weight), obtained by powder metallurgy using uniaxial pressing and conventional pressures (400, 600 and 800 MPa), followed by sintering in an inert atmosphere. The main results show that in the compaction pressures used in this study, there is the occurrence of mechanical twinning in the particles of titanium and also their fracture, as well as changes in the shape of the niobium particles, due to the plastic deformation that occurred during the compaction. After sintering, the recrystallization of the Ti particles was verified. The contour between several Nb particles was eliminated due to the occurrence of atomic diffusion.*

Keywords: *Powder Metallurgy, Titanium alloys, Conventional pressures.*

1. INTRODUCTION

The application of a material as a permanent implant in the human body requires that it has, in addition to adequate mechanical properties, corrosion resistance and biocompatibility (Tamirisakandala et al., 2003). In the case of metallic materials used in orthopedic implants, some titanium alloys can comply with these requirements.

High strength-to-weight ratio and excellent corrosion resistance are important characteristics of titanium alloys, in addition, they also have a good combination of mechanical and physical properties, making them desirable for a variety of applications, such as in the chemical and biomedical industries. Through the combination of microstructures, it is possible to obtain adequate mechanical properties for each application, as a result of phase transformations in these alloys (Karthikeyan et al., 2008).

The Ti-6Al-4Va alloy, from the group of titanium alloys of the $\alpha+\beta$ type, was widely used as an implant material, replacing pure titanium, due to the better mechanical resistance of the components made with this alloy. However, research has found that vanadium is toxic and can damage the health of the implant (Eisenbarth et al., 2004) and aluminum can also be harmful, disturbing the biological system and causing local inflammation (Zaffe et al., 2004), can also be related to disorders in the implanted neurological system (Silva et al., 2004).

Other alloys used as materials for orthopedic implants are alloys based on Cr, Co and Mo and stainless steels. One of the problems when using these materials is the mechanical properties they present, especially the Young's modulus, as materials with these properties much higher than that of human bone, when implanted in the patient, can lead to insufficient transfer of tension, it can generate bone degradation and consequently, osteoporosis (Tarr et al., 1983), and it can also cause fractures due to it.

Titanium alloys stand out for having mechanical properties that can be modified through thermo-mechanical processing, which result in the attainment of optimized microstructures in relation to the type, morphology and distribution of phases (Ankem and Greene, 1999; Banerjee et al., 2005). In the case of type β titanium alloys, due to their physical, chemical and mechanical characteristics, they have the potential to replace, in the near future, type $\alpha+\beta$ alloys in the manufacture of orthopedic implants. This fact has stimulated the acquisition of knowledge about the mechanisms and influencing parameters involved in defining the microstructure of these alloys, which allows designing materials with ideal behavior for application in orthopedics.

Type β titanium alloys are composed of non-toxic and biocompatible elements, such as niobium, and have corrosion resistance and biocompatibility, which are important requirements for such alloys to be used as a permanent implant in the human body, as mentioned above. Furthermore, they are also immune to bodily fluids.

Such titanium alloys can be obtained by melting processes in an inert atmosphere or by powder metallurgy processes from elementary powders, which is a viable alternative, as through it is possible to manufacture parts with complex geometries and dimensions close to the final ones, with a good surface finish. In addition, the sintering temperatures used are low compared to other metallurgical processes, and can be carried out in simple design furnaces, with lower energy consumption (Chaverini, 1986). As the compaction pressure used is increased, the degree of compaction of the metallic powders is also increased, which directly influences the behavior of the material, both during compaction and during the sintering process.

In this context, the aim of this work is to evaluate the microstructural and mechanical behavior of the Ti-35Nb alloy (% by weight), obtained via powder metallurgy using uniaxial pressing and conventional pressures, followed by sintering at 800°C for 60 min.

2. EXPERIMENTAL

Initially, titanium and niobium elementary powders were classified by sieving using Tyler Series sieves mounted on a magnetic sieve shaker. After classification, the powders were weighed on an analytical balance, 35% by weight of each sample corresponding to niobium and 65% by weight corresponding to titanium, then the powders were mixed in rotating cylinders for 48 h, in order to allow their homogenization.

After mixing, the uniaxial pressing of the powders was carried out at high pressures, considering the pressures of 400, 600 and 800 MPa, using a hydraulic press for this, giving rise to the green compact, that in the sequence were sealed in quartz tubes under vacuum. Sintering was carried out at temperatures of 800°C for 60 min, followed by air cooling.

The metallographic preparation of the green compacted was carried out, consisting of sanding, using silicon carbide sandpaper 220, 400, 600, 800, 1000 and 1200, followed by polishing using DP-PLUS polishing cloth and colloidal silica (OP-S), both from Struers. The chemical attack was performed using the following reagent 100 ml H_2O, 10 ml HNO_3 and 5 ml HF, in varying times of 3 to 10 seconds. The characterization was performed using optical light microscopy and Vickers hardness (microhardness), 10 microhardness measurements were performed, using a load equal to 0.98 N for 20 seconds.

3. RESULTS AND DISCUSSION

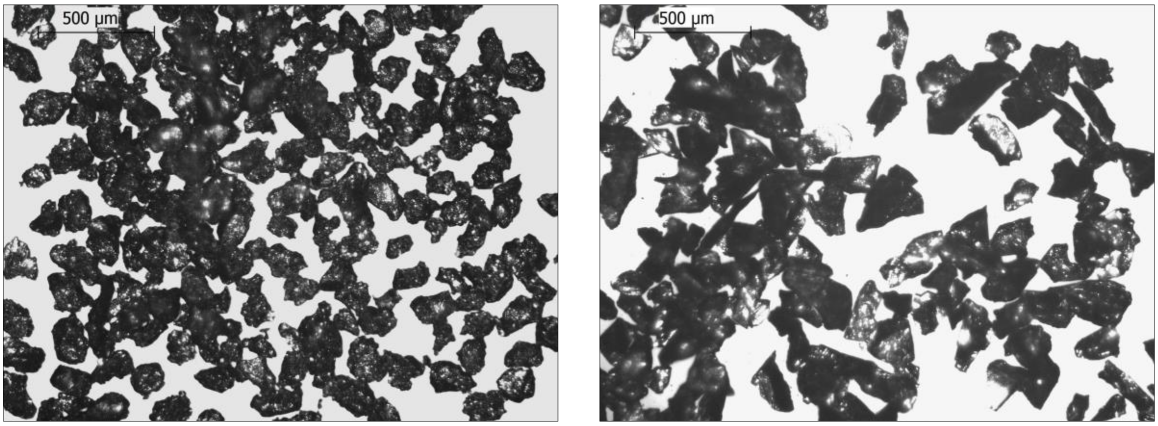
The results obtained for the characterization of the titanium and niobium powders and also the results of the Ti-35Nb alloy after uniaxial compaction at pressures equal to 400, 600 and 800 MPa followed by sintering at 800°C for 60 min are presented below.

3.1 Characterization of titanium and niobium powders

After classifying the titanium and niobium powders individually, their characterization was carried out, which was divided into 2 steps: verification of the morphology and size of the powders of each element via optical microscopy and measurements of the apparent density of the powders.

The results referring to the average size of the powders of the metals involved are qualitative, being an estimate. The measurements were performed considering the largest dimension presented by the powder particles. Thus, titanium powders have an average size corresponding to 190 ± 43 µm and niobium powders have a size corresponding to 207 ± 65 µm. According to the microscopic analysis of the powders of both materials, they have an irregular shape, with titanium powders having a more uniform size (Figure 1a) and niobium powders having a higher aspect ratio (length greater than width), as can be seen in Figure 1b.

The apparent density measurements were performed by measuring the mass presented by each material considering a predetermined volume. According to the apparent density measurements, the density of the titanium powders is equal to 1.99 g/cm3 and the niobium is equal to 4.14 g/cm3. Comparing with the theoretical density of these materials, dTi = 4.5g/cm3 (Lampman, 1998) and dNb = 8.57 g/cm3 (Lambert, 1998), apparent density was found to be lower than these, this is due to the fact that the initial powders occupy more volume, since they do not present compaction, having many empty spaces among the dust particles.



(a) (b)

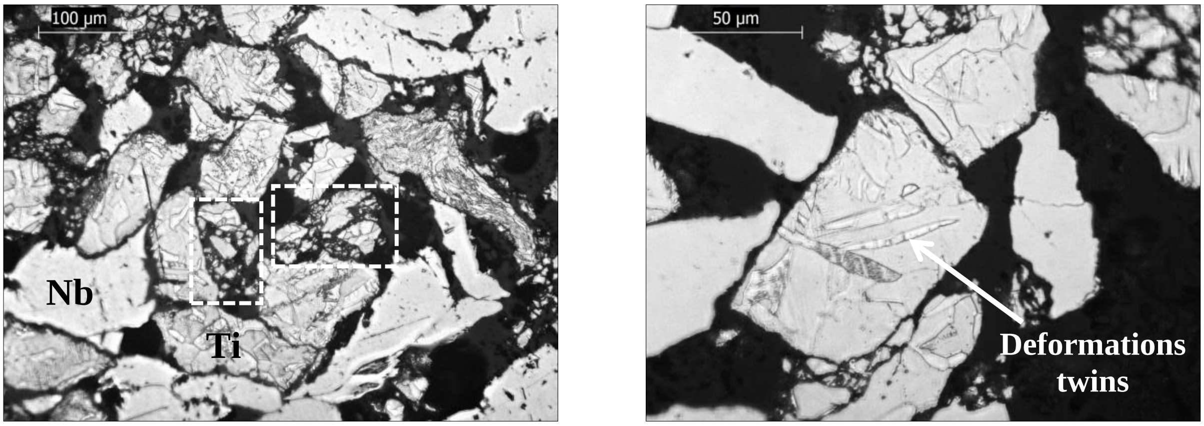
Figure 1. Powders of: (a) titanium; (b) niobium

3.2 Characterization of green compacted

After characterizing the elementary titanium and niobium powders, they were weighed according to the chemical composition of the alloy and then mixed in a rotating drum for 48 hours, in order to ensure their compositional homogeneity. This was followed by pressing in a matrix, using a hydraulic press for this. The pressures used were equal to 400, 600 and 800 MPa.

Figure 2a shows the micrograph of the Ti-35Nb alloy compacted at 400 MPa. It is verified the presence of porosity (dark regions) and also individual niobium and titanium powders (identified in the figure). It was found that the titanium grains fractured during compaction (highlighted in Figure 2a) and also showed deformation twins (Figure 2b). According to Humphreys and Hatherly (2004), the deformation of materials with a compact hexagonal structure (HC), as well as titanium, is due to the fact that this crystalline lattice presents less symmetry and also fewer slip systems than cubic structures, this deformation mode being more significant at low strain and is an important deformation mode.

The identification of deformation marks presented by titanium grains is due to their morphology, because, according to Padilha (1997), the marks formed during cold plastic deformation have lenticular contours, as observed in this study.

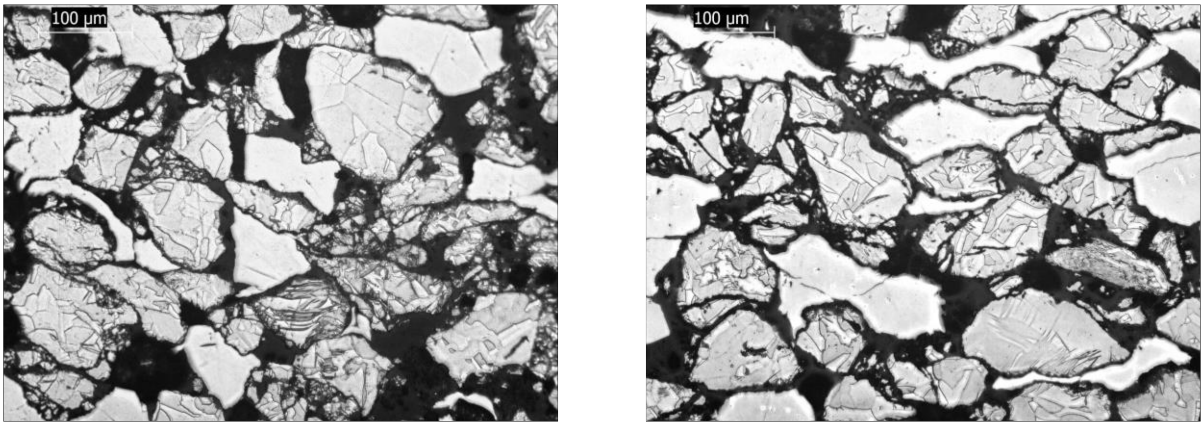


(a) (b)

Figure 2. Micrograph of Ti-35Nb alloy uniaxially compacted to 400 MPa, showing: (a) highlighted fractured titanium powder particles; (b) deformation twins presented by titanium powders.

The samples compacted at 600 MPa presented similar behavior to those compacted at 400 MPa, the titanium powders also presented deformation twins and the samples still presented accentuated porosity, as can be seen in Figure 3a.

Unlike other compacted microstructures, the one produced at 800 MPa and close to 1000 MPa, was where a significant reduction of large pores occurred in the microstructure. The samples compacted at 800 MPa presented a different behavior from the previous ones, as it was found that the niobium powders changed more clearly, due to the plastic deformation caused by the higher compaction pressure, as can be seen in Figure 3b. Titanium particles behaved similarly to samples compacted at pressures below 800 MPa and continue to show deformation twins.



(a) (b)

Figura 3. Micrograph of Ti-35Nb alloy uniaxially compacted to: (a) 600 MPa; (b) 800 MPa.

According to the Vickers hardness results it was verified that as the compaction pressure was increased, the measured hardness values also increased, this behavior was observed for both titanium and niobium. The compact produced in 800 MPa and close to 1000 MPa presented microhardness with lower standard deviation and consequently representative of the material. This may be associated with a simultaneous action of the uniform distribution of the degree of hardening, a reduction in the size of the porosity and an increase in your amount present in the compacted microstructure. This can be explained by the greater mechanical energy supplied to the material during compaction, causing a greater density of the powders and, consequently, greater plastic deformation of the same. It is also verified that niobium had lower hardness than titanium, which explains its shape change during compaction, as it has less hardness, thus being more ductile, corroborating the results presented. Table 1 presents the results of the Vickers hardness measurements of the samples uniaxially compacted at 400, 600 and 800 MPa.

Table 1. Result of hardness measurements of Ti-35Nb alloy samples uniaxially compacted at 400, 600 and 800 MPa.

Compaction pressure (MPa)	**Vickers hardness (Mean ± Standard Deviation)**	
	Titanium	**Niobium**
400	170 ± 19	102 ± 14
600	188 ± 18	113 ± 6
800	200 ± 13	121 ± 8

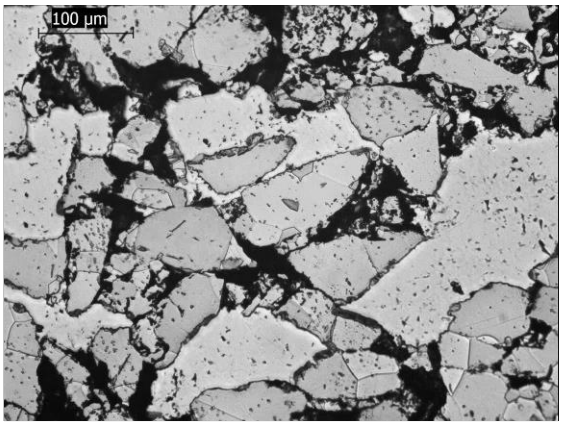
3.3 Characterization of sintered samples

Comparing the results of Figures 2 and 3 to those presented in Figure 4, it was observed that the temperature was sufficient to increase densification and reduce the degree of hardening produced in the sample compacted at 800 MPa. The main driving force for the sintering of the compact was temperature. According to the microstructure analysis of the compacted samples and then sintered at 800°C for 60 min, it was found that the Ti particles recrystallized, as there is a microstructural change when compared to the microstructure presented by the compacted sample. Note that the microstructure presented by these particles is that typical of recrystallized material, that is, it has equiaxed grains (with dimensions approximately equal in all directions).

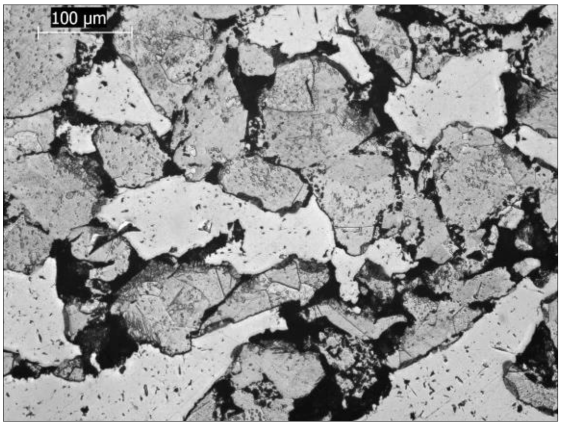
Regarding the microstructure, it is possible to defer a recovered material from a deformed one, because during the recovery process there is no modification of the deformed structure, with only a decrease and distribution of the density of defects in the structure, while in recrystallization, if the nucleation of new grains with a different shape from the deformed material, which are equiaxed (Padilha and Siciliano Jr., 2005).

Regarding the Nb particles, it was found that sintering occurred in several of them, with their union, thus, the boundary between them was eliminated. This union of particles is due to the occurrence of atomic diffusion, linking the particles in such a way as to make them a single one with a size larger than those verified in the green compact.

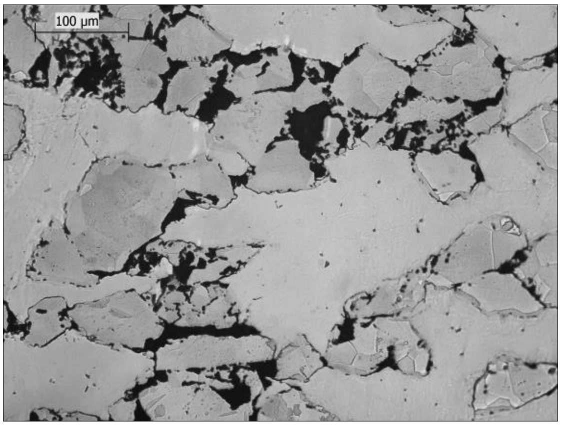
The microstructure of the Ti-35Nb alloy samples that were compacted and then sintered at 800°C for 60 min is shown in Figure 4.



(a)



(b)



(c)

Figura 4 – Micrograph of Ti-35Nb alloy compacted and sintered: (a) 400MPa, 800°C/60min; (b) 600 MPa, 800°C/60min; (c) 800 MPa, 800°C/60min.

The results of the Vickers hardness measurements (microhardness) presented refer to the average of 10 measurements performed. In general, it was found that samples sintered at 800°C showed that there was a decrease in hardness in relation to compacted samples, and this behavior was observed for all pressures studied and also for both Ti and Nb, as stated. in Table 2, where the hardness values of the green compact were included for comparison purposes.

Table 2 - Result of Vickers hardness measurements of Ti-35Nb alloy samples uniaxially compacted at 400, 600 and 800 MPa and sintered at 800°C/60 min.

Condition	**Titanium**			**Niobium**		
	Vickers hardness ± Standard deviation			**Vickers hardness ± Standard deviation**		
	400 MPa	**600 MPa**	**800 MPa**	**400 MPa**	**600 MPa**	**800 MPa**
800°C/60min	153 ± 12	162 ± 15	179 ± 11	73 ± 6	87 ± 7	90 ± 5

Comparing the results in Table 1 with those presented in Table 2 was noted that the mean microhardness values shown in Table 2 were significantly reduced. This is mainly due to the elimination of the degree of hardening present in the compacted microstructure and total dependence on a highly densified microstructure, typical of the final sintering step. With respect to Ti particles, the decrease in hardness values observed in samples sintered at 800°C was due to the recrystallization of the material, which can be confirmed by the microstructure. According to Humphreys and Hatherly (2004), during the recrystallization process there is an annihilation of a large number of dislocations and the consequent softening of the material, with the formation of new grains with low density of dislocations and with an equiaxed shape.

With respect to Nb, the Vickers hardness presented by the sintered samples is lower than that of the green compact. This decrease in hardness values is related to the occurrence of recovery and/or recrystallization of the material, and it is not possible to specify which phenomenon is occurring, since it was not possible to visualize the microstructure of the

Nb particles with the chemical attack used, as it occurred emphasis on Ti and also if the formation of a diffusion layer between Ti and Nb had occurred, such chemical attack would be efficient in revealing the resulting microstructure.

4. CONCLUSIONS

According to the results obtained, it can be concluded that at the compaction pressures used in this study there is the occurrence of mechanical twinning in the titanium powder particles and also its fracture, as well as changes in the shape of the niobium particles, due to the plastic deformation that occurred. during compression. In samples sintered at 800°C, the occurrence of recrystallization of Ti particles was verified. The boundary between several Nb particles was eliminated due to the occurrence of atomic diffusion. The main driving force for the sintering of the compact was temperature. The sample compacted at 800 MPa and sintered at 800ºC was the one that presented a microstructure in the final stage of sintering.

5. ACKNOWLEDGEMENTS

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