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Microstructure investigation of Ti-26Nb alloy manufactured from elemental powders by electron beam melting

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Abstract. Alloys which are planned to be used for implants fabrication must possess excellent biocompatibility, high strength, and low Young's modulus. A low elastic modulus, close to that of the cortical bone could significantly reduce the stress-shielding phenomenon usually occurring after surgery. Beta-titanium alloys such as Ti-Nb are good candidates for this purpose. Nb is known as a biocompatible metal used for titanium β-phase stabilization. Previous investigations indicate that the increase of Nb content results in the increase of β phase amount but the decrease of β grain size. In this study, we were aiming at the investigation of the microstructural properties of a titanium alloy manufactured by electron beam melting from the elemental powders mixture of Ti and Nb with 26 at.% of Nb. The influence of operating parameters on the efficacy of dissolving Nb particles in Ti was studied. The results obtained by SEM analysis demonstrated that electron beam energy has a significant effect on the homogeneity of the manufactured specimens. To obtain homogeneous and porosity-free specimens high energy level is required. The microstructure of these specimens was characterized.

1. Introduction

Titanium and its alloys have been widely used as biomedical materials for segmental bone reconstruction due to their lightweight, high strength, low elastic modulus, good biocompatibility, and corrosion resistance. The most widely used material for orthopedic implants fabrication by additive manufacturing is Ti–6Al–4V with two-phase $\alpha+\beta$ structure, characterized by its high strength and fracture toughness. However, its high elastic modulus E=110 GPa causes stress shielding and can be a serious concern since it is much higher than the elastic modulus of bone E=40 GPa [1].

It was proved that titanium β phase has a lower modulus than that of the α phase, therefore the β phase is more effective in avoiding mismatch in modulus between the implant and adjacent bone [2]. In order to decrease elastic modulus, Nb is used as β stabilizing element in titanium alloys [3]. A number of studies [4-7] indicate that niobium concentration approximately 26 at. % is sufficient for β phase stabilization in the whole volume of a specimen and obtaining low-modulus alloy. Thus, the elastic modulus of Ti-Nb alloys manufactured by Fischer *et. al.* by selective laser melting (SLM) from a mixture of Ti and Nb elemental powders was about 77 GPa [4]. From the point of view of biocompatibility Nb considered to be non-toxic [8]**.** In studies [11-13] it was reported that Ti-Nb alloys with the concentration of niobium of 45 at.% have Young`s modulus about 60 GPa. In case if these alloys were obtained by

additive manufacturing a prealloyed precursor powder was used. Supposingly, big difference in fusion tempretures of Ti and Nb would prevent from homogenious alloy fabrication.

Electron beam melting (EBM) additive manufacturing allows forming complex components from powdered precursor material by sequentially and selectively melting layers using CAD models and embedded software to control the electron beam. However, Ti-26Nb is not an average material for additive manufacturing, therefore the regimes for melting were chosen by trail-and-error method.

The objective of the current study was to estimate the defects of manufacturing and investigate the microstructural properties of the Ti-26Nb alloy obtained by EBM.

2. Materials and methods

2.1. Specimens

To obtain Ti-Nb alloy with 26 at. % of Nb elemental non-spherical niobium and spherical titanium powders were mixed with a weight proportion of 40.5 w. % of Nb to 59.5 w. % of Ti, figure 1, *a*. Bulk specimens with a size of 1×1×1 cm were manufactured by EBM system (Arcam EBM, Mölndal, Sweden). The additive manufacturing equipment was described elsewhere [9].

The mixture of powders is presented in figure 1. Particles` sizes were estimated by FiJi software [10]. The size distribution of titanium particles was D10 = 42 μm, D50 = 63 μm and D90 = 91 μm and as received niobium particles sizes ranged from D10 = 7 μm to D90 = 14 μm with D50 = 28 μm, figure 1, *b*.. As far as Nb particles were smaller than Ti particles Nb was expected for better melting and better Nb diffusion in Ti because fusion temperature of niobium is 2477 °C, while fusion temperature of titanium is only 1668 °C. This approach was also used by Fischer *et. al.* in their study [4]. The flowability of the precursor powder inspected with Hall flowmeter emerged to be poor, for this reason, the powder supplying system of EBM-machine was partly improved.

The manufacturing parameters impacting the global energy delivered to the surface, such as scanning velocity and line offset were varied to reach optimal melting regime. The manufacturing regimes with area energy of 8,5 and 13 J/mm^2 were chosen by the trail-and-error method to reach uniformly distributed *β*-phase of titanium.

To our knowledge, Ti-26Nb alloy was firstly manufactured by electron beam melting (EBM) using a mixture of Ti and Nb elemental powders by our research group. After the specimens had been manufactured they were cut for further inner structure and defects characterization.
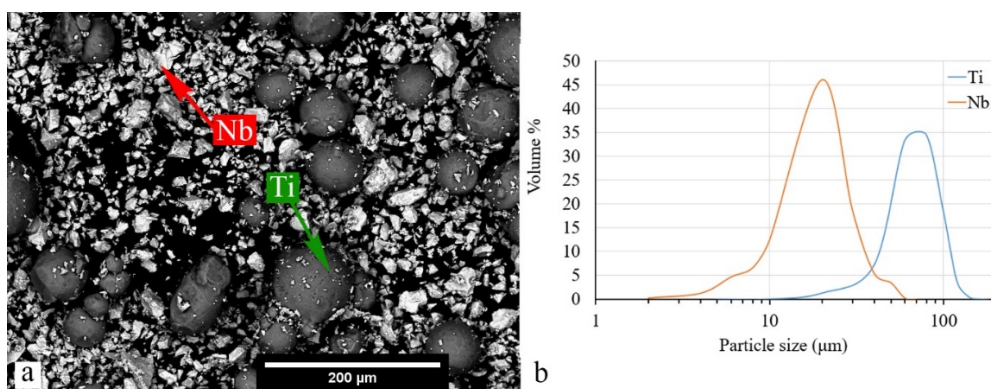
Figure 1. Ti and Nb powder mixture**:** *a* – SEM image of the mixture of elemental Ti and Nb powders, *b* – Ti and Nb particles size distribution

2.2. XRD and SEM analysis

Scanning electron microscopy SEM analysis of the specimens was performed with FEI Quanta 200 3D equipped with energy-dispersive X-ray spectroscopy (EDS; Genesis 4000, SUTW-Si(Li) detector) operating in a high vacuum. The microstructure properties and phase composition of the Ti-Nb samples were analyzed by X-ray diffraction in θ-2θ geometry using Shimadzu XRD 7000S diffractometer

equipped with high-speed wide-range 1280 channels detector OneSigth. The diffraction data were analyzed using Sleve+ program.

3. Results and discussion

XRD patterns of Ti and Nb powders mixture and bulk specimens obtained at various regimes are presented in figure 2 and enable to identify the phases present in the materials. Two phases corresponding to hcp α titanium and bcc niobium were identified in the powders mixture. In the manufactured bulk specimens, diffraction patterns show clearly the elimination of the titanium α-phase with the applied energy growth.
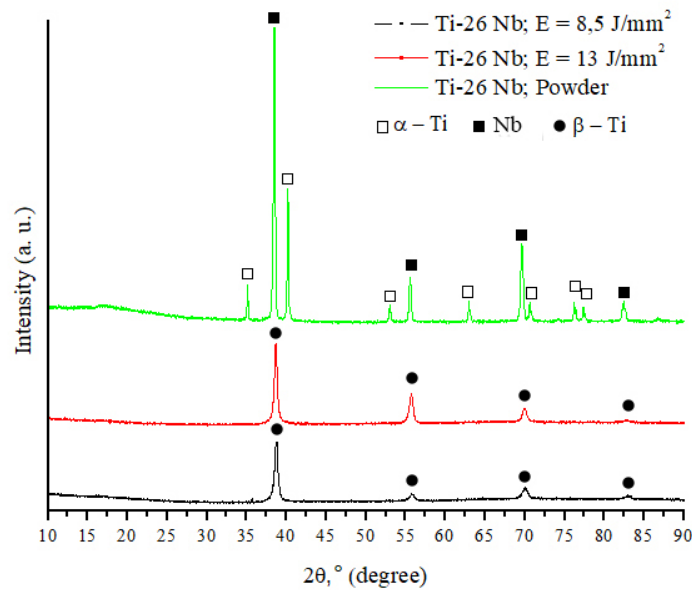
Figure 2. XRD pattern of Ti-Nb powder and different energy level

The peaks for higher 2θ may correspond both to Nb bcc structure or Ti–26Nb β-phase because lattice parameters of Nb are close to that of metastable β TiNb which makes them difficult to differentiate. They may correspond to the Nb particles and the TiNb alloy, as they are slightly wider than usual peaks [4]. Amount of niobium was sufficient to completely remove α-phase no matter of energy applied. The only phase which is present in the samples fabricated at 8.5 and 13 J/mm² is β phase. This is evidence of the energy was sufficient for two metals to diffuse into each other and form a homogeneous alloy for a short period of time. A β-metastable phase could retain at room temperature due to the energy the cooling speeds are high enough. Lattice parameters were recalculated with the experimental diagram and presented at table 1. With the energy growth from 8.5 J/mm² to 13 J/mm² the crystallite size of β-phase decreased from 22 to 20 nm, and the lattice parameter increased from 3.29 to 3.30.

Table 1. Microstructure characteristics of Ti-26Nb alloy and Ti-Nb powders mixture

Name	Phase	Phase concentration, volume. %	Lattice parameter	Crystallite size, nm	Microstrain
Powder	Ti_hexagonal	31.1	a=2.9483 c=4.6930	51	0.000524
	Nb_cubic	68.9	a=3.3063	49	0.000817
Energy density 8,5 J/mm²	Ti_cubic	100	a=3.2910	22	0.002217
Energy density 13 J/mm²	Ti_cubic	100	a=3.3015	20	0.001866

Despite the only *β*-phase of Ti was found by XRD analysis, the real structure of the alloy is not homogeneous. SEM examination of the cut made perpendicular to the building direction of the alloy fabricated at 8.5 J/mm^2 reveals inhomogeneity and phase distribution on the surface of specimens cut and presented in figure 3. For the selected fragment an EDS analysis was performed in order to confirm the light regions are fused niobium particles.

A large pore with the length of more than 1 mm and width of approximately 250 mm was observed, which make it a crack-like void. This means that the manufacturing regime is imperfect and can cause the appearance of large pores with a low value of sphericity, which can negatively affect the mechanical properties of the future product [14]. During the manufacturing process, some surface defects such as swelling [15] or pits [16] also were observed which are usually associated with metal overheating. However, in our case, they could be caused by the mismatch of Ti and Nb powder particles sizes and forms, that affected their flowability. In some cases, the alloy demonstrated a poor connection between layers or shift of newly manufactured layer from the verticle axis that were easily estimated visually.
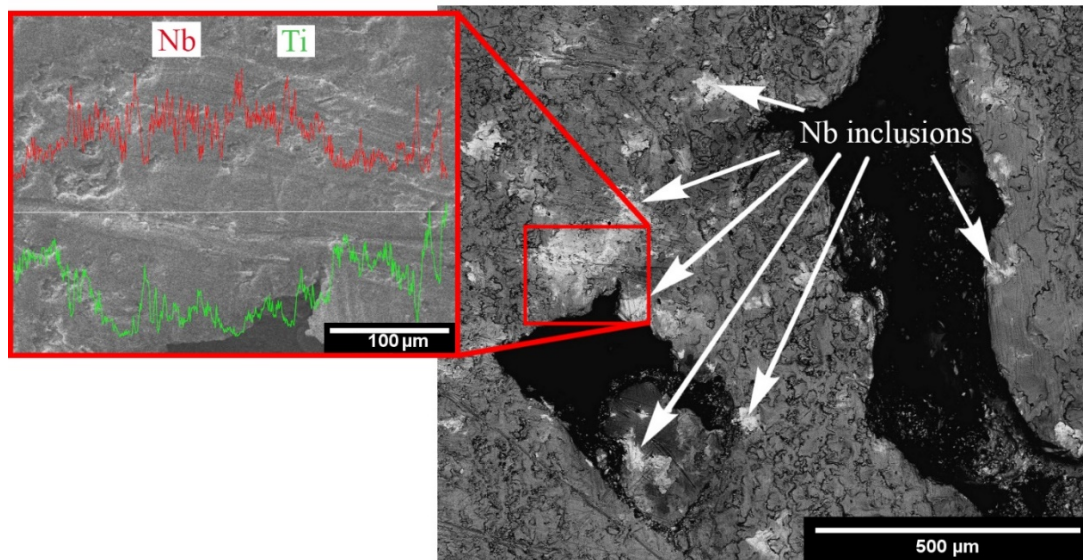

Figure 3. SEM image of a large pore

The face energy of 8.5 J/mm^2 was not sufficient for the elaboration of a homogeneous, nonporous alloy. SEM image presented in 4, *a* demonstrates a pore oriented perpendicular to the building direction. Such crack-like voids located between layers can cause poor layers connection and further delamination. Nb particles and *α*-phase islands located nearby presumably indicates on the local lack of energy for Ti and Nb complete melting and diffusion. However, a large part of the specimen is *β*-phase Ti which is confirmed with XRD analysis. In the case with the specimen, obtained at face energy of 13 J/mm^2 allows a large part is filled with *β*-phase Ti, figure 4, *b*. The islands of the remaining *α*-phase of Ti and small interstitial of Nb formed a border-line between the inner part and right part can be observed. The right part is an outline which was manufactured in multibeam mode. Consequently, the heating between the inner and the outer part was is not sufficient and this caused the occurrence of phase heterogeneity.

4. Conclusion
In this work, Ti-26Nb alloy was manufactured by electron beam melting from the elemental powders mixture of Ti and Nb using two regimes. In the precursor powders mixture, two phases corresponding to hcp α titanium and bcc niobium were identified. The alloy fabricated at both regimes mainly consists of Ti β phase. With the energy growth, the crystallite size of β-phase decreased and the lattice parameter increased.
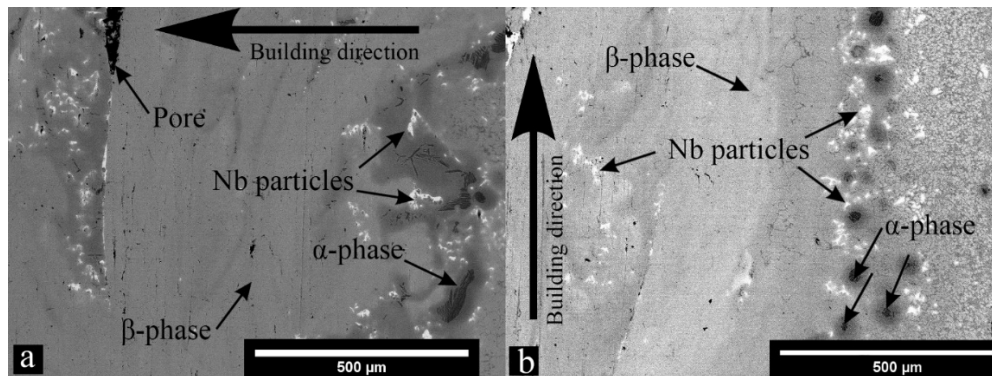
Figure 4. SEM image of Ti-26Nb specimens: a – 8.5 J/mm^2, b – 13 J/mm^2

It was revealed that the efficacy of dissolving Nb particles and alloy homogeneity are determined by the electron beam energy. Despite the only *β*-phase of Ti was found by XRD analysis, the real structure of the alloy is not homogeneous. Crack-like voids which can negatively affect the mechanical properties of the future product were found. Small incorporations of *α*-phase of Ti and Nb particles were found, that can be caused by insufficient local heating.

Overall, the present investigation demonstrated that manufacturing of binary alloy from elemental powders is possible, the homogeneity of the alloy increases with electron beam energy.

Future work will focus on the improvements to the manufacturing process parameters to obtain completely homogeneous alloy.

Acknowledgments

References
[1]   M A Fernandez-Yague, S A Abbah, L McNamara, D I Zeugolis, A Pandit, and M J Biggs 2005 *Adv. Drug Deliv. Rev*. **84** 29
[2]   Y L Zhou, M Niinomi, and T Akahori 2004 *Mater. Sci. Eng. A* **371** 1–2 283–290
[3]   K Zhuravleva et al. 2013 *Materials* (Basel) **6** (12) 5700–5712
[4]   M Fischer, D Joguet, G Robin, L Peltier, and P Laheurte 2016 *Mater. Sci. Eng. C* **62** 852–859
[5]   M Fischer et al. 2017 *Mater. Sci. Eng. C* **75** 341–348
[6]   G Dercz and I Matuła 2017 *Mater. Tehnol.* **51** 5 795–803
[7]   P Didier, B Piotrowski, M Fischer, and P Laheurte 2017 *Mater. Sci. Eng. C* **74** 399–409
[8]   A H Hussein, M A H Gepreel, M K Gouda, A M Hefnawy, and S H Kandil 2016 *Mater. Sci. Eng. C* **61** 574–578
[9]   Y Zhong et al. 2017 *J. Nucl. Mater.* **486** 234–245
[10]   E Schindelin, J Arganda-Carreras, I Frise 2012 *Nat. Methods* **9**(7) 676–682
[11]   Q Wang et al. 2017 *Mater. Des.* **126** 268–277
[12]   K Prashanth, K Zhuravleva, I Okulov, M Calin, J Eckert, and A Gebert 2016 Technologies **4**(4) 33
[13]   H Schwab, K G Prashanth, L Löber, U Kühn, and J Eckert 2015 *Metals* (Basel) **5** 686–694
[14]   G Kasperovich, J Haubrich, J Gussone, and G Requena 2016 *Mater. Des.* **112** 160–161
[15]   X Tan, Yihongkok, S B Tor, and C K Chua 2014 Application of electron beam melting (EBM) in additive manufacturing of an impeller May
[16]   H Gong, K Rafi, H Gu, T Starr, and B Stucker 2014 *Addit. Manuf.* **1** 87–98