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Effect of Sn-addition on the properties of the biomedical Ti-17Nb-6Ta alloy.

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Abstract. The effect of Sn-addition (0, 1.5, and 3 Sn, at.%) to the biomedical Ti-17Nb-6Ta alloy has been investigated in this study. The three alloys were proved using XRD analysis to be β -type alloys. Microstructural analysis using optical microscope showed martensite lathes in all alloys and proved that Sn addition stabilized β phase and suppresses the martensite formation during quenching. Micro-hardness results showed a slight increase with 1.5% Sn adding but superior addition of Sn with 3% at. has negligible effect on the hardness compared to Sn-free alloy. The compressive yield stress for the three alloys located between 400 to 500 MPa, and the strain values increased with the increasing of Sn percentages in the TNT alloy.

Keywords : β Ti-alloys, Ti-17Nb-6Ta alloy, Sn-addition, phase stability, biomedical alloy.

1. Introduction

Recently, researchers gave a great attention for new biocompatible materials as a solution of allergic problems in some commercial biomedical alloys. These alternatives opened the door for new alloy generations which are nickel-free ones, these alloys target vital elements to reach the highest biocompatibility [1,2]. Recent statistics showed that nearly two third of the patents in US over 13 years period in the last two decades went to biomedical sector[3].

The elements added to titanium to form an alloy show different effects on the stability of the titanium phases in projected alloy. The alloying elements of titanium alloys are classified into three different categories; α -stabilizers, β -stabilizers, and neutral elements. In the first type, α -stabilizers elements, the added element is working on increasing the temperature range at which α -phase could present. On the other hand, β -stabilizers elements are those elements that contribute to lower the temperatures of β -phase field. Neutral elements do not show a significant effect on stabilizing any of the two phases [4]. Sn was proved to be generally weak α -stabilizers but recent studies showed that in β -type alloys Sn acts like a weak β -stabilizing element [5].

The TNT alloy was developed recently by Gepreel et al, [6] and showed many promising mechanical properties (such as controllable strength, high ductility and excellent cold workability) [7,8]. High corrosion resistance besides excellent biocompatibility were confirmed by cytotoxicity tests [9]. The purpose of this study is to investigate the effect of Sn adding to Ti-17Nb-6Ta (TNT) alloy on the microstructure and mechanical properties. Sn was added to the TNT with two different percentages and compared with the original TNT alloy.



2. Experimental method

2.1 Alloy design considerations

Ti-17Nb-6Ta (TNT) alloy has \overline{Bo} , \overline{Md} and e/a values very close to that of GUM metals [10]. Here, \overline{Bo} is defined as the average bond order between alloying element and Ti atom, and \overline{Md} is the average d-orbital energy level and (e/a) is the valence electrons per atom ratio of the alloy [5]. Sn is known to be high biocompatible element for developing new Ti-alloys. Also, Sn is known as α -stabilizing element but it acts as a weak β -stabilizing element in β type alloys. The addition of Sn to TNT alloy will not affect the e/a value but decrease both the \overline{Bo} and \overline{Md} values and may result in higher β -phase stability, as shown in Table 1. This will give a room to control the phases stability and so the mechanical properties of this TNT alloy system. In this study, Sn is added to TNT alloy with amounts of 1.5 and 3 at % (named hereafter as TNT-1.5 and TNT-3, respectively).

Table 1. \overline{Bo} , \overline{Md} and e/a values of TNT, TNT-1.5 and TNT-3 alloys.

Alloy	\overline{Bo}	\overline{Md} (eV)	e/a
TNT	2.86377	2.44813	4.23
TNT-1.5	2.856165	2.442925	4.23
TNT-3	2.84856	2.43772	4.23

2.2 Material preparation

The three alloys were produced using arc melting furnace with electromagnetic stirrer option in argon atmosphere. Then cold rolling deformation was carried out on the cast ingot using rolling mill to ~30% of ingot thickness. After that the rolled ingot was cut into smaller specimens using precision cutting saw under lubricant cooling. Solution treatment was carried on the samples at 900° C for 10 min followed by ice-water quenching. Oxide layers were removed using sandpapers. Specimens were mounted for microstructure analysis then grinded with abrasive papers up to 2400 grit and followed by polishing. Specimens were followed by chemical etching as immersed in etching solution of 10% HF (40% conc.) and 20% HNO₃ (70% conc.) [11]. Samples were prepared for XRD analysis and hardness testing by grinding them up to 1000 grit. Also, samples for compression tests were prepared using immersed wire cutting to cut cylindrical specimens with 6mm diameter and 7.5mm height. Then, these samples were re-solution treated at 900° C for 10 min followed by ice-water quenching to insure of removing any heat effect occurred while cutting the samples. After that, oxide layers were removed using manual grinding set. Also, a special set was used in order to get a fine parallel surfaces in compression samples.

2.3 Material testing

The optical micrographs of the alloys were observed using optical microscope. Existed phases of the alloys were investigated through Cu K α X-ray diffraction with conditions 30 kV and 30 mA at a scan rate of 1 degree/min using (XRD-6100, Shimadzu, Japan) diffractometer. The micro-hardness was measured by (Shimadzu HMV-FA) tester under the load of 5 N and holding for 15 seconds. Every specimen was measured 5 times, and then the mean values were calculated. Also, the error deviations were obtained. Compression tests were operated using (Shimadzu AGS-X, 100 kN) universal testing machine in air at room temperature with stain rate 5×10^{-4} sec⁻¹. The compression test was stopped at 50% engineering strain.

3. Results and discussion

The microstructure of the solution treated TNT, TNT-1.5 and TNT-3 alloys are showed in Figure 1. The martensite lathes are observed in the three alloys with different amounts. For TNT martensite lathes are clearly appeared inside the grains and at the grain boundaries as presented in Figure 1(a), almost everywhere. Also, in TNT-1.5 the martensite lathes are observed but with less amounts than in TNT, as shown in Figure 1(b). This suggests and confirms the previous idea that Sn acts as β stabilizing element

in this β -type TNT alloy. In the same way for TNT-3 alloy, the martensite lathes also appeared with less amount but scattered in random areas because of the confirmed higher β -phase stability in this alloy as shown in Figure 1(c),(d). These results have a good agreement with XRD ones shown in Figure 2 as discussed below.

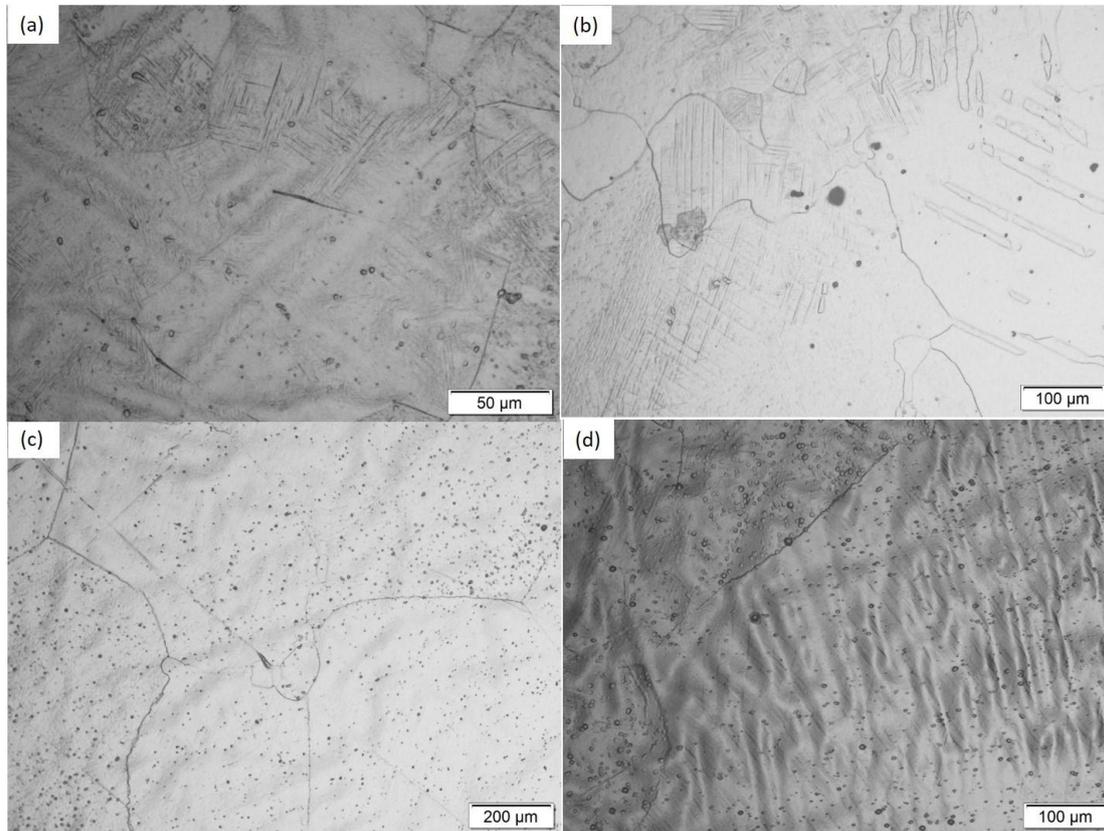


Figure 1. Optical micrographs obtained for (a)TNT, (b) TNT-1.5 and(c),(d) TNT-3 alloys in the solution treated condition.

The XRD patterns showed the changes in phases existed in the solution treated TNT, TNT-1.5 and TNT-3 alloys. Main Bragg reflections are related mainly to the α'' martensite phase in addition to small amounts of β -phase in the solution treated TNT alloy. The α'' martensite phase was formed during the quenching from austenitizing temperature. However, the β -phase is the predominant phase in the TNT-3 alloy. Besides the β -phase, the presence of α'' martensite phase is also observed in this TNT-3 alloy. Martensite peaks of TNT-1.5 alloy are moderate between TNT-3 and TNT alloys with the predominance of β -phase. This confirms that the Sn-addition have a proven influence on stabilizing β -phase in the TNT β -type alloy through both microstructural and XRD investigations.

The micro-hardness change with the addition of Sn to the TNT alloy in the solution treated condition is measured and reported in Figure 3. The hardness value increased by the addition of 1.5%at. Sn from around 190 HV in TNT alloy to 220 HV in TNT-1.5 alloy. But with further addition of 3%at. Sn, in TNT-3 alloy, had almost no effect on the hardness and same values recorded as TNT alloy. Although the β -phase stability in TNT-3 alloy is higher than TNT, both showed the same hardness. The reason behind this is unclear at the moment and further detailed study is needed.

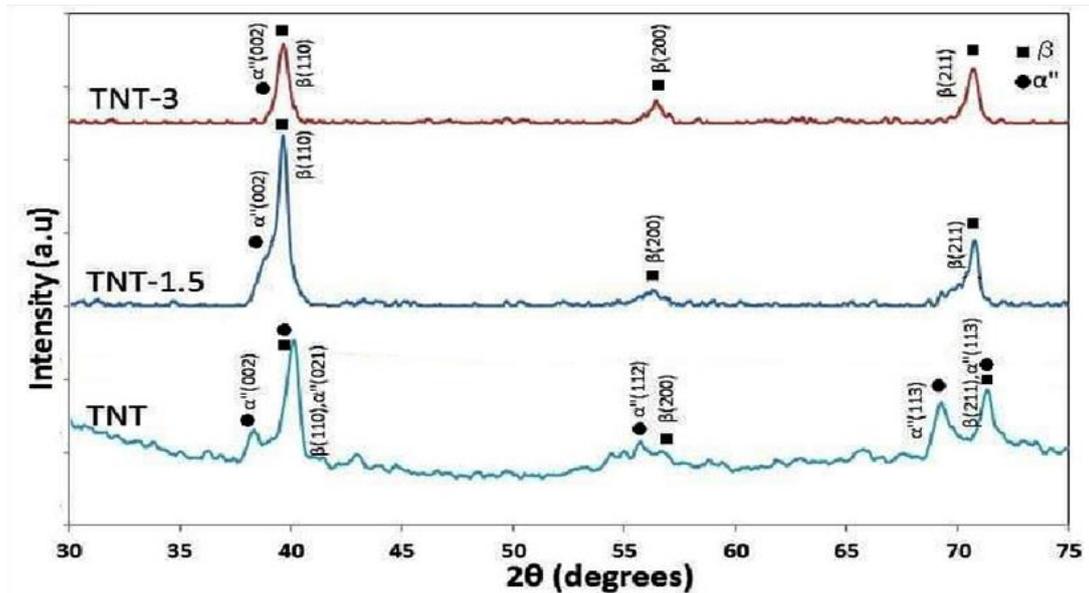


Figure 2. XRD patterns of TNT, TNT-1.5 and TNT-3 alloys in the solution treated condition.

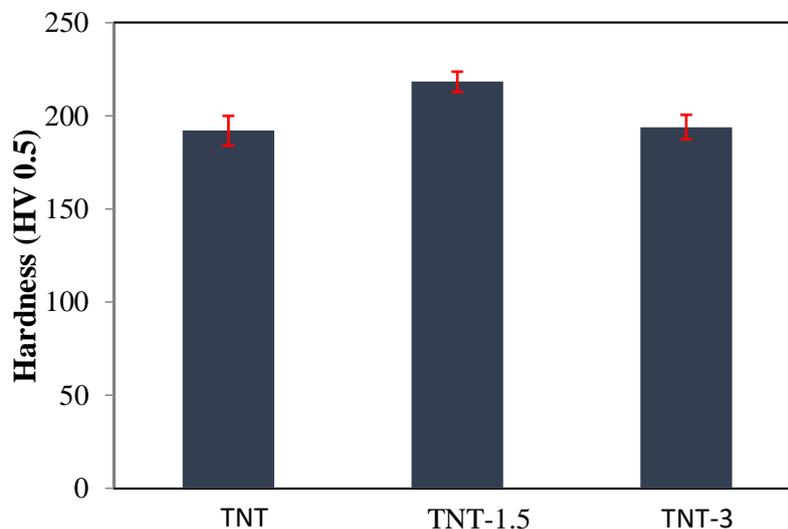


Figure 3. Micro-hardness changes of TNT, TNT-1.5 and TNT-3 alloys.

Compression tests were carried out on the solution treated samples of the alloys and the true stress – true strain curves were plotted in Figure 4. From this figure, it is clear that the elasticity/elastic strain of both alloys are high since the apparent yielding of the alloys is at compression true strain of more than 10%. Also, a double-yielding-like phenomena is clearly observed in the TNT-3 alloy. In addition, the alloys plastic yielding is in the range between 450 and 550 MPa. The lowest yielding strength is observed in TNT alloy and the highest is in TNT-1.5 alloy in good agreement with the hardness trend. The ultimate

compression strength is showing the same trend; TNT-1.5 > TNT > TNT-3 with values 750, 700 and 600 MPa, respectively. The alloys show ultimate compression strength at a compression true strain of ~40%, while the test was stopped at ~50% true strain without specimens failure.

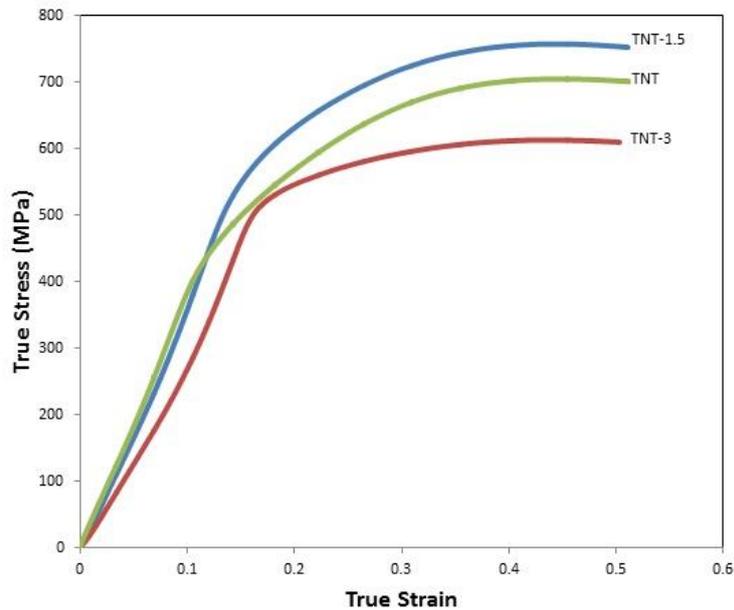


Figure 4. True stress - true strain compression curves for TNT, TNT-1.5 and TNT-3 alloys in the solution treated condition.

A similar study by the research group was done on TNT alloy with addition of Zr element instead of Sn element [12]. That study done on Ti-17Nb-6Ta-3Zr alloy. Zr-addition did not show any significant effect on stabilizing β -phase compared to the original TNT alloy, on the contrary, Sn-addition in this study proved to act like β -stabilizing element. But, for both Zr and Sn addition the microstructure and XRD results agreed that β -phase is the predominant phase, beside lower amount of α'' martensite phase. Hardness measurements showed that Zr-addition to TNT alloy resulted in relatively higher hardness values compared with values of Sn-addition.

4. Conclusion

Sn addition to the Ti-17Nb-6Ta (TNT) alloy stabilizes β -phase and suppresses the α'' martensite phase formation during quenching from austenitizing temperature zone. Hardness, Yield stress and ultimate compression stress increased by the addition of 1.5%at. Sn (TNT-1.5) alloy, however, further Sn addition (i.e., 3% Sn in TNT-3 alloy) proved to have no effect on the yield stress and ultimate compression stress as well.

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