

Effect of Hydrogenation Pressure on Microstructure and Mechanical Properties of Ti-13Nb-13Zr Alloy Produced by Powder Metallurgy

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Abstract: The effects of the hydrogenation stage on microstructure and mechanical properties of Ti-13Nb-13Zr alloy produced by powder metallurgy have been studied. Powder alloys have been produced by hydrogenation with 250 MPa or 1 GPa and via high energy planetary ball milling. Samples were isostatically pressed at 200 MPa and sintered at 1150 °C for 7, 10 and 13 hours. Elastic modulus and microhardness were determined using a dynamic mechanical analyzer (DMA) and a Vickers microhardness tester. Density of the samples was measured using a liquid displacement system. Microstructure and phases presents were analyzed employing scanning electron microscopy (SEM). Elastic modulus was 81.3 ± 0.8 and 62.6 ± 0.6 GPa for samples produced by 250 MPa and 1 GPa hydrogenation, respectively when sintered for 7h.

Introduction

Hydrogen has been used as a pulverizing agent for rare earth-transition metals alloys due to its extremely high diffusion rate at low and high temperatures [1]. Biocompatible alloys are being studied with great emphasis on corrosion behavior, surface properties and biocompatibility on implants [2]. Titanium and its alloys show lower elastic modulus, superior biocompatibility and enhanced corrosion resistance [3,4]. However, the use of titanium alloys has been limited to commercially pure titanium (Ti_{Cp}) and Ti-6Al-4V alloy [4]. Notwithstanding, in recent years vanadium has been found to cause cytotoxic effects and adverse tissue reactions and aluminum has been associated with potential neurological disorders [5-9]. Recently, Ti-13Nb-13Zr alloy has been developed and classified as totally biocompatible. This alloy shows low modulus of elasticity and high mechanical properties [10]. The advantage of obtaining titanium alloys by powder metallurgy is to produce a porous structured surface which is a requirement for application in dental implants [11]. Other advantages include better surface finishing, better homogeneity and near-net-shape of the final product [10]. In this study, hydrogen has been used to pulverize Ti, Nb and Zr to produce by powder metallurgy a Ti-13Nb-13Zr alloy. Microstructures and physical properties have also been studied.

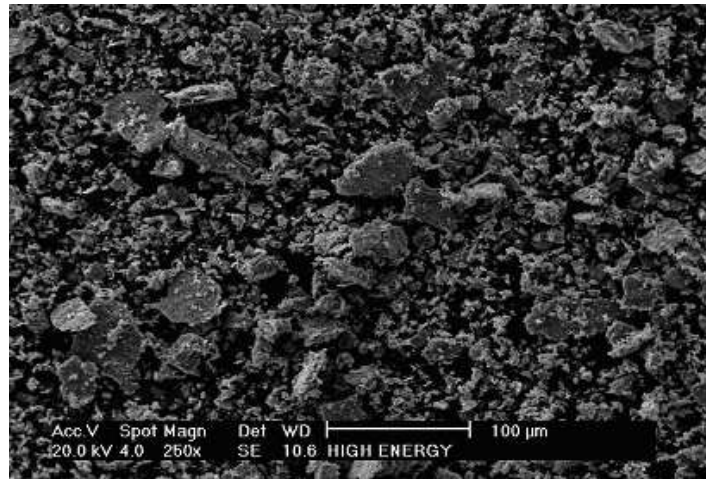
Experimental

Titanium, niobium and zirconium were heat treated under hydrogen atmosphere of 250 MPa and 1 GPa (2500 and 10000 mbar) at temperatures of 700, 600 and 500 °C, respectively, and then mechanically broken in small particles (<425 μm). The hydride powder was weighted, Ti - 74%, Nb - 13%, Zr - 13% (%wt) and milled in high energy planetary ball milling at 200 rpm for 90 minutes, leading to two groups of samples, HEPBM (hydrogenation under 250 MPa) and 10-HEPBM (hydrogenation under 1 GPa). The milled powders were isostatically pressed at 200 MPa and sintered at 1150 °C for 7, 10 and 13 hours under high vacuum.

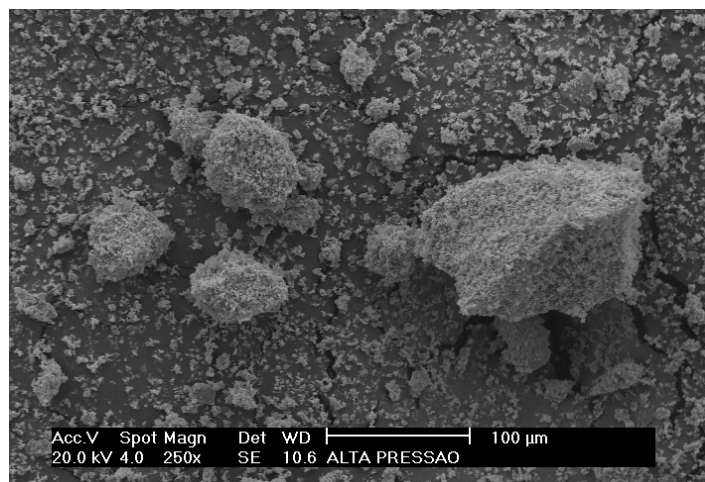
Microstructures and phases were characterized using scanning electron microscopy (SEM). Density and porosity were determined via Archimedes method using water as liquid displacement, microhardness using a Vickers microhardness tester and elastic modulus using a dynamic mechanical analyzer (DMA). Particle size distribution was determined utilizing CILAS 1064 equipment.

Results and Discussion

Fig. 1a and b show SEM micrographs of the Ti-13Nb-13Zr hydride powders milled using high energy planetary ball milling hydrogenated using 250 MPa and 1 GPa, respectively. The materials hydrogenated under 250 MPa presented some difficulties on the milling stage, resulting in some particles with larger dimensions. The hydrogenation under 1 GPa possibilities a reduction of the medium particle size of the milled materials.



(a)



(b)

Figure 1 – SEM micrographs of the Ti-13Nb-13Zr hydride milled powders in High Energy Planetary Ball Milling at 200 rpm for 90 min: (a) HEPBM hydrogenated under 250 MPa and (b) 10-HEPBM hydrogenated under 1 GPa.

Fig. 2 shows the particle size distribution of the milled materials obtained via CILAS. The hydrogenation under 1 GPa produced a more uniform particle size distribution in comparison with the powder produced from materials hydrogenated under 250 MPa, achieving a smaller medium particle size.

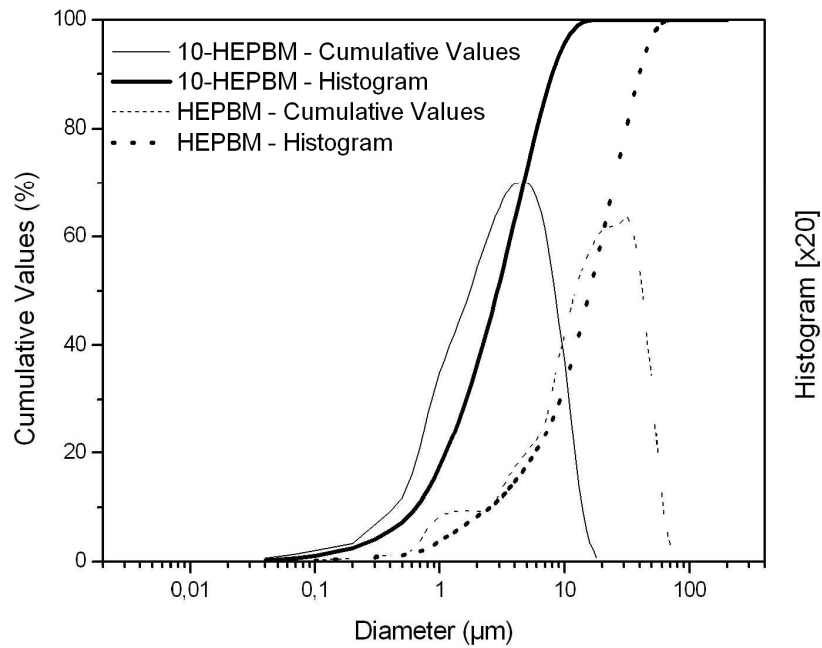
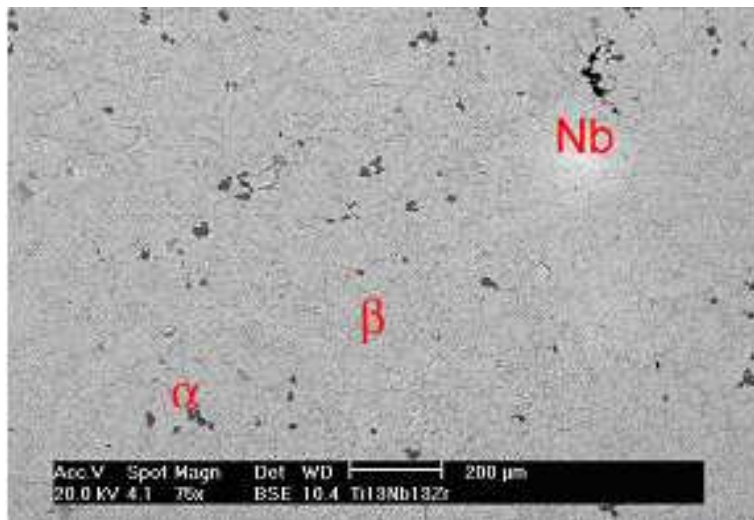
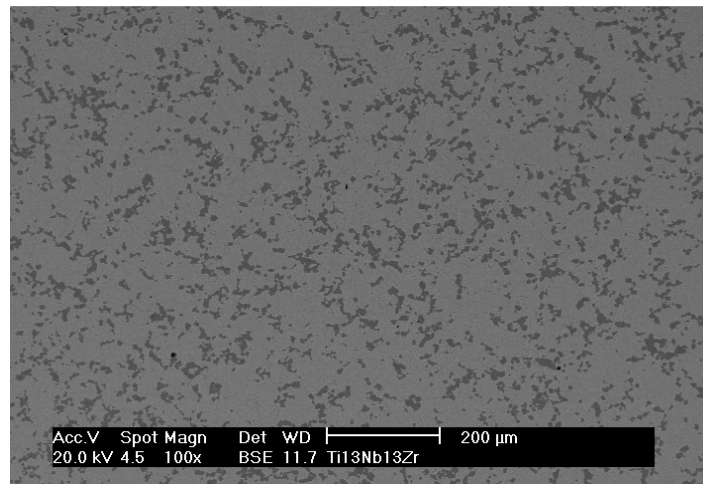


Figure 2 – Particle size distribution obtained for the Ti-13Nb-13Zr hydride milled powder via CILAS (HEPBM and 10-HEPBM).

Fig. 3a and b show the microstructure of the Ti-alloy sintered at 1150 °C for 7 h. The HEPBM sample shows a small area containing Nb free material and formation of a $\alpha+\beta$ phase. The better powder characteristics obtained via 10-HEPBM preparation method resulted in a more homogenous microstructure after sintering, and $\alpha+\beta$ phase structures are also exhibited.



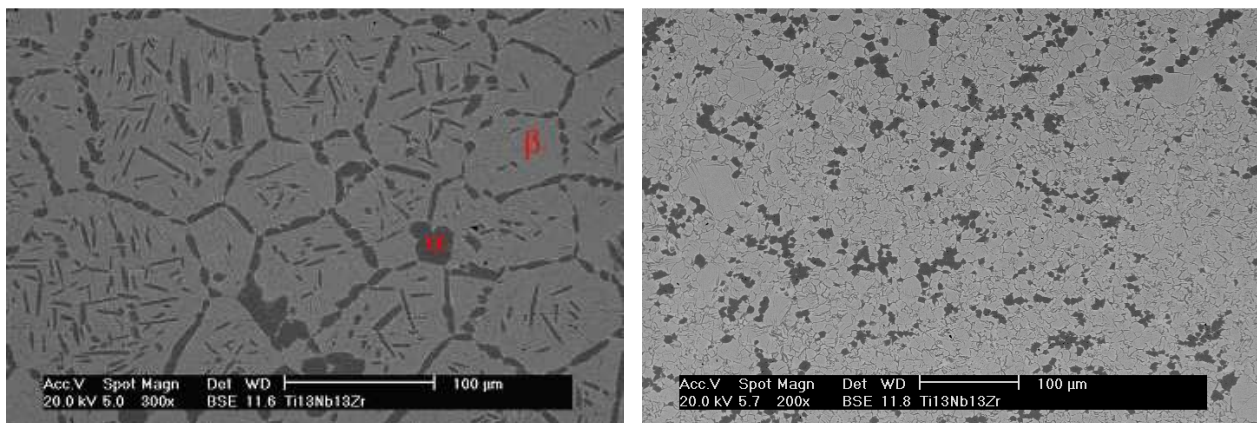
(a)



(b)

Figure 3 – Ti-13Nb-13Zr alloy produced with powder obtained via (a) HEPBM and (b) 10-HEPBM and sintered at 1150°C for 7 h.

Fig. 4a and b show the microstructures of the samples produced with HEPBM and 10-HEPBM, sintered for 10 h, respectively. In this condition, areas containing free niobium are no longer observed in the HEPBM sample. Precipitation of α phase was observed with the increasing of sintering time from 7 to 10 hours on HEPBM sample, and in a lesser scale on 10-HEPBM.



(a)

(b)

Figure 4 – Backscattered electron images of the Ti-13Nb-13Zr alloy sintered at 1150°C for 10h and produced with powder obtained via (a) HEPBM and (b) 10 HEPBM.

Fig. 5a and b show the microstructures of HEPBM and 10-HEPBM samples sintered for 13 h, respectively. Precipitation of α phase was also observed with the increase of sintering time to 13 h, but in a lesser scale on 10-HEPBM sample, similarly of 10 h. Medium grain size showed an increase with the increasing of sintering time for HEPBM and 10-HEPBM samples, however the latter still exhibits a more refined structure, with smaller medium grain size.

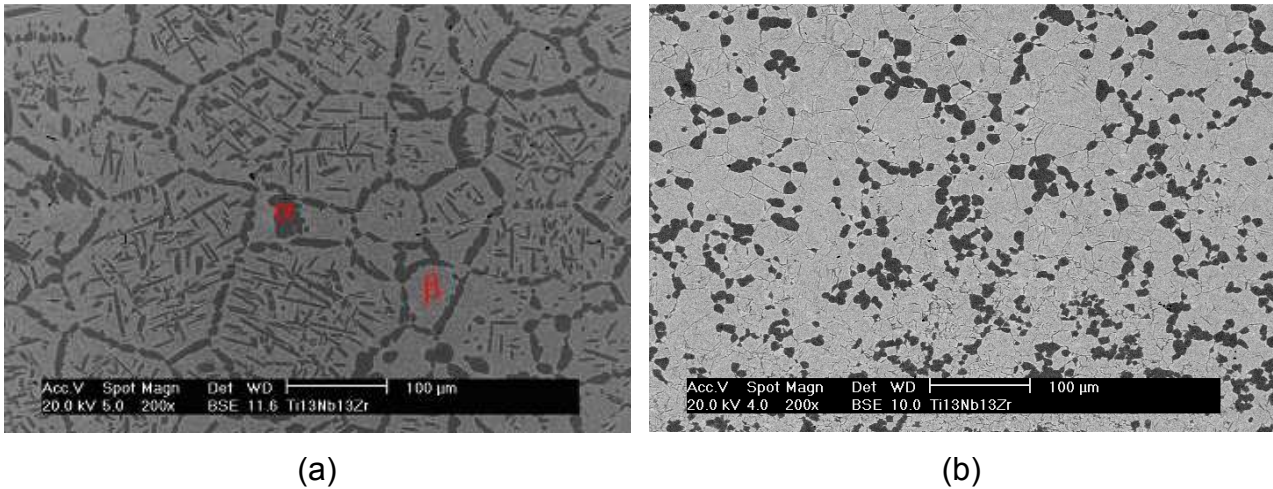


Figure 5 – Backscattered electron images of the Ti-13Nb-13Zr alloy sintered at 1150°C for 13h and produced with powder obtained via (a) HEPBM and (b) 10-HEPBM.

Density, porosity, microhardness and elastic modulus of samples produced via HEPBM and 10-HEPBM are given in Table 1. The smaller particles obtained via 10-HEPBM presented difficulties during the isostatic pressing stage. The more refined structure observed on 10-HEPBM SEM microstructures and the smaller medium grain size, contributed to a decrease in elastic modulus, in comparison with HEPBM samples. The precipitation of α phase, as well as the increase of the grain size of the structures, caused by the increase of sintering time, reflected in the rise of microhardness and elastic modulus values.

Table 1 – Density, porosity, microhardness and elastic modulus values obtained for the Ti-13Nb-13Zr alloys.

| Sample identification | Density (g/cm ³) (±0.02) | Porosity (%) (±0.2) | Microhardness (HV) | Elastic Modulus (GPa) |
|-----------------------|--|---------------------------|-----------------------|--------------------------|
| HEPBM - 7h | 4.94 | 1.3 | 437 ± 25 | 81.3 |
| HEPBM - 10h | 4.98 | 1.0 | 459 ± 25 | 88.2 |
| HEPBM - 13h | 5.00 | 0.5 | 465 ± 25 | 89.5 |
| 10-HEPBM - 7h | 4.77 | 3.1 | 415 ± 20 | 62.6 |
| 10-HEPBM - 10h | 4.78 | 2.4 | 421 ± 20 | 67.8 |
| 10-HEPBM - 13h | 4.95 | 1.8 | 494 ± 20 | 66.2 |

Conclusion

It has been shown that hydrogenation under 250 MPa and 1 GPa, and high energy planetary ball milling are efficient to produce a Ti-13Nb-13Zr alloy. The latter yielded a more uniform particle size distribution and a smaller medium particle size. Alloys produced via 10-HEPBM also showed superior microstructural homogeneity than those produced via HEPBM, and lower elastic modulus was achieved without large decreases to the microhardness. It is feasible to produce a Ti-13Nb-13Zr alloy by powder metallurgy using a sintering temperature of 1150 °C during 7 h and hydrogenation under 1 GPa.

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