

Study of elastic modulus of $Ti_{(70-x)}-Nb_x-Ta_{25}-Zr_5$ alloys for biomedical applications

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Abstract. In this study, titanium alloy $Ti_{(70-x)}-Nb_x-Ta_{25}-Zr_5$ ($x = 5, 10$ at.%) was synthesized using arc melting followed by water cooling and a series of heat treatments, specifically a solution treatment at 950°C for one hour, water quenching, and aging at 480°C for twelve hours under an argon atmosphere. The resulting microstructures predominantly exhibited a dendritic β phase, and prior β grain boundaries. Nanoindentation tests indicated that the lowest elastic Young's modulus recorded for heat-treated sample $Ti_{60}-Nb_{10}-Ta_{25}-Zr_5$ was 66.6 ± 15.2 GPa. The XRD, EBSD and indentation and hardness results are presented in the paper.

1 Introduction

Titanium-based alloys have gained significant attention in the biomedical field, primarily due to their remarkable biocompatibility, corrosion resistance, and excellent mechanical properties [1] [2]. These alloys are highly suitable for applications such as dental and orthopedic prosthetics because they interact favorably with biological tissues, promoting osseointegration and reducing the likelihood of implant failure [3][4]. Among titanium alloys, near- β titanium alloys offer a balanced combination of strength, ductility and toughness, and generally exhibit better hot processing performance than most industrial titanium alloys [5]. However, these alloys are still too stiff compared to human cortical bone (10-30 GPa) [6], and this mismatch leads to a phenomenon called 'stress shielding effect'. Biomechanical compatibility requires high strength to elastic modulus ratio, i.e. high permissible strain. As far as modulus is concerned, β titanium alloys offer significant advantage. Recently, TNTZ, a new metastable β Ti-based alloy, was created in response to the growing need for medical implants. This alloy satisfies important biomaterial characteristics, such as low elastic modulus, and contains non-toxic elements including tantalum (Ta), niobium (Nb), and zirconium (Zr) [7].

The present study aims to evaluate the elastic modulus of $Ti_{(70-x)}-Nb_x-Ta_{25}-Zr_5$ (at.%) alloys, fabricated through arc melting techniques, while also assessing the impact of heat treatment on their microstructural characteristics and mechanical properties. Ultimately, the alloy must possess elastic modulus closer to that of human cortical bone for implant

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applications. Due to the high melting points of Ti (~1668°C), Nb (~2477°C), Ta (~3017°C) and Zr (~1852°C) arc melting technique was used ensure complete melting and mixing. Heat treatment was employed to homogenize the alloy while balancing high strength and low elastic modulus [8].

2 Materials and methods

Elemental powders CP-Ti, Nb, Ta and Zr were premixed and pressed into 50 mm diameter buttons. Metal powders of Nb, Ta and Zr were supplied by Standford Advanced Materials, USA. The CP-Ti powder was supplied by TLS Technik GmbH & Co. Spezialpulver KG, Germany. All powders were spherical.

The buttons were melted in a copper-hearth arc melting equipment to produce composition $Ti_{(70-x)}Nb_xTa_{25}Zr_5$ ($x = 5, 10$ at.%). The samples underwent water cooling and were subsequently heat treated under controlled conditions [9]. Heat treatment included solution treatment at 950°C for 1 hour, followed by water quenching (WQ) and aging at 480°C for 12 hours with furnace cooling (FC) under argon atmosphere. The heating rate during heat treatment was kept at 5°C/min. The resulting microstructures were characterized by using optical microscope (OM), x-ray diffraction (XRD), electron backscatter diffraction (EBSD). To assess the elastic modulus and hardness of the samples, nanoindentation and Vickers micro-hardness methods were used. The XRD method was done using monochromatic Cu K α radiation ($\lambda = 0.154$ nm) at 45 kV and 40 mA. Scans were conducted continuously over a 2θ range of 10 – 100°. Hardness was done using 1 kgf load for 10 seconds while nanoindentation was performed at 400 mN. Both the testing methods followed ASTM-E384-17 and ASTM E2546 standards.

3 Results and discussion

The microstructural analysis revealed a dominant body-centered cubic (bcc) β phase (bright) stabilized in a dendritic morphology within the hexagonal closed-pack (hcp) α matrix (dark), Figures 1. The as-cast samples had fine dendrites while increase in Nb content led to finer microstructure. After heat treatment, the dendrites grew thicker indicative of improved β stabilization. The heat treated $Ti_{60}Nb_{10}Ta_{25}Zr_5$ had prior β grain boundaries. The prior β grain boundaries contribute to phase transformation and texture and ultimately the mechanical properties. The dominant microstructure of β phase is expected to lower the elastic modulus of the alloys [10]. The XRD patterns in Figure 2 showed strong peaks of β phase especially at $2\theta = 38.48^\circ$ evident of β stabilization with increasing Nb content. The stronger peak at the latter angle was also attributed to the presence of α peak. The peaks were analysed against the bcc β -Ti reference pattern (JCPDS file no. 00-044-1288) and hcp α -Ti reference pattern (JCPDS file no. 00-044-1294). The peaks grew wider after heat treatment suggesting α phase precipitation observed in the microstructures. The effect of Nb content was also noticeable in terms of peak height.

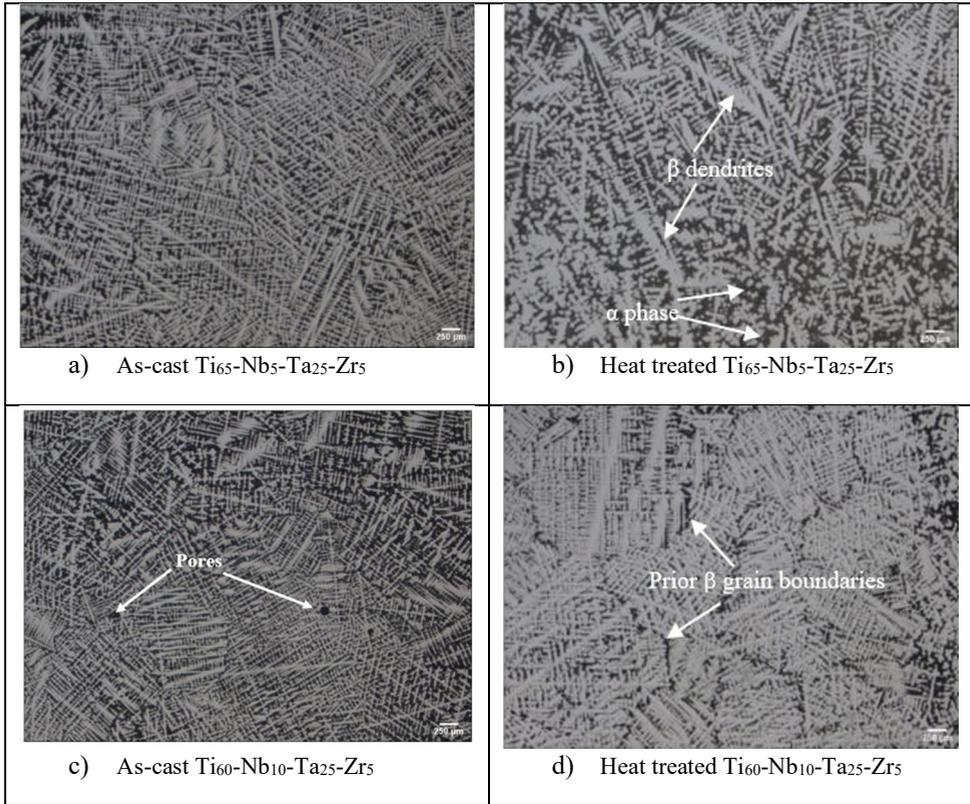


Fig. 1. Optical micrographs of alloys $Ti_{(70-x)}-Nb_x-Ta_{25}-Zr_5$ before and after heat treatment treated ($950^{\circ}C/1$ h WQ + $480^{\circ}C/12$ h FC). Scale bar = 250 microns.

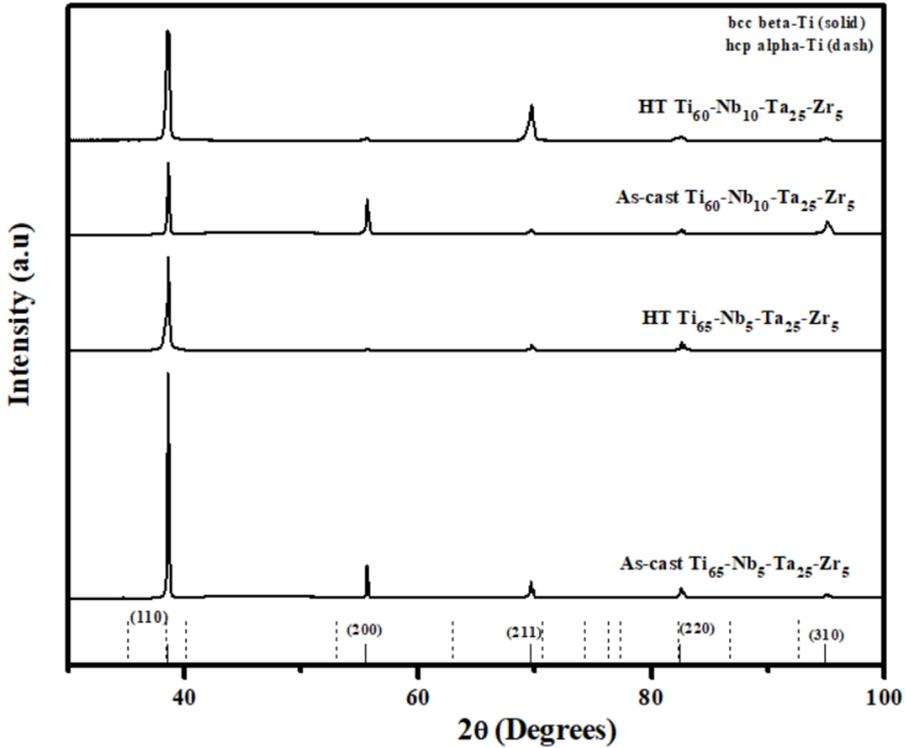
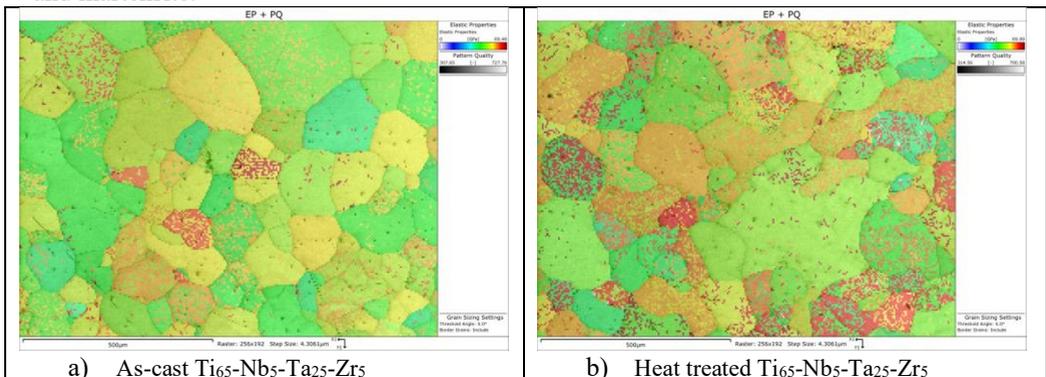


Fig. 2. The XRD patterns of alloys Ti_(70-x)Nb_xTa₂₅Zr₅ before and after heat treatment.

Figure 3 shows the microstructures of alloys Ti₆₅-Nb₅-Ta₂₅-Zr₅ and Ti₆₀-Nb₁₀-Ta₂₅-Zr₅ characterized EBSD. The alloys generally displayed a mixture of larger and smaller grains. The EBSD maps revealed more clearly grains of β phase. The analysis further estimated the elastic modulus before and after heat treatment, revealing a modulus range of 50–70 GPa for the processed samples. The ω phase was also detected during EBSD analysis. According to the micrographs, Nb content did not influence the morphologies of the β grains. Higher content of β-stabilizers such as Nb increases the risk of forming secondary phases such as ω and martensite.



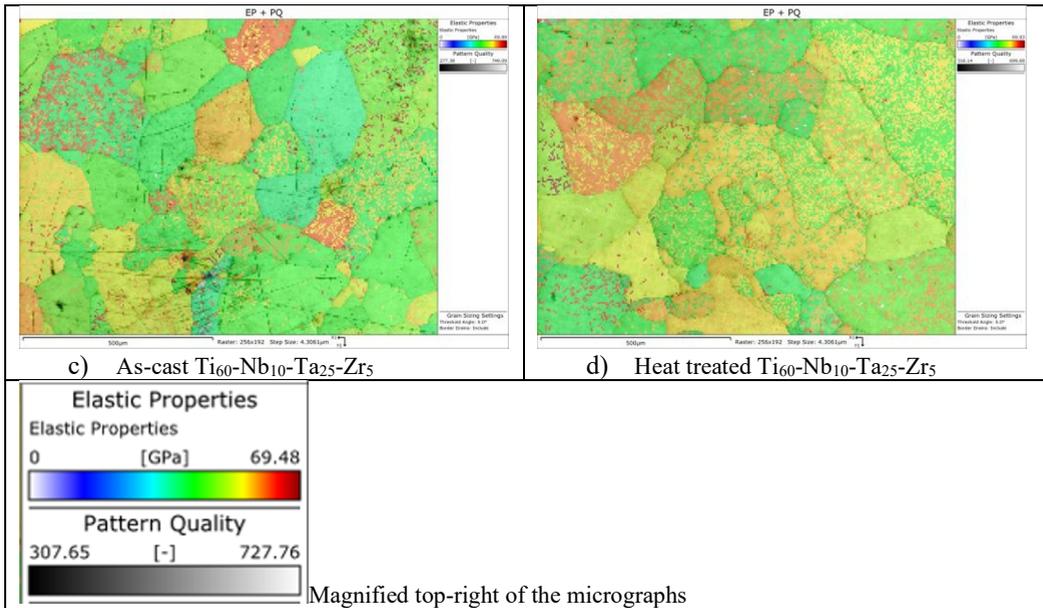


Fig. 3. EBSD micrographs of the samples in the as-cast and heat treatment conditions

Nanoindentation testing was employed to estimate the elastic modulus, showing that the heat treated sample Ti₆₀-Nb₁₀-Ta₂₅-Zr₅ attained the lowest elastic modulus of 66.6 ± 15.2 GPa, despite the large standard deviation. The as-cast sample of the latter alloy had the elastic modulus of 78 ± 0.1 GPa. Alloy Ti₆₅-Nb₅-Ta₂₅-Zr₅ had the elastic modulus of 80 ± 2.6 GPa in the as-cast condition and the modulus increased to 93 ± 0.5 GPa after heat treatment. The increase in elastic modulus after heat treatment can be attributed to precipitation of α phase, as supported by XRD data. Figure 4 shows plastic recovery curves of the alloys. Increased Nb content resulted in higher penetration depth suggesting a softer microstructure. Heat treatment resulted in harder Ti₆₅-Nb₅-Ta₂₅-Zr₅ microstructure indicative of α precipitation. Moreover, less β content was expected at 5 at.% Nb resulting in harder microstructure. Sample Ti₆₀-Nb₁₀-Ta₂₅-Zr₅ exhibited improved ductility after heat treatment. Hardness data was as follows: Ti₆₅-Nb₅-Ta₂₅-Zr₅ (as-cast 661 ± 20 , heat treated 381 ± 12 Hv) and Ti₆₀-Nb₁₀-Ta₂₅-Zr₅ (as-cast 535 ± 33 , heat treated 317 ± 7 Hv). Alloy Ti₆₀-Nb₁₀-Ta₂₅-Zr₅ had lower hardness especially after heat treatment. These findings aligned with β microstructure in Figure 1, confirming that the niobium content and heat treatment effectively modulated β stabilization and elastic properties of the alloys.

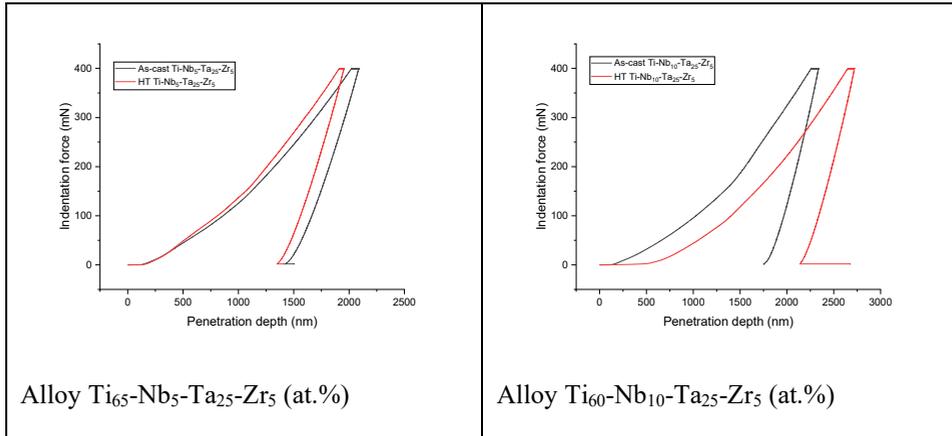


Fig. 4. The plastic recovery curves of as-cast and heat Ti_(70-x)-Nb_x-Ta₂₅-Zr₅ alloys

4 Conclusions

The analysis of the microstructural, XRD, EBSD, and nanoindentation results confirmed that increasing niobium (Nb) content and applying heat treatment promoted β phase stabilization in the Ti_(70-x)-Nb_x-Ta₂₅-Zr₅ alloys. This study demonstrated that the alloys exhibit a tunable elastic modulus based on niobium content and heat treatment conditions, particularly in Ti₆₀-Nb₁₀-Ta₂₅-Zr₅ sample. This alloy exhibited a dominant β phase with finer dendrites pre-treatment and thicker ones after heat treatment, indicating enhanced β stability. XRD and EBSD analyses supported these observations, revealing stronger β phase peaks and distinct β grain boundaries. Despite the presence of α and ω phases, especially after heat treatment, the elastic modulus remained low (50–70 GPa), with Ti₆₀-Nb₁₀-Ta₂₅-Zr₅ achieving the lowest modulus of 66.6 ± 15.2 GPa and highest ductility after heat treatment. The decreasing hardness values post-treatment further validated the softening effect and β phase stabilization. Overall, the results highlighted the effectiveness of Nb content and heat treatment in tailoring microstructure and improving mechanical properties suitable for biomedical applications. Alloy Ti₆₀-Nb₁₀-Ta₂₅-Zr₅ is a promising candidate for biomedical applications.

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