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STRUCTURE OF NANOCRYSTALLINE Ti-BASE ALLOYS OBTAINED BY MECHANICAL ALLOYING AND ULTRA HIGH PRESSURE SINTERING

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Abstract. The Ti-base alloys with additions of Ta and Nb had been ball milled and subsequently sintered using ultra high pressure Bridgeman method. The milled powders with structure of α-Ti solid solution and average crystallite size of 10 nm and particle size of 5 μm were hot pressed under 4 GPa for 1 min at temperature 650 °C. This technique allowed preserving the structure of the examined alloys at a nanometric level. The grain size of alloys after ultra high pressure (UHP) sintering changed from 25 up to 90 nm with an increase of content of the elements stabilizing the β phase in the alloy. The nucleation and growth of the β phase during sintering were major factors, which controlled the final structure of the sintered alloy. The Ti alloys containing additions of either Ta or Nb consisted mainly of the α-Ti structure with different fractions of β-Ti phase, whereas additions of both Ta and Nb generally increased the fraction of the β-Ti phase. Conventional TEM diffraction studies confirmed the presence of α-Ti and β-Ti phases in the investigated powders and HREM was applied to measure the grain size of both α and β phases.

1. INTRODUCTION

Among biomedical metallic materials, pure titanium and titanium alloys (Ti–6Al–4V ELI) are considered more suitable for implant applications as compared to other alloys such as 316L stainless steel and cobalt-base ones because of their lower elastic modulus similar to that of a bone [1]. They reveal good biocompatibility, corrosion resistance and high mechanical properties. Some biocompatible elements like Ta, Zr, Nb, Fe, Cr have found application as the additions to titanium alloys instead of vanadium and aluminum recently considered as toxic elements [2,3]. It has been well known that titanium and tantalum are difficult to melt even in inert atmosphere furnaces, because of their high reactivity and different specific weights. That is why new techniques like cold crucible levitation melting [2] or mechanical alloying [4,5] are elaborated for production of these alloys. This paper presents results of structure examination of titanium alloys produced by mechanical alloying and subsequent ultra high pressure sintering.

2. EXPERIMENTAL

Powders of titanium (of 110 μm size and purity > 99.9%), tantalum (of 150 μm size and purity 99.98%) and niobium (of 100 μm size and of purity > 99.8%) were used as starting materials. The powders were initially blended to the desired compositions of Ti-10Ta, Ti-10Nb and Ti-10Ta-10Nb (in at.%) under argon atmosphere in a glove-box and subjected to ball milling for up to 80 hrs in a high energy planetary mill (Fritsch Pulverisette P5/4) and then sintered at 650 °C under ultra high pressure of 4 GP using the Bridgeman method. The structure of sintered samples were examined in a Philips

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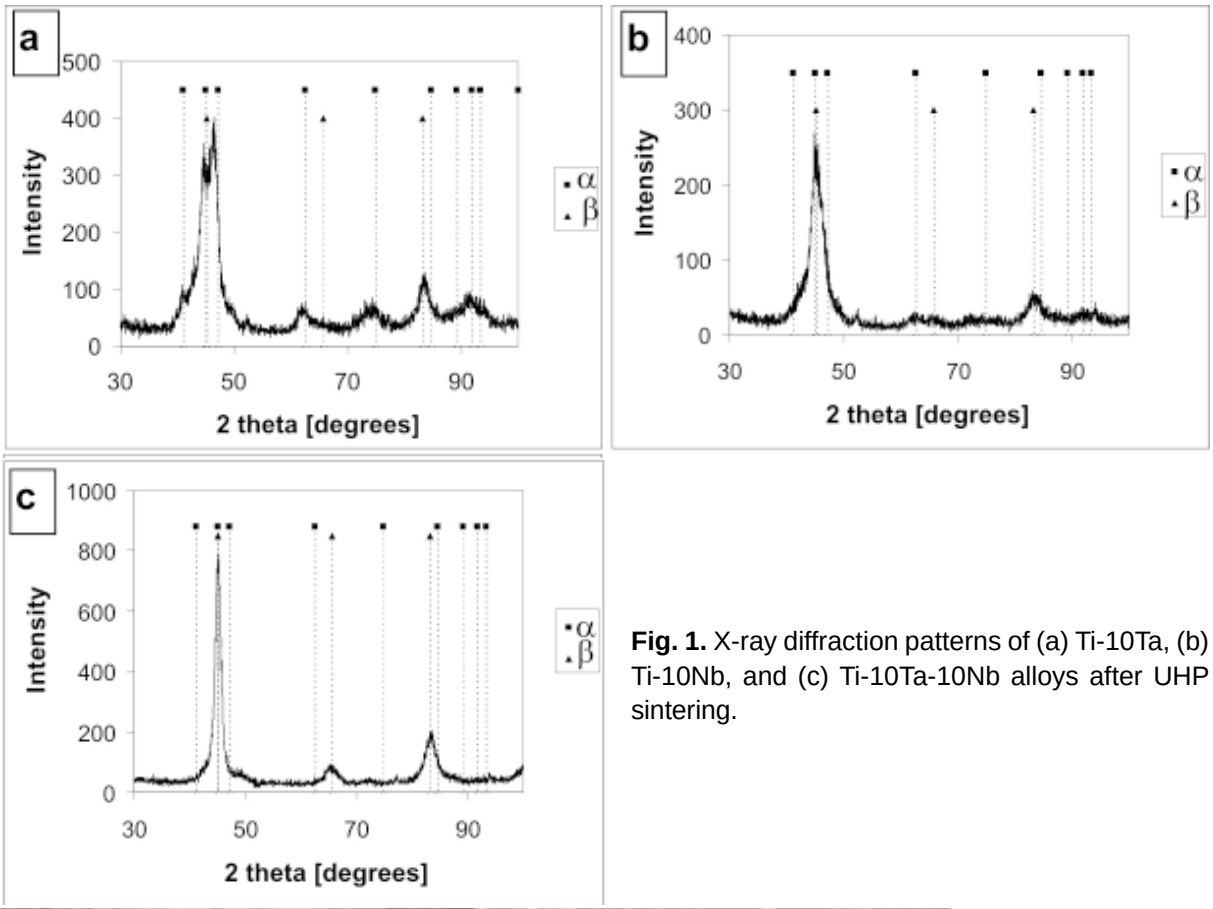

Fig. 1. X-ray diffraction patterns of (a) Ti-10Ta, (b) Ti-10Nb, and (c) Ti-10Ta-10Nb alloys after UHP sintering.

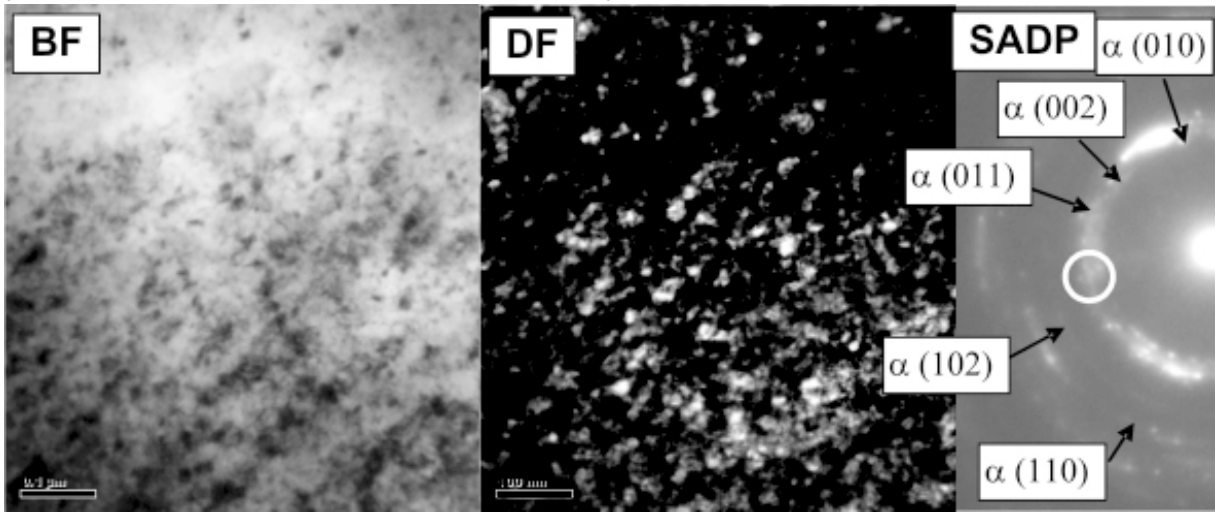

Fig. 2. TEM bright, dark field (BF, DF) micrographs and corresponding selected area diffraction pattern of Ti-10Ta (SADP) alloy after UHP sintering.

PW 1830 diffractometer using Cu K_{α} radiation and transmission electron microscopy (TEM) in a Philips CM 20 equipped with a Phoenix energy-dispersive X-ray analysis system or Technai G2 FEG for high resolution. Thin foils of sintered samples were prepared by dimpling and ion beam milling using Gatan equipment.

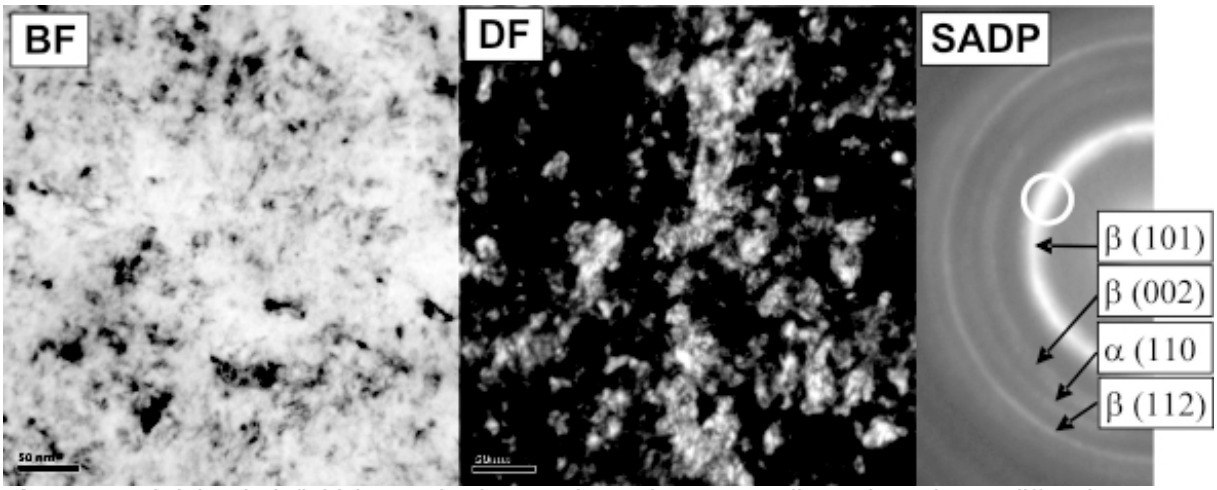

Fig. 3. TEM bright, dark field (BF, DF) micrographs and corresponding selected area diffraction pattern (SADP) of Ti-10Nb alloy after UHP sintering.

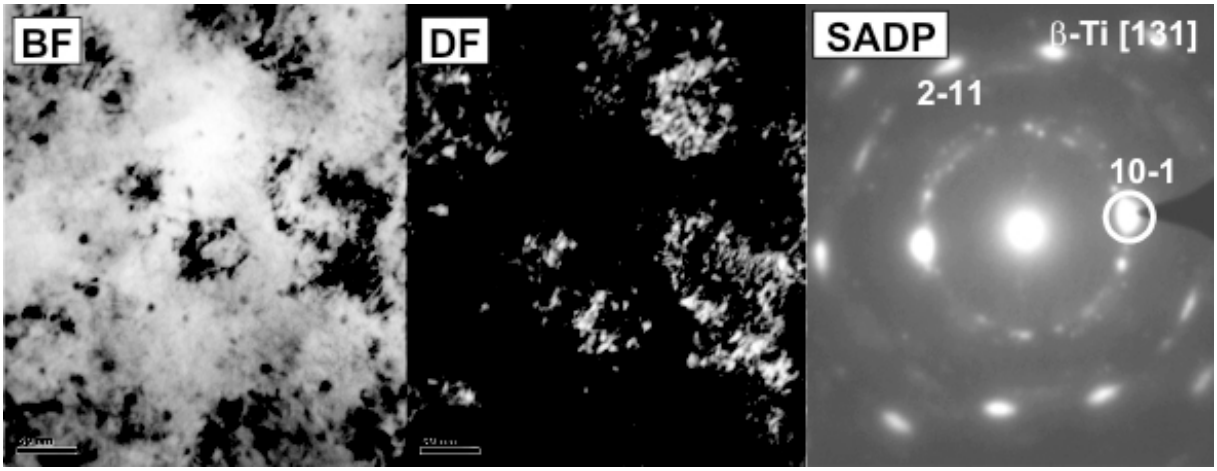

Fig. 4. TEM bright, dark field (BF, DF) micrographs and corresponding selected area diffraction pattern (SADP) of Ti-10Ta-10Nb alloy after UHP sintering.

3. RESULTS AND DISCUSSION

The phase transformations, structure of powders and their chemical homogeneity after the ball milling process were presented in previous papers [6,7]. Generally, the ball milling process of the investigated alloys led to the formation of the mixture of amorphous and nanocrystalline metastable α-Ti solid solution with average crystallite size of 10 nm and particle size of 5 μm with small variation in chemical composition of particulate particles. Because the consolidation of the milled powders by pulse plasma sintering brought about the formation of two-phase α + β structure with a mean grain size of 220 nm [7] another technique called the Ultra High Pressure (UHP) based on Bridgeman method was applied for the consolidation of milled powders in order to retain the nanocrystalline structure in bulk samples. Fig. 1 presents the X-Ray diffraction patterns of the UHP samples. The nanocrystalline character of the samples is visible in the all patterns demonstrated by relatively low intensities of the diffracted peaks as well as their broadened shape. The highest intensities of peaks and their smallest broadening were detected in the Ti-10Ta-10Nb alloy.

The crystallographic structure changed with the chemical composition of alloys. In the Ti-10Ta alloy two-phase α+β structure existed with the α phase dominating, in the Ti-10Nb alloy the two-phase α+β structure was also observed but with

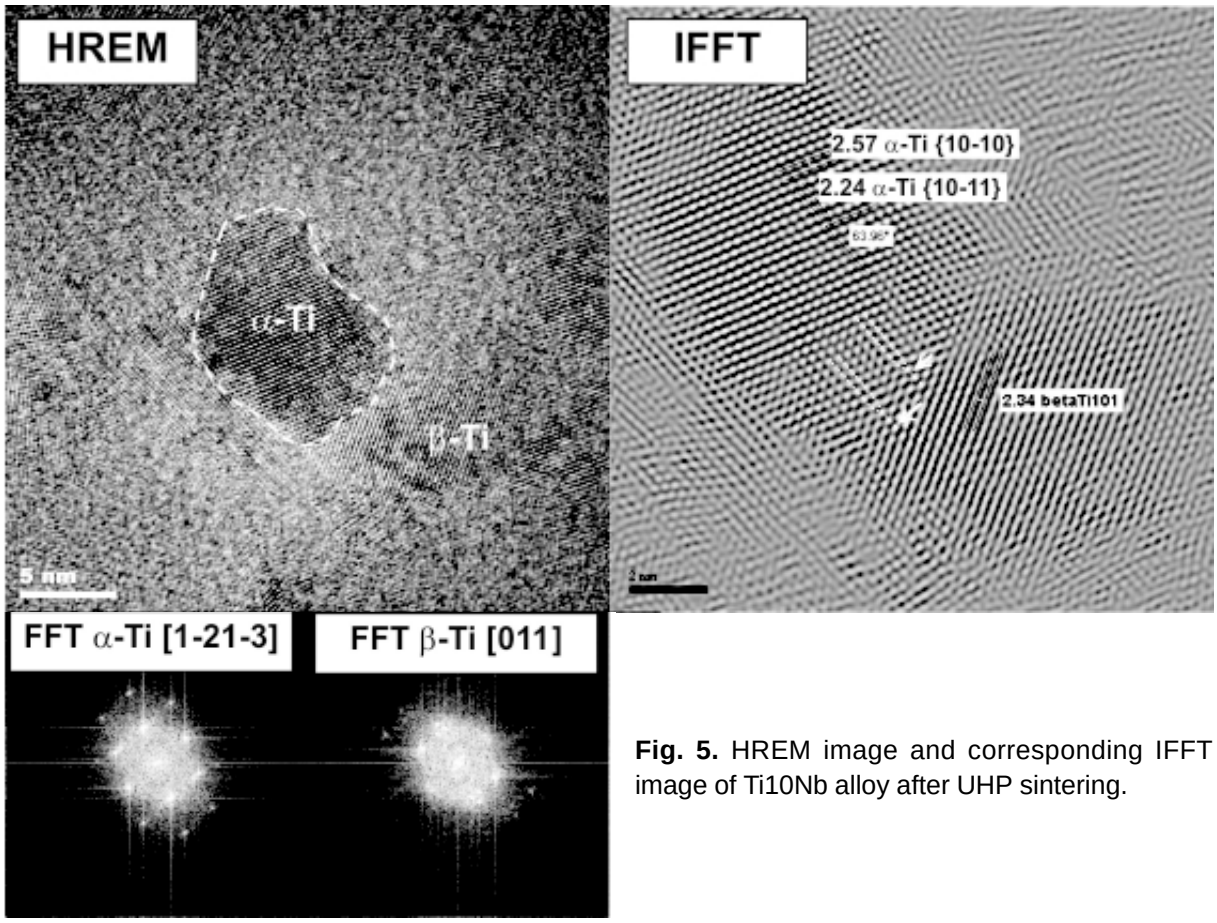

Fig. 5. HREM image and corresponding IFFT image of Ti10Nb alloy after UHP sintering.

slightly larger fraction of the β phase. In the Ti-10Ta-10Nb alloy the β phase structure was mainly detected with a minor content of the α phase. Figs. 2-4 show sets of TEM macrographs, taken using bright (BF) and dark field (DF) using objective aperture placed as marked by the white circle, and corresponding selected area diffraction patterns (SADP) of the sintered alloys. It can be seen that for the Ti-10Ta alloy (consisting mainly of the α phase) the grain size was about 25 nm, but when the phase composition changed to the β+α or β phase the grain size increased up to 50 and 90 nm for the Ti-10Nb and Ti-10Ta-10Nb alloys, respectively. Therefore it can be concluded that although the milled powders had similar structure (the mixture of amorphous and metastable α-Ti solid solution) before sintering, the formation of α phase affected the final grain size of the compacts. The reason for the increase of grain size in alloys containing more β phase is the nucleation and growth processes of the β phase during sintering at 650 °C because the temperature of allotropic transformation β→α (883 °C) shifted to lower temperatures by the Ta or Nb additions. When the contents of the elements which stabilized the β were too small for the nucleation of the β phase, the a structure prevailed because the sintering time was very short (about 1 min) at relatively low temperature. The differences in structure between the alloys with 10 at.% addition of Ta and Nb can be also explained taking into account the binary Ta-Ti and Nb-Ti phase diagrams [8]. The addition of 10 at.% Ta to titanium shifted the β→α transformation temperature down to about 740 °C whereas 10 at.% of Nb even more, to about 700 °C, which could be compensated by high pressure.

Fig. 5 presents the HREM image and corresponding IFFT image of the Ti-10Nb alloy after the sintering process. It allowed the assessment of the grain size of the α phase at the level of about 5

nm, almost the same as in the powders. Due to the high pressure consolidation, high stresses were introduced into the sample which was manifested in the HREM image by the strain field contrast. Additionally, the interface dislocations (marked by arrows) at the interface between the α-Ti and β-Ti grains accommodating $(\bar{1}01\bar{1})$ α-Ti lattice planes parallel to (211) β Ti correspond to the slip system $(11\bar{2}2)$ $[\bar{1}\,\bar{1}23]$ reported also in [9]. Hexagonal pyramidal planes were also found in this alloy. The interface dislocations might have played an important role in the deformation process increasing the plasticity of compacts.

4. CONCLUSIONS

1. The ultra high pressure consolidation led to a different structure of alloys depending on the chemical composition of the sintered alloy. In the Ti-10Ta alloy two-phase α+β structure existed with the α phase as a dominating one, in the Ti-10Nb alloy the two-phase α+β structure was also observed but with a slightly larger fraction of the β phase and the Ti-10Ta-10Nb alloy consisted mainly of the α phase with a minor content of the α phase.
2. The grain size of alloys after UHP sintering changed from 25 up to 90 nm with an increase of content of the elements stabilizing the β phase in the alloy. The nucleation and growth of the β phase during sintering were major factors, which controlled the final structure of the sintered alloy.
3. The interface dislocations were observed in the $\{10\bar{1}1\}$ pyramidal planes of the α-Ti phase at the interface with the β-Ti one.

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