

Microstructural Evolution of Ti-6Al-7Nb Alloy During High Pressure Torsion

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Ti-6Al-7Nb alloys are being evaluated for biomedical applications, in substitution of the more conventional Ti-6Al-7V. Both types of alloys present a microstructure containing the α and the β phases, which result in good compromise for mechanical applications. In the present work Ti-6Al-7Nb alloys were processed by High Pressure Torsion (HPT), varying the number of revolutions and thus the total imposed strain. X-Ray Diffraction (XRD) results revealed the formation of different crystallographic textures in samples subjected to HPT. Microhardness distribution, across the diameters of the disks, is rather homogeneous for all samples, with higher values for those subjected to 03 and 05 turns. Transmission electron microscopy (TEM) micrographs have showed that an ultra-fine grained microstructure was obtained in all the samples.

Keywords: *high pressure torsion, severe plastic deformation, titanium alloys, microstructural analysis*

1. Introduction

Ti-6Al-7Nb alloy is widely used in biomedical applications; this alloy was conceived in 1977 with the objective to create an alloy to substitute the more conventional Ti-6Al-7V, since there were concerns over vanadium toxicity. Both types of alloys present excellent biocompatibility and high specific strength. These alloys have a bi-modal microstructure containing α and β phases, which result in good compromise for mechanical applications¹.

The mechanical properties of these alloys can be enhanced through different ways, and severe plastic deformation (SPD) processes such as high pressure torsion (HPT) and equal-channel angular pressing (ECAP) are being extensively studied. HPT processing is one of the most widely used among the SPD techniques, mainly due to its effectiveness in grain refinement²⁻⁷. Another aspect being studied on the titanium processing through HPT is the formation of metastable phases due to the deformation process. The main phases being reported by the literature are the ω and the martensite α' phase, these phases can have an effect on the tenacity of Ti alloys and also affect their elastic modulus, an important property for biomedical applications⁸⁻¹⁰.

During HPT, a sample in the shape of a disk is compressed between two anvils with a pressure of several gigapascals, and then it is subjected to a torsional strain which is imposed by the rotation of one of the anvils. The sample is deformed by shear due to surface frictional forces,

the deformation process proceeds under a quasi-hydrostatic pressure⁴. The most important parameters concerning HPT processing are the number of turns and the applied pressure. These parameters have direct influence on the grain refinement and microstructural evolution during HPT⁶. The influence of the number of turns has been investigated by the present work.

2. Experimental Procedures

The Ti-6Al-7Nb alloy was obtained through vacuum arc-melting commercially pure elements. An 8 mm rod from the alloy was made using copper-mold casting; this rod was machined and cut as thick disks that could fit in a 7 mm cavity in the anvil. The as-cast samples were subjected to an applied pressure of 5 GPa and then processed by constrained HPT using 02, 03 and 05 turns at room temperature.

The microstructural evolution was analyzed using transmission electron microscopy (TEM), X-ray diffraction (XRD) and measurements of local microhardness.

Samples for transmission electron microscopy were first grinded and dimpled, and then the thin foils were obtained with Gatan Precision Ion Polishing System - PIPS. The analysis was performed using a TEM FEI Company – TECNAI operated at 200 kV and a TEM Philips CM-120 operated at 120 kV. Selected area electron diffraction (SAED) analyses were carried out using standard camera length. The selecting of diffracted spots by smaller objective aperture available (10 μm size) has allowed obtaining dark field (DF) micrographs, which

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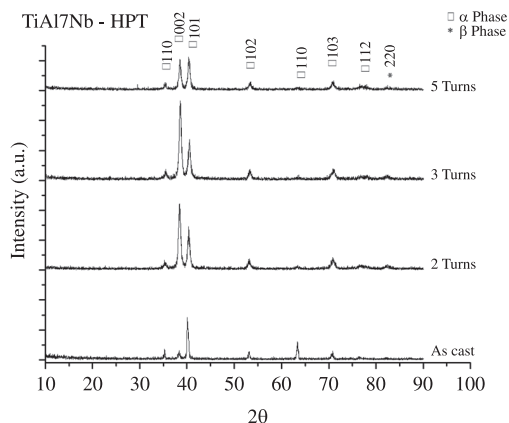


Figure 1. X-ray diffraction patterns for Ti6Al7Nb processed by HPT with different number of turns.

were applied to investigate ultrafine grain microstructure. That experimental condition implies that diffracted beams came from regions with crystallographic misfit lower than 15 degrees in a polycrystalline TEM sample (ring-type electron diffraction pattern). As a consequence, sub-grain size measurement was conducted by averaging the equivalent diameters (Feret) of the contrast domains observed in dark field images, taken from different areas in the thin foil samples.

X-Ray Diffraction was conducted with a Siemens DL5005 diffractometer operated with monochromatic Cu K α 1 radiation. Diffraction spectra in 10-90 $^{\circ}$ range were collected with a step of 2 degrees per minute.

Vickers microhardness measurements were made along the diameter of the specimens using a Brivisor KL2 device under a load of 200 g for 15 seconds.

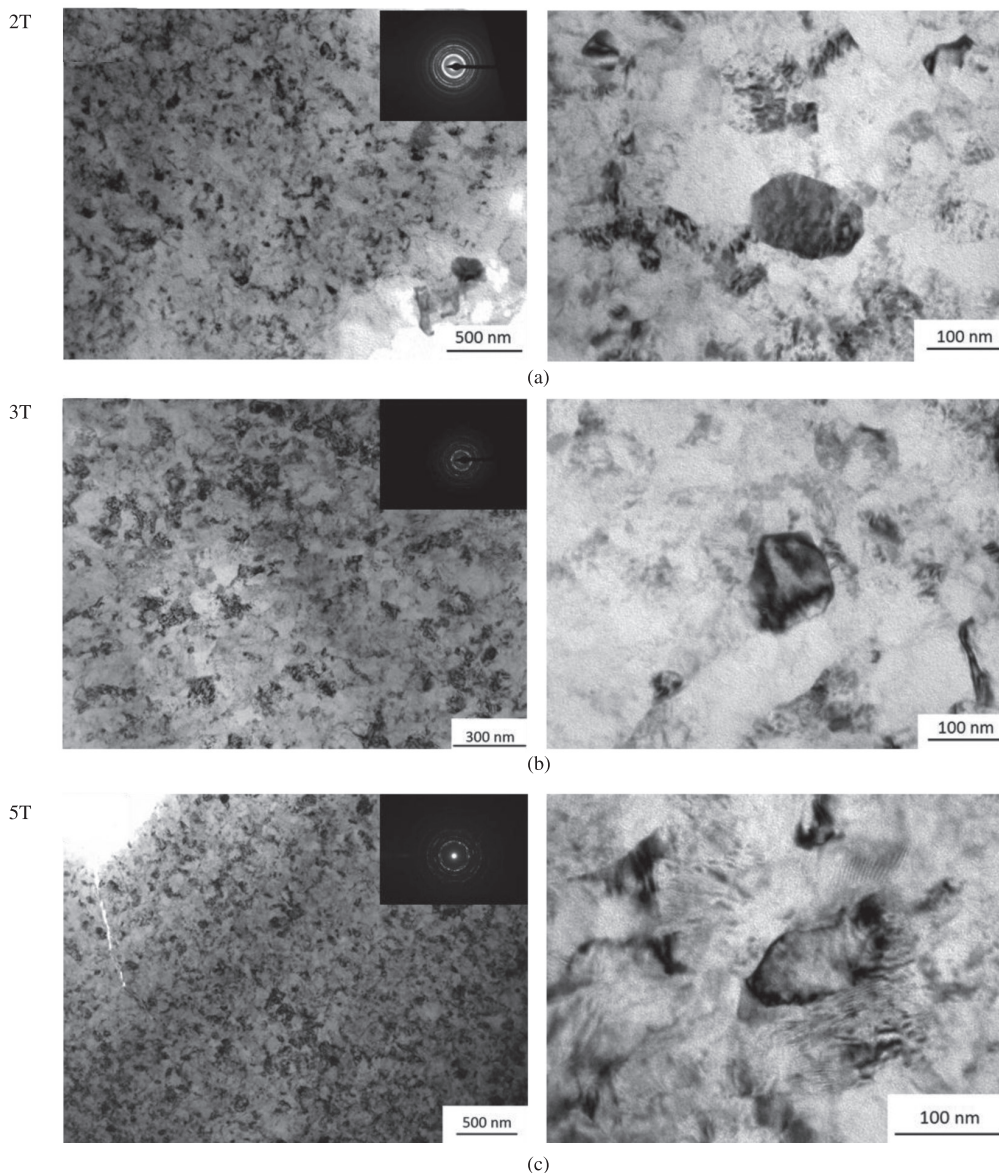


Figure 2. TEM bright-field micrographs and SAED patterns for samples processed by HPT after: 2 turns (a), 3 turns (b) and 5 turns (c).

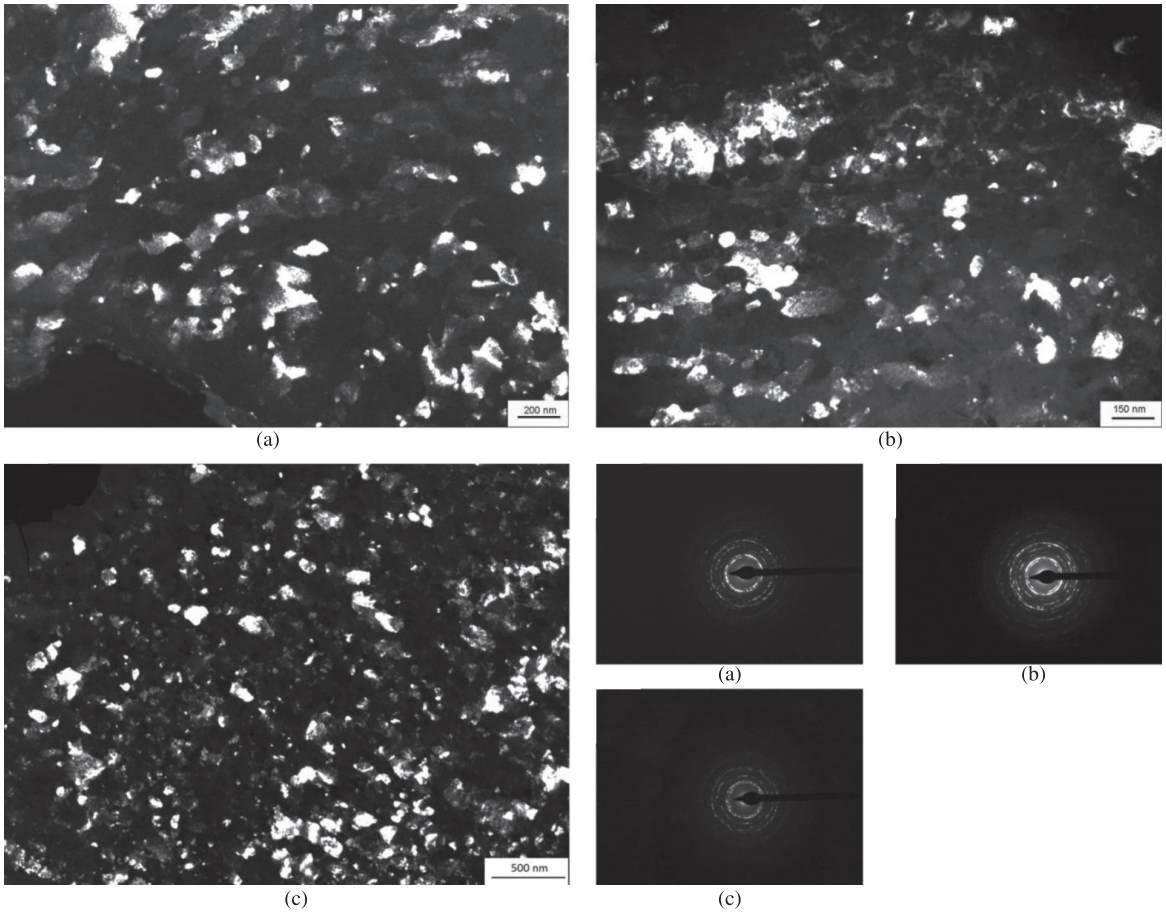


Figure 3. TEM dark-field and the corresponding SAED patterns for samples processed by HPT after: 2 turns (a), 3 turns (b) and 5 turns (c).

3. Results and Discussion

The microstructure of the as-cast rod consisted of equiaxed prior- β grains with a variety of α -morphologies within these grains. The average prior- β grain size was within the 300-500 μm range. The mean value for Vickers microhardness of the as-cast rod was 190 HV.

The XRD patterns present in Figure 1 indicate the α -phase as the main phase in the studied alloy. The broadening of the XRD peaks can be associated with the microstructural refinement obtained by HPT, this feature was observed in all the samples. It was also observed the formation of a crystallographic texture in the samples, which is more prominent in those subjected to 02 and 03 turns; samples subjected to 05 turns develop a different texture, possibly due to a different competition stage between deformation and recovery, which can result in a saturation of grain refinement⁴.

The TEM bright-field micrographs and SAED patterns in Figure 2 and the dark-field micrographs in Figure 3 show the result and characteristics of the grain refinement process. These micrographs show an ultrafine grained (UFG) microstructure present in all samples, although sub-grain size measurements show that samples processed by 03 and 05 turns have finer microstructure, with average size of 97 nm and 107 nm respectively, while the sample processed by 02 turns has an average sub-grain size of 160 nm. SAED

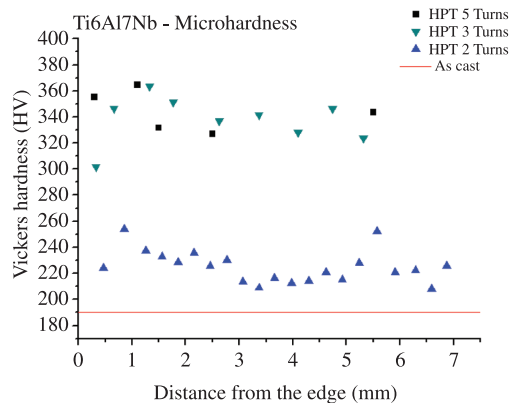


Figure 4. Microhardness profiles along the diameter of samples processed by HPT.

ring-like patterns suggest the coexistence of both high-angle and low-angle boundaries.

The lack of further grain refinement after 03 turns indicates, as did the XRD results, that a different competition stage between recovery and deformation is reached. Upon this stage, further plastic deformation does not introduce new lattice defects, in the form of vacancies and dislocations, but is transformed in heat, which promotes the recovery phenomenon⁴.

A typical feature of the microstructures is the presence of non-equilibrium boundaries which are characterized by being curve/wavy and poorly delineated. Another feature is the presence of complex contrast inside the grains, suggesting large internal stresses. Both features are considered typical of the UFG structures achieved by severe plastic deformation processes^{4,5}.

The presence of metastable phases, such as the ω and the α'' , were not observed in any of the alloys processed by HPT, although the lack of this observation could have been caused by the difficulties inherent to the TEM analysis of highly deformed samples or even by the low volume fraction of these phases in the samples.

The results of microhardness measurements, Figure 4, show that the SPD process has enhanced the mechanical properties of the alloy. When compared to the "as cast" condition, hardness was increased by 17.40%, 75.06% and 78.70%, after 02, 03 and 05 turns respectively. The distribution of hardness values is rather homogeneous for all samples.

These results were expected and confirm the XRD and TEM investigations. The high imposed strain enhances the mechanical properties until there is a saturation level in microhardness, which can be attributed to the saturation of microstructural refinement.

4. Conclusions

The severe plastic deformation process using HPT proved to be an effective way of refining the microstructure of Ti-6Al-7Nb alloy, enhancing mechanical properties, manifested by the increase in hardness of 78.70% after 05 turns. Observation of microstructural evolution suggests that the grain refinement increases with the imposed strain until, at least, 03 turns. After 03 turns, as indicated by the results of 05 turns samples, it appears that the dynamic recovery process reaches a quasi-equilibrium stage with the deformation process, where further plastic deformation does not introduce new defects to the structure, leading to a saturation of the grain refinement process.

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