# Effect of microstructure state of titanium alloy Ti-6Al-4V on structure and mechanical properties of joints produced by diffusion bonding process

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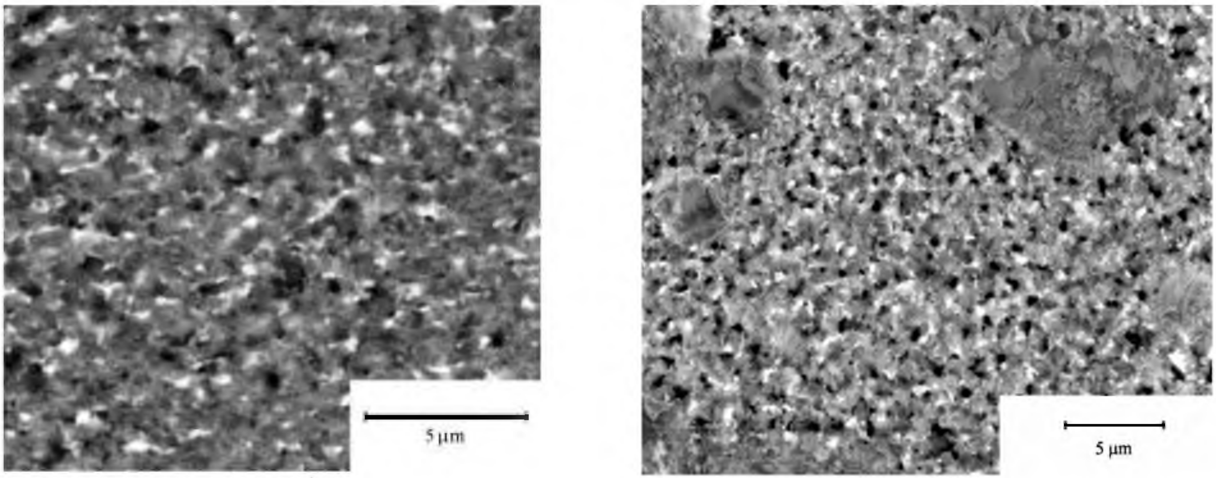
**Keywords:** Ultra-fine grained Ti-6Al-4V, Microcrystalline Ti-6Al-4V, Nitinol, Diffusion bonding, Microstructure, Shear strength.

**Abstract.** The studies of diffusion bonded samples of Ti-6Al-4V and Nitinol alloys were carried out considering the titanium alloy in two states: ultra-fine grained and bi-modal microstructures, the last one consisted of small and large α-phase grains. Depending on microstructure and chemical composition of the alloys, the diffusion bonding had been made at temperatures from 600°C to 850°C. The microstructures of joints was studied by scanning electron microscope using detector of backscattering electron diffraction. The shear strengths of joints were measured. It was concluded that the ultra-fine grained Ti-6Al-4V alloy could be applied for joints manufactured at a temperature lower than 750°C. The bi-modal Ti-6Al-4V alloy is an effective material for producing the joints at the temperature larger that 750°C.

## Introduction

Diffusion bonding (DB) is the process of solid state joining. It is widely used for producing titanium alloys’ parts in aerospace and medicine. The joints of the parts should have high strength and high reliability that can be achieved by the bonding at temperatures which are slightly lower than the beta transus temperature of the titanium alloy. There is a challenge to improve efficiency of the process by further decreasing of the bonding temperature down to 700°C if DB is accompanied by superplastic forming [1]. In the works [2,3] it is demonstrated that high strength of the joints can be achieved if the ultra-fine or nanocrystalline grained materials are applied. However the advantages of ultra-fine grained material are not survived because of an intensive grain growth taking place during bonding. Another issue is the bonding of different alloys like Ti-6Al-4V and NiTi if intermetallic phases can be formed in the contact area. It is shown that the strength of such joint is much lower than the strength in the base materials and it depends on thickness of intermediate layers and regimes of welding or bonding [4-6]. In the present work are compared the properties of joints produced by using ultra-fine grained and bi-modal microcrystalline Ti-6Al-4V alloys, to understand the advantages of fine-grained materials for DB process.

## Materials and methods



a) b)

**Fig. 1.** Microstructure of the Ti-6Al-4V alloy (a) the ultra-fine grained state, b) the bi- modal state).

The materials used in this study are Ti-6Al-4V alloy and Nitinol alloy. The Ti-6Al-4V alloys are in two states. The first one is the ultra-fine grained(UFG) alloy with the grain size of α-phase 0.3μm±0.1, which is fragmented by boundaries on fragments, size of 120±50 nm (Figure 1a). The second one is the bi-modal (BM) alloy with the grain size of the primary α-phase 10 μm and the secondary α-phase 0.8μm±0.1. The volume fraction of the primary α-phase is 22%. The Ti-6Al-4V alloy chemical composition is (wt.%) of 6.2 Al, 3.9 V, 0.083 Fe, 0.17 O, 0.003 N, 0.005 C, and Ti bal. The Nitinol alloy with the grain size of 100μm and chemical composition (wt.%) of 54.2 Ni, 0.14 O, 0.01 C, 0.001 H, 0.001 N and Ti bal. All samples were cut into rectangular samples with dimensions of 5x5x2 mm. Before bonding, the joining surfaces of the samples were electropolished in a solution of 80% $(CH_3CO)_2O$ and 20% $HClO_4$ and cleaned in acetone. The bonding was carried out under vacuum in a furnace. The samples were heated up to the bonding temperatures, which ranged from 650 to 850°C. The samples were heated up to the bonding temperatures for 25 minutes, and after that they were held at a constant temperature for another 25 minutes in order to achieve a uniform temperature distribution in the samples. During this process, the external pressure is not applying. Afterwards, pressure of 15, 30 and 70 MPa applied to the samples of Ti-6Al-4V alloy heated to 650, 700 and 750°C, respectively. The samples of UFG Ti-6Al-4V and Nitinol were bonded under the pressure of 10 MPa at temperatures from 700 to 850°C. The samples of BM Ti-6Al-4V alloy were bonded at 750°C and 800°C. All samples were held under a constant pressure during 1 hour and then they were cooled down under a vacuum for 1 hour.

The microstructure of the samples was examined by a scanning electron microscope (SEM, Quanta-600) with a back-scattered electron detector (BSE). The shear strengths of the joints were measured using a testing machine (Instron-LS300).

## Results and discussion.

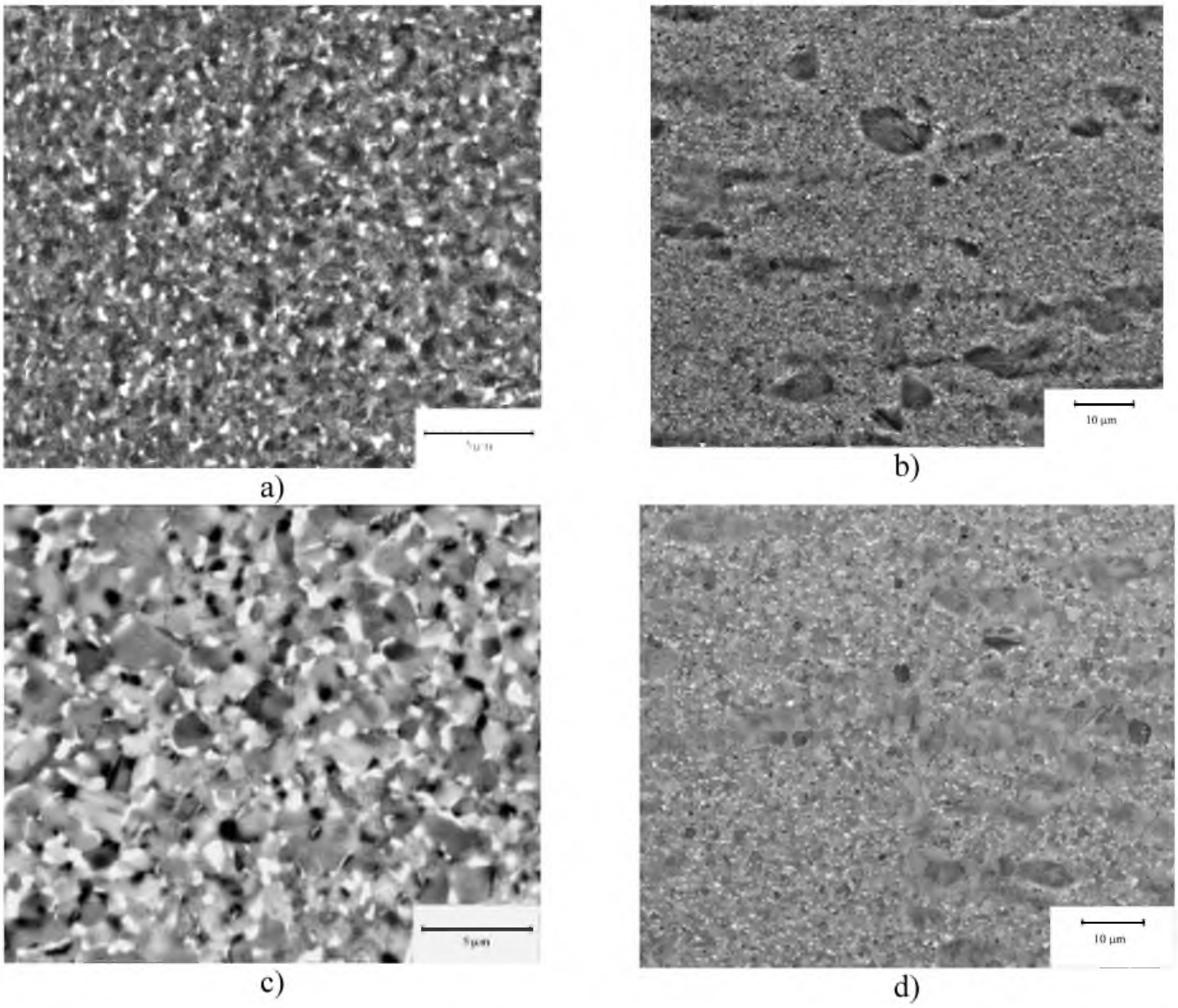


**Fig. 2**. Microstructure of bonded samples of the Ti-6Al-4V alloy (a, c – UFG state, b,d – BM state; a – bonding at 650°C; b – bonding at 750°C, c – bonding at 750°C, d – bonding at 800°C)

The microstructures of Ti-6Al-4V alloy samples after bonding are shown in Fig. 2. Several voids are observed at the bonding interface of the UFG alloy after joining at temperatures of 650°C. The voids at the interface disappear if the temperature of the bonding increases. Only a few voids situated in the sample's edges are revealed in the sample bonded at the temperature of 700°C. There are no voids in the bonding interface after bonding at 750°C. Increase of the bonding temperature leads to intensive grain growth of the α-phase, which size increase to 0.7μm, 1.2μm and 1.5μm depending on temperature of bonding. Simultaneously, the volume fractions of the β-phase increase to 4.7%, 8%, and 9.2% after bonding at temperatures of 650°C, 700°C, and 750°C, respectively. The grains of the β-phase are located on grain boundaries and in the triple junctions.

The microstructure of bonded samples of BM Ti-6Al-4V alloy is present in Fig 2 (b, d). The joint without any voids formed after bonding at temperatures higher than 750°C. The bonding interface can be revealed by analyzing form of boundaries of the primary α-phase grains situated in the interface surface. The recrystallization process takes place in the primary α-phase during annealing of the samples. The recrystallization process completed after bonding at the temperature of 800°C. The recrystallized primary α-phase grain sizes are 1.25μm and 2.3μm after bonding at temperatures of 750°C and 800°C, respectively. The secondary α-phase grains size increases to 1 μm after bonding at the lower temperature and to 1.46 μm after bonding at the higher temperature.

The volume fractions of the β-phase in the former (α+β) colonies are 9.4% and 9% after bonding at temperatures 750°C and 800°C.

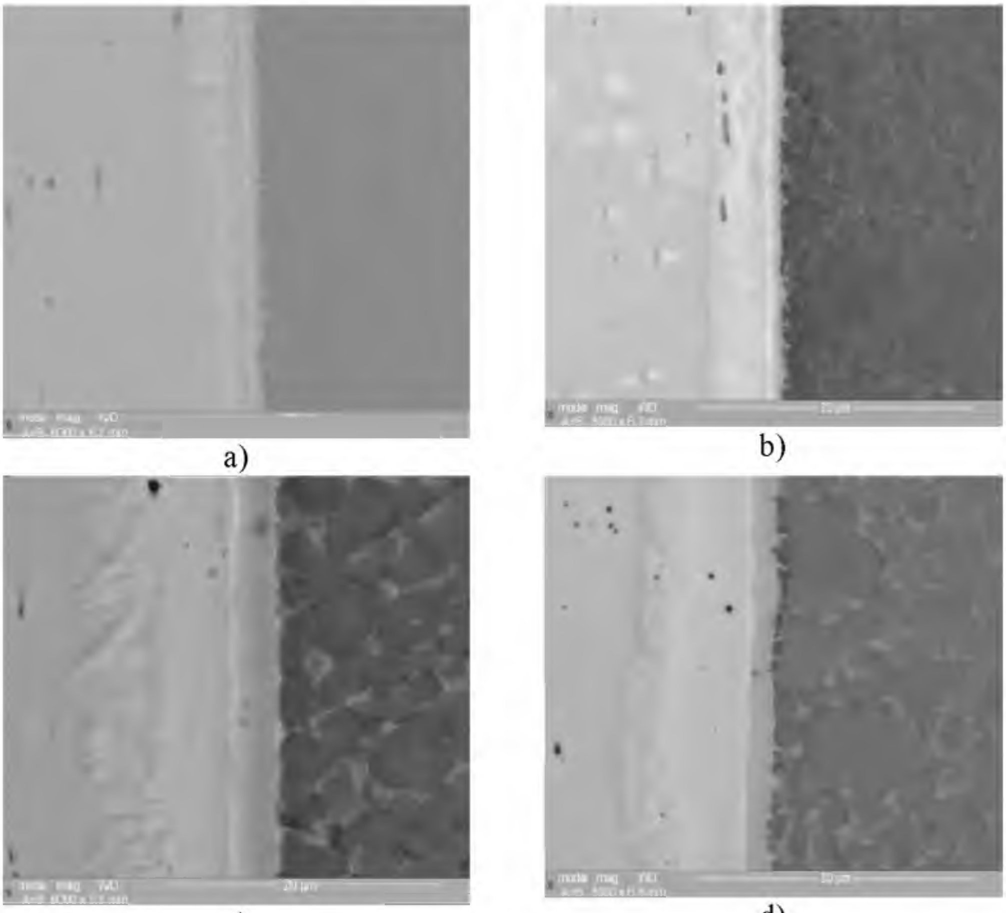

a) b) c) d)

**Fig. 3.** Microstructure of bonded samples of Ti-6Al-4V alloy and Nitinol alloy (a, c – UFG state, b, d – BM state; a, c – bonding at 700°C, b, d – bonding at 800°C)

The microstructure of bonded UFG and BM Ti-6Al-4V alloys with Nitinol is shown in Fig. 3. Three layers are visible in the microstructure of Ti-6Al-4V and Nitinol couples. The first one is NiTi phase enriched by Al and consists of 53Ni 46Ti 1Al (wt%). Because Al increases the temperature of martensite transformation in Nitinol alloy[7], this layer is the martensite. The second layer is intermetallic which consists of 35.8Ni, 60.4Ti, 2.7Al and 1V(wt%). The last one is Ti-6Al-4V enriched by Ni; the layer composition is 6Al, 4V and Ni from 8 to 0 wt% and Ti bal. Because Ni stabilizes β-phase in titanium alloys, this layer can be recognized by analyzing the β-phase volume fraction and the size of the β-phase grains. Note, that the (α+β) lamellas are formed after cooling in samples bonded at a temperature higher than 850°C. The lower temperature of the bonding allows preserving the small β-phase in which lamella of the α-phase is not formed during cooling. The measured thicknesses of the layers are summarized in Table 1. Comparison of the layer thicknesses allows us to conclude that the diffusion of Ni and Ti is faster in the couples of Nitinol with UFG Ti-6Al-4V alloy than in the couple Nitinol and BM Ti-6Al-4V. Although the final grain size of α-phase of UFG Ti-6Al-4V alloy is larger than one of BM Ti-6Al-4V alloy, the difference in diffusion rate can be associated with contribution of grain boundary part of diffusion at the beginning of the process and migration of boundaries during the process.

**Table 1.** The thicknesses of the layers and final grain size of the α-phase in the samples after bonding.

| Materials of a couple | Temperature of bonding, C | NiTi enriched by Al | $Ti_2Ni$ | Final grain size of the α-phase |
|---|---|---|---|---|
| NiTi/ UFG Ti-6Al-4V | 700 | 4,4 | 0,89 | 1,49 |
| | 750 | 13,2 | 2,2 | 2,9 |
| | 800 | 11 | 2,9 | 2,7 |
| | 850 | 18,6 | 3,68 | 2,8 |
| NiTi/ BM Ti-6Al-4V | 700 | 4,3 | 0,8 | 1 |
| | 750 | 6 | 1.17 | 1 |
| | 800 | 8,36 | 1,75 | 1,7 |
| | 850 | 12,8 | 3,1 | 2,7 |

The mechanical properties of the bonded samples are summarized in Table 2.The increase of bonding temperatures leads to increasing of the shear strengths of the Ti-6Al-4V joints. However, the shear strengths of the base material (UFG Ti-6Al-4V) reduced after the DB process. The shear strengths of the UFG Ti-6Al-4V junctions changed from 250 MPa to 643 MPa, which is equal to 29% and 86% of shear strengths of the base material after bonding at 650°C and 750°C, respectively. The shear strengths of the joined BM Ti-6Al-4V samples are 561 MPa and 765 MPa after bonding at 750°C and 800°C, respectively. These magnitudes constitute 70% and 96% of the base material shear strength.

The shear strength of joints of Nitinol and the titanium alloys are smaller than the strength of these alloys. It is interesting to note that the strength of the Nitinol and UFG Ti-6Al-4V is decreased with increasing the temperature of bonding, but the strength of couple Nitinol and BM Ti-6Al-4V is increased with temperature increasing. This phenomenon may be associated with effects of microstructure stability of the titanium alloys and the thicknesses of intermediate layers formed during bonding. The detailed study of this phenomenon will be discussed in a future work.

**Table 2** Mechanical properties of the joints.

| Materials of a couple | Temperature of bonding, C | Shear strength, MPa | |
|---|---|---|---|
| | | UFG Ti-6Al-4V | BM Ti-6Al-4V |
| Ti-6Al-4V | 650 | 250 | - |
| | 700 | 429 | - |
| | 750 | 643 | 561 |
| | 800 | - | 765 |
| NiTi and Ti-6Al-4V | 700 | 130 | 0 |
| | 750 | 64.8 | 56 |
| | 800 | 39 | 97 |
| | 850 | - | 155 |

## Conclusions

The results of the present work demonstrate that UFG Ti-6Al-4V alloy has perspectives for manufacturing parts in which bonding temperature is lower than 750°C. That is caused by their low microstructure stability at elevated temperature that leads to intensive grain growth during annealing. The alloy having microcrystalline structure is most suitable for bonding at a temperature higher than 750°C. The high shear strength joints of the titanium alloy with Nitinol alloy can be formed at temperatures of 700°C and 850°C; the temperature magnitudes depend on the titanium alloy's microstructure.

## Acknowledgement

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