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STRUCTURE AND PROPERTIES OF NANO-CRYSTALLINE Ti-BASE ALLOYS OBTAINED BY VACUUM HOT PRESSING

ABSTRACT

The cp-Ti and Ti-base alloys with additions of Ta and Nb were ball milled and consolidated using vacuum hot pressing. This novel technique allowed to obtain a high level of densification of milled powders up to about 98% and the nanometric grain size level. In the samples of vacuum hot compacted cp-Ti grain size of a single α phase was estimated at 140 nm. With the increase of content the β-stabilizing elements in alloys such as Ta and Nb, structure and a grain size has been changed. In the case of Ti-5Ta-5Nb alloy, also single α phase was observed but with grains size was much smaller, close to 85 nm. The further increasing of the content of Ta and Nb caused further refinement of grain size down to 60 nm and change of structure into two phase α+β and β in case of Ti-10Ta-10Nb and Ti-15Ta-15Nb alloys respectively. The hardness and Young Modulus were measured using the dynamic hardness tester and calculations of hardness and elastic modulus values were based on Oliver and Pharr model.

Key words: *Ti-base alloys, mechanical alloying, powder consolidation, TEM microstructure*

INTRODUTION

Titanium and its alloys have been widely used in the aircraft, aerospace and other industries, like biomedical or automotive. However, titanium alloys cannot be used on a large scale in the automotive industry, unless a cost of titanium alloy parts is lowered to an acceptable level. Powder metallurgy (PM) is one of technological processes, that lower the cost of titanium alloy parts since 1970s [1]. PM of some titanium parts have been used successfully for specific applications [2–4]. Such titanium alloys can be classified into three categories: pre-alloyed PM Ti alloys, rapidly solidified PM Ti alloys, and blended elemental PM Ti alloys [5]. Blended elemental powder is considered as much more cost-effective due to a relatively low cost of Ti and other elemental powders. Mechanical alloying process of blended elemental powders is becoming more widely used due to a possibility of formation of a metastable, nanocrystalline, chemically homogenous structures [6]. Another aspect of cost reduction is the feasibility of achieving nearly fully dense Ti alloys by a simple sintering method. In most cases, HIP-ping is involved in processing PM Ti alloy parts using pre-alloyed powder, however it makes the materials processing much more costly. Therefore the new alternative methods like pulse plasma sintering [7], uni-axial hot pressing

combined with vacuum annealing [8], hot pressing under extremely high pressure [9] or vacuum hot pressing are under development.
The aim of this paper was to determine a technology of hot vacuum compaction and of the structure and the mechanical properties of Ti-base alloys compacted by mechanical alloying and hot vacuum pressing.

EXPERIMENTAL

Powders of titanium (110 μm size and of purity > 99.9 %), tantalum (150 μm size and of purity 99.98 %) and niobium (10 μm size and of purity > 99.8 %) were used as a starting materials. The powders were initially blended to desired compositions of cp-Ti, Ti-5Ta-5Nb, Ti-10Ta-10Nb and Ti-15Ta-15Nb (numbers indicate at. %) in a glove-box under argon atmosphere and subjected to ball milling up to 80 hrs in a high energy planetary mill (Fritsch Pulverisette P5/4). Subsequent Vacuum Hot Pressing (VHP) at 650°C under 400MPa was applied. The duration time of sintering after reaching the pressure and temperature was 5 min. The operated vacuum during sintering was 10^{-2} torr. Structure of consolidated samples was studied a Philips CM 20 transmission electron microscope (TEM) equipped with a Phoenix energy-dispersive X-ray analysis system. Thin foils from hot pressed samples were prepared by dimpling and ion milling using Gatan 660 Duo Mill. The dynamic microhardness test was performed on CSEM Mikro-Combi-Tester with load of 100 mN.

RESULTS AND DISCUSSION

Investigations started with 80 hrs milled cp-Ti powder considered as the reference material. Fig. 1 presents the set of bright and dark field (BF and DF) TEM micrographs and corresponding selected area diffraction pattern (SADP) of cp-Ti after VHP process.

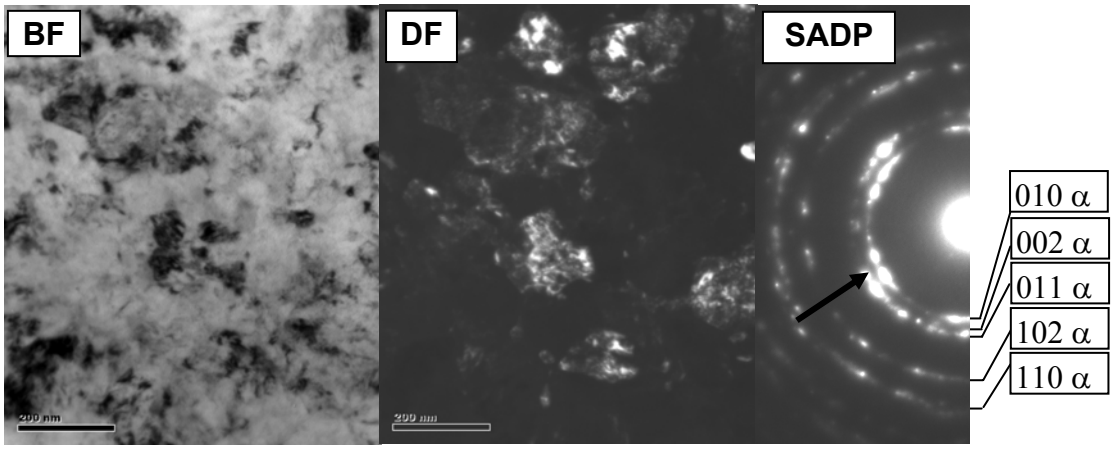

Fig.1. Bright and dark field (BF and DF) TEM micrographs and corresponding selected area diffraction pattern (SADP) of cp-Ti after VHP process

As results from the SADP image in this sample one phase structure of α-Ti type can be identified. The DF image taken high intensity part (arrow) of {010}, {002} and {011} reflections shows individual grains of α-Ti. The mean grain size has been measured using several DF micrographs and was determined at 140 nm. The TEM microstructure of Ti-5Ta-5Nb alloy after VHP process is presented in Fig. 2. In this case also only one phase α-Ti structure was observed. However the grain size was reduced down to 85 nm.

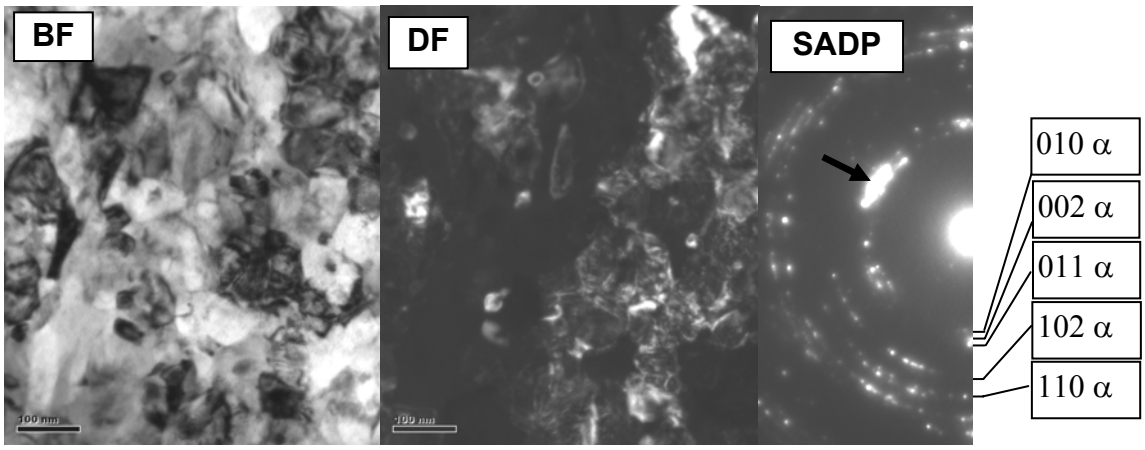

Fig.2. Bright and dark field (BF and DF) TEM micrographs and corresponding selected area diffraction pattern (SADP) of Ti-5Ta-5Nb alloy after VHP process

The elemental mapping images of Ti, Ta and Nb are presented in Fig. 3. The chemical point analyses performed in regions with different chemical contents of elements shows two types of grains with following compositions: Ti-4.3Ta-.7Nb and Ti-12Ta-13Nb. Additionally the Nb is more homogenously distributed then either the Ti or Ta. It can be concluded that some grains possess the β structure due to diffusion controlled process of nucleation of the β phase in enriched in Ta and Nb regions in this alloy.

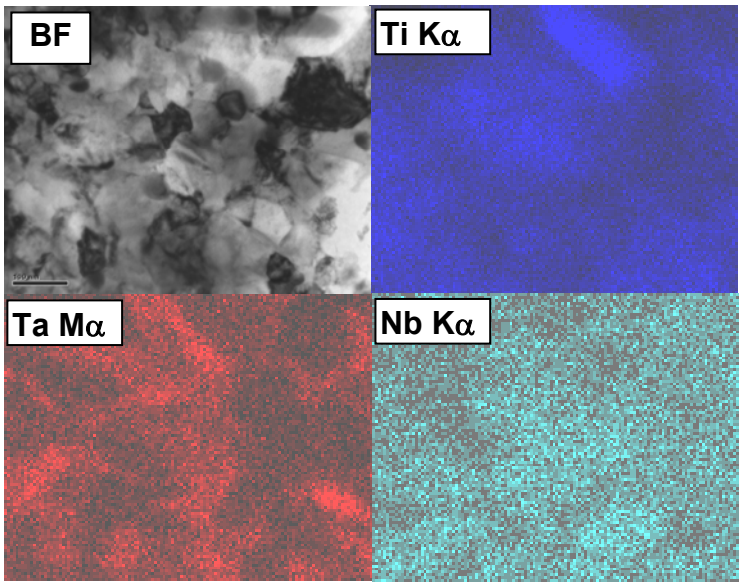

Fig. 3. Elemental mapping images of Ti-5Ta-5Nb alloy after VHP process

Further increasing of the amount of β stabilizing elements up to 10 at.% of Ta and Nb did not change the grain size but caused formation of two phase α+β structure. Fig. 4 presents a set of BF and DF TEM micrographs and corresponding SADP of Ti-10Ta-10Nb alloy after VHP process. The mean grain size was measured at 85 nm. Two phase α+β structure is identified using SADP. The DF image taken using both α and β reflections indicates that two types of grains can be distinguished; the larger ones above 100 nm and a smaller below 100 nm. The smaller ones has been determined as the β phase what is shown in Fig. 5, where the BF image and corresponding SADP from the individual β grain of [001] orientation can be seen. This indicates that nucleation of β phase and increase of its fraction in the structure may lead to further refinement of the grain size. This assumption has been confirmed in Ti-15Ta-15Nb alloy where only one phase β structure with the mean grain size of about 60 nm was observed (Fig. 6). The DF image taken from β{101} plane shows very small rounded grains. In contrast to the alloys with the lower content of β stabilizing elements, one phase β structure was characterized by the best homogeneity determined by elemental mapping.

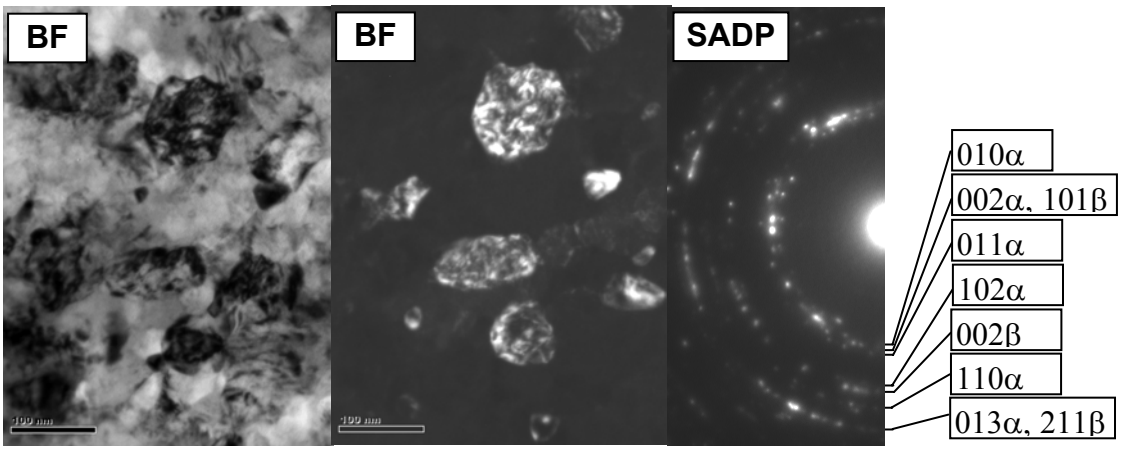

Fig.4. Bright and dark field (BF and DF) TEM micrographs and corresponding selected area diffraction pattern (SADP) of Ti-10Ta-10Nb alloy after VHP process

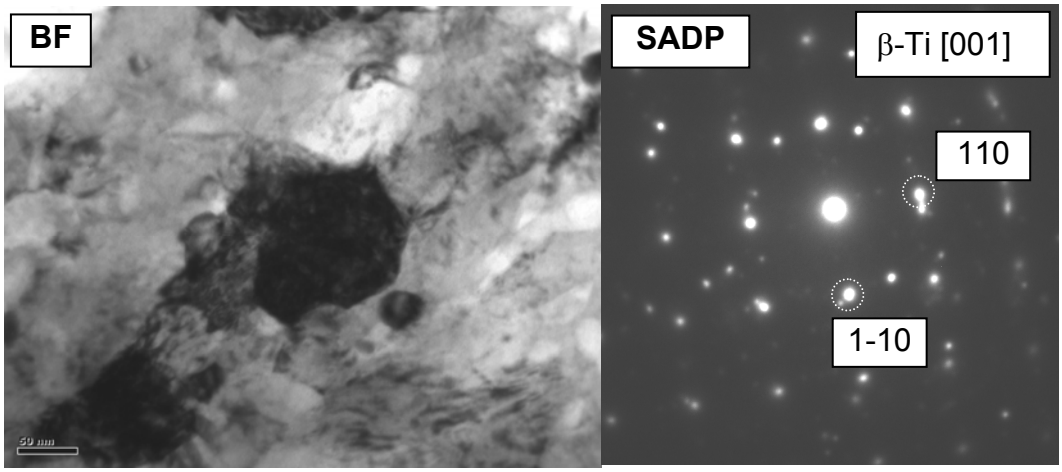

Fig.5. Bright field BF TEM micrograph and corresponding selected area diffraction pattern (SADP) of Ti-10Ta-10Nb alloy after VHP process

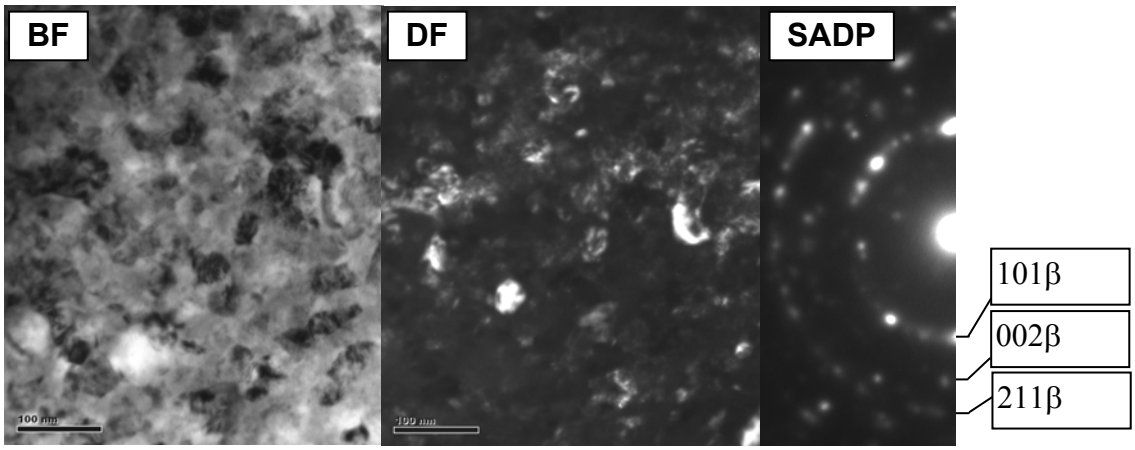

Fig.6. Bright and dark field (BF and DF) TEM micrographs and corresponding selected area diffraction pattern (SADP) of Ti-15Ta-15Nb alloy after VHP process

The mechanical properties (hardness and Young Modulus) of sintered alloys have been determined in the dynamic hardness tests. Fig. 7 presents a graph of hardness and Young Modulus versus the alloy's composition.

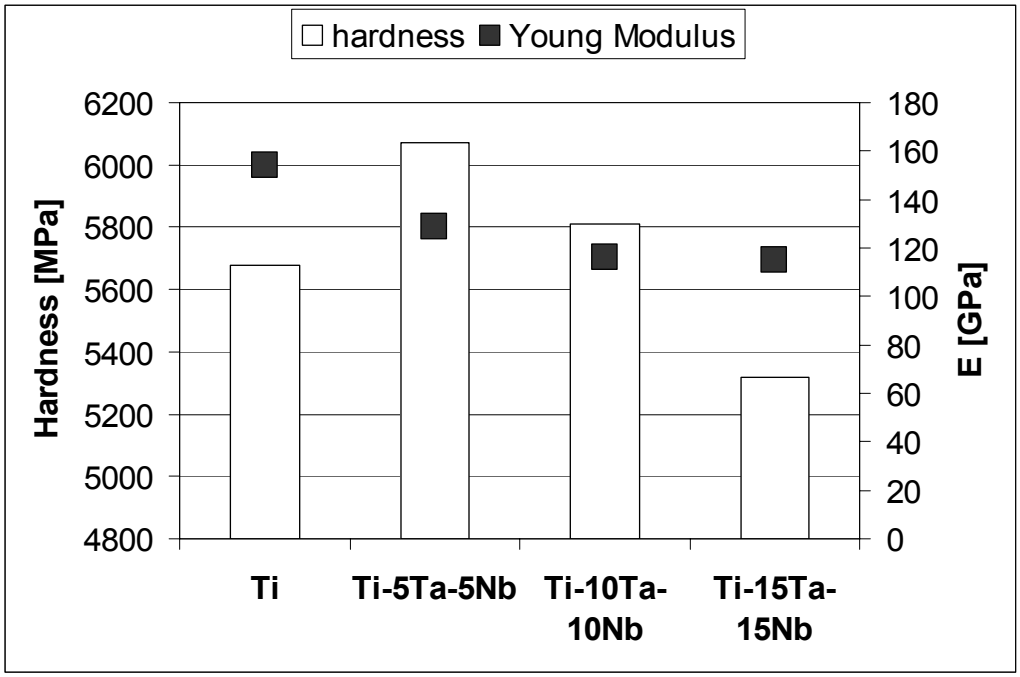

Fig.7. Hardness and Young Modulus versus alloy's composition

Two different trends are visible on the graph. The hardness increases initially (from 5700 MPa for cp-Ti) with an increase of the β stabilizing elements and reaches maximum (6200MPa) for Ti-5Ta-5Nb alloy, then decreases down to 5300 MPa for Ti-15Ta-15Nb alloy. The first increase can be attributed to the solution hardening due to solution of Ta and Nb in the α phase and to grain size decrease as results from Hall-Petch relationship, since both cp-Ti and Ti-5Ta-5Nb alloys have the same α phase structure. The decline of hardness in alloys with higher concentration of Ta and Nb most probably is due to formation of the β phase. Another trend was observed in the case of changes of Young Modulus. The highest value of 152 GPa was found for cp-Ti,

then decrease down to 112 GPa for Ti-15Ta-15Nb alloy was observed. These changes are associated with the phase transition and finally different structure of alloys.

CONCLUSIONS

1. Nanometric α and β grain structure in Ti-base alloys with Ta and Nb additions compacted using VHP technique from mechanically alloyed powders was observed.
2. In the compacted cp-Ti single α phase structure was found with the grain size estimated at 140 nm. With an increase of a content of the β-stabilizing elements in alloys the phase structure and grain size has been changed. In the case of Ti-5Ta-5Nb alloy, also single α phase was observed but with smaller grain size of 85 nm. Further increase of the content of Ta and Nb causes refinement of grain size down to 60 nm and a change of the structure into α+β and β in the case of Ti-10Ta-10Nb and Ti-15Ta-15Nb alloys respectively.
3. Two different trends of changes of hardness and Young Modulus were observed in VHP Ti-base alloys. The hardness increases initially (from 5700 MPa for cp-Ti) with an increase of β stabilizing elements and reaches maximum (6200 MPa) for Ti-5Ta-5Nb alloy, then decreases down to 5300 MPa for the β phase Ti-15Ta-15Nb alloy. The highest Young Modulus value of 152 GPa was reported for cp-Ti and decreases down to 112 GPa for the β phase Ti-15Ta-15Nb alloy.

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