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Influence of the Heat Treatment on the Tribological Characteristics of the Ti-based Alloy for Biomedical Applications

The influence of diverse heat treatments on microstructural and tribological characteristics of Ti-6Al-4V ELI (mass%) alloy, which is used as implant material in biomedical engineering, was investigated on a block-on-disc tribometer. Aim of the present study was to explore the possibility of Ti-6Al-4V ELI alloy wear resistance improvement, examining the effects of different heat treatments on alloy microstructure, as well as on alloy wear characteristics in simulated body conditions using light optical microscopy (LOM) and scanning electron microscopy (SEM). Results presented in this paper show that the influence of heat treatment on microstructural and tribological characteristics of the investigated Ti-based alloy is significant and that it can be used for further wear resistance improvement of this widely used implant material.

Keywords: *Ti-6Al-4V alloy; microstructural characterization; Vickers hardness; wear resistance; LOM; SEM.*

1. INTRODUCTION

Last few decades, because of their outstanding characteristics, titanium based materials are widely used in diverse industry branches, as well as in biomedical engineering [1]. Biomedical and dental application of titanium based materials is in constant increase because of their lightweight characteristics, high corrosion resistance (up to 500 °C), excellent biocompatibility and good balance of mechanical characteristics in wide temperature range (from 200 to 600 °C) [2]. Nowadays most widely used titanium based materials for biomedical applications are CP (commercially pure) Ti and Ti-6Al-4V (mass%) alloy [3,4]. Most commonly these biometallic materials, *i.e.* biocompatible metallic materials, are used for diverse implants manufacturing and as such are implanted into human bodies to compensate the structural components loss of shape or function originated from aging, diseases and accidents. Because of their exceptional mechanical characteristics and excellent electroconductivity, Ti-based biocompatible materials are used for production of diverse medical implants, such as artificial joints, dental implants, artificial hearts, bone plates, staples, wires and stents, as well as for electronic devices, such as pacemaker electrodes and artificial inner ears.

In recent years main concern, for further development of biometallic implant materials, is, among others, stress transmission between hard tissue and biometallic components which are in contact since further bone degradation and bone adsorption should be avoided [5,6]. Namely, great difference between bone and biometallic implant materials rigidity and other mechanical and tribological characteristics may lead to further bone loss and degradation. Since mechanical and tribological characteristics of Ti-6Al-4V alloy are most compatible with bone characteristics, in comparison with other biometallic materials such as stainless steels and Co-Cr based alloys, further development and research of this low rigidity titanium alloy is recently increased. One of the basic concerns for further development of Ti-6Al-4V alloy for biomedicall applications is undoubtedly its fretting and wear resistance, since surface fretting of metallic materials inside the human body causes wear, which leads to successive release of metal ions, metallic compounds and

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debris into the implant surrounding tissues which may provoke toxicity on local tissue [7]. Also, great difference in bone and implant material tribological characteristics may lead to increased bone degradation and failure.

It is well known that the tribological property of Ti-6Al-4V alloy, such as wear resistance is highly dependent on its microstructural characteristics. Heat treatment variations are traditionally used to control the alloy microstructure and in turn its performance. In order to achieve enhanced performance in terms of the wear behaviour, it is highly essential to select appropriate heat treatment procedure. Therefore, the influence of different heat treatment conditions on the microstructure and wear resistance of Ti-6Al-4V alloy was investigated in the present work. The wear behaviour was studied in simulated body fluid solution. The effect of experimental parameters, including applied load and sliding speed on the wear rate of heat treated alloy was also presented.

2. EXPERIMENTAL PROCEDURE

2.1 Material preparation, microstructural characterization and hardness measuremets

The investigated Ti-6Al-4V ELI (Extra Low Intersitial) alloy was industrially produced. Alloy bars, of 38 mm diameter, were supplied by Krupp VDM GmbH, Germany.

The samples were cut from the alloy bars and metallographically prepared prior to heat treatment. Standard techniques, consisting of grinding with SiC paper, polishing by 3 μm diamond suspension and cleaning for 30 min in an ultrasonic bath with ethyl alcohol, were used. The polished samples were afterwards solution treated (ST) at two different temperatures for 1 h under the protective argon atmosphere and followed by water quenching (WQ). One group of samples was subjected to solution treatment at 1000°C, while second group of samples was solution treated at 750°C (above and below the β transus temperature, respectively). The microstructural evolutions of heat treated samples etched with Kroll's reagent were investigated using a Reichert Jung light optical microscope (LOM).

The Buehler Indentamet 1100 Series Semi Macro & Macro Vickers Indentation Hardness Tester was utilized to determine the heat treated alloy hardness values. The Vickers hardness was determined by applying the 300 gf load for 5 s. Obtained hardness values represent the mean value of five measurements performed on the same sample.

2.2 Sliding wear tests

Wear resistance of heat treated Ti-6Al-4V ELI alloy was investigated using the computer aided TR-95 block-on-disc sliding wear testing machine with the contact pair geometry in accordance with ASTM G 77 – 83 standard. The test block was loaded against the rotating steel disc, which provides a nominal line contact Hertzian geometry for the contact pair (Figure 1).

Figure 1. The schematic overview of the contact pair geometry and wear scars created on the sample surface during sliding wear tests.

The test blocks, 6.35 x 15.75 x 10.16 mm in size, were prepared from the heat-treated Ti-6Al-4V ELI alloy. Test blocks contact surfaces were metallographically prepared to a roughness level of $R_a = 0.2$ μm. The counter face (disc with 35 mm diameter and 6.35 mm thickness) was fabricated using the casehardened 30CrNiMo8 steel with hardness of 55 HRC. The roughness of the ground contact surfaces was $R_a = 0.49$ μm.

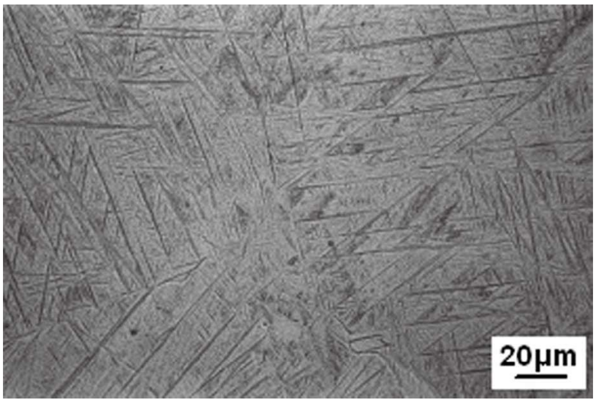
In order to simulate conditions existing in human body, lubricated tribological tests were performed using the Ringer's solution as lubricant. Tests were performed applying 20, 40 and 60 N loads and 0.26, 0.5 and 1.0 m/s sliding speeds for 10 min. During these lubricated tests, the discs were continuously immersed in 30 ml of Ringer's solution up to 3 mm of depth.

Monitoring of the Ti-6Al-4V ELI alloy blocks wear behaviour was conducted by determination of the wear scar width. Using the wear scar width and geometry of the contact pair, calculation of the wear volume and wear rate was achieved. The JEOL JSM-5800 scanning electron microscope (SEM) was used in order to examine the worn surfaces of the tested wear blocks.

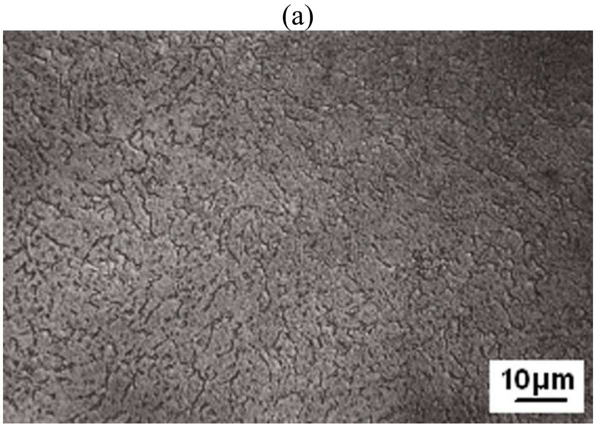
3. RESULTS AND DISCUSSION

3.1 Microstructural characteristics and hardness

Microstructures of Ti-6Al-4V ELI alloy solution treated in the β phase and (α+β) phase field are presented in Figure 2.



(a)



(b)

Figure 2. LOM micrographs of heat treated Ti-6Al-4V ELI alloy specimens. Microstructure of (a) β ST WQ (1000 °C + WQ) and (b) (α+β) ST WQ (750 °C + WQ).

As can be seen from the LOM micrographs, the appearance of produced microstructure strongly depends upon the solution treatment temperature. Very fine platelet-type features in microstructure of β ST samples (Figure 2a) are indicative of martensitic transformation. The presence of hexagonal α′ martensite with some amount of retained β phase in WQ samples is typical of β ST condition. When the solution treatment temperature is decreased, martensite is not formed on water quenching. The microstructure of the (α+β) ST samples consists of primary α and untransformed or retained β (Figure 2b). Namely, much less β phase is present at 750°C than at 1000°C, so that it is enriched with β-stabilizing element vanadium. This reduces the martensitic start (M_s) temperature of β phase to below room temperature. Upon a subsequent water quenching from 750°C, the β phase does not transform into martensite. Thus, the (α+β) ST samples show globular β in α matrix.

As a result, the samples solution treated above β transus exhibited higher hardness than samples solution treated below it. It seems that the formation of α′ martensite in this alloy increases its hardness. The effect of the solution treatment temperature on hardness of the heat treated Ti-6Al-4V ELI alloy is presented in Figure 3.

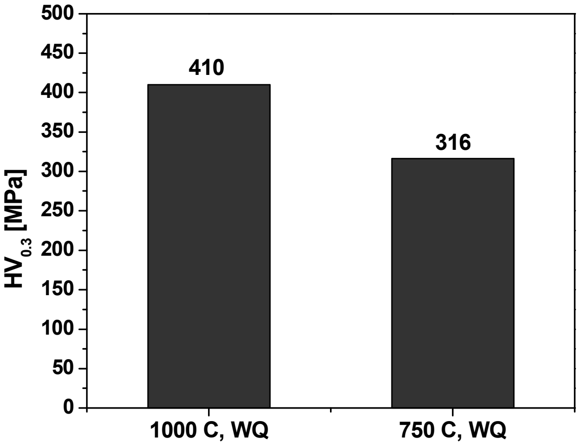


Figure 3. Effect of heat treatment on Ti-6Al-4V ELI alloy hardness.

Water quenching from 1000°C increased the hardness markedly. It has been reported [8] that titanium martensites are relatively soft compared to iron-carbon martensites in steel, because they are only supersaturated with respect to β-stabilizing elements. However, the grain refinement due to the BCC-to-HCP transformation and an increase in dislocation density caused by the rapid cooling to form α′ martensite increase the hardness of β solution treated Ti-6Al-4V ELI alloy. Based on the results of the Vickers hardness measurements it can be expected that the β ST samples show higher wear resistance during sliding tests than the (α+β) ST samples.

3.2 Wear behaviour

The wear rate of heat treated Ti-6Al-4V ELI alloy as a function of sliding speed for the different applied loads is shown in Figure 4.

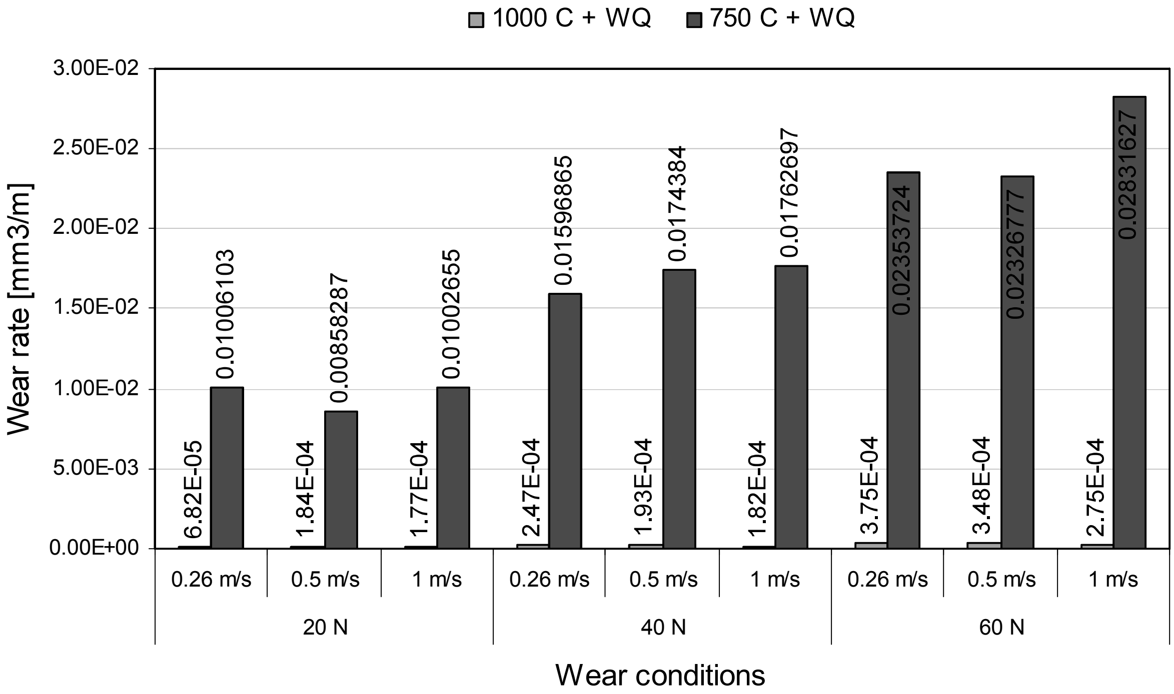


Figure 4. Effect of heat treatment on Ti-6Al-4V ELI alloy wear rate.

In all cases, a significant variation of the wear rate with solution treatment temperature is observed. It is clearly evident that the wear loss is more severe in the (α+β) ST condition. This indicates that the wear resistance of Ti-6Al-4V ELI alloy is very sensitive to heat treatment, which modifies the microstructure. The presence of primary α increases the wear rate substantially. The low wear resistance of the (α+β) ST samples is found even at the applied load of 20 N. At 20 N the wear rate decreases slightly with the sliding speed and shows a minimum at about 0.5 m/s. The wear rates increase as the applied load is increased. But, in the case of the tests at 40 and 60 N, the observed wear rate increases as the sliding speed is increased. As long as the applied loads are low, the contribution of sliding speed to total wear loss is of minor importance, but as the applied load is increased its contribution increases in importance. The wear loss of the β ST samples is much lower than that of the (α+β) ST samples. Their hardness, which is 94 MPa higher than that of the (α+β) ST samples may be the reason for this improvement in wear resistance. The difference in wear mechanisms of two microstructural conditions can also be explained by their difference in mechanical properties. Namely, at all applied loads the wear rate of the β ST samples decreases continuously as the sliding speed is increased. Although the wear rate increases as the applied load is increased, the trends are very similar to those observed at 20 N.

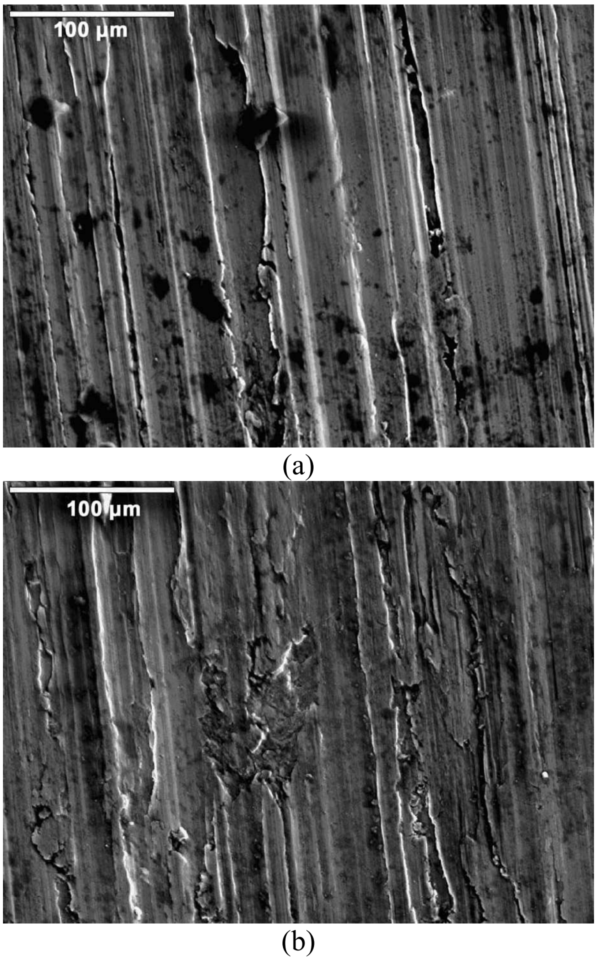


Figure 5. SEM micrographs showing worn surface morphology after sliding under 40 N and 0.5 m/s of (a) β ST WQ and (b) (α+β) ST WQ Ti-6Al-4V ELI alloy.

SEM obervations of the worn surfaces of the β ST and (α+β) ST samples confirm the above results. Figure 5 presents SEM micrographs relevant to the sliding speed of 0.5 m/s and applied load of 40 N. It can be seen that worn surfaces of all samples exhibit similar appearances. Typical characteristics of plowing along the sliding direction and tongue-shaped wedges are observed at worn surfaces of both the β ST and (α+β) ST samples. The wedges, emerged at the worn surfaces of all tested samples, show a plastic deformation characteristic. Formation of a laminar surface structure similar to fracture morphology was also observed on surface of all samples after wear. However, more distinct fracture-like wear traces were detected at the worn surfaces of the (α+β) ST samples (Figure 5b).

Severe embrittlement occurred on the worn surface of these samples showing a large number of cracks indicates more progressive wear of the (α+β) ST samples compared with the worn samples of the β ST alloy (Figure 5a). This phenomenon can be explained by refinement of the alloy microstructure during the β-solution treatment resulting in dissipation of a large amount of energy produced by sliding influencing the higher resistance to cracks formation on the surface of worn specimens [9].

The lamilar structure, which appears at the surface of all worn samples, indicates that some kind of deformation process occurs during sliding wear. The surface of Ti-6Al-4V ELI alloy, at the contact spots, was deformed due to the mechanical and adhesive forces applied by the steel counter disc. Namely, during sliding the material at the contact spots is bunched up and pushed forward [10], forming strip-shaped wedges. Moreover, the wedges were compressed and rolled onto the front surface of the Ti-6Al-4V ELI alloy blocks forming the worn surface morphology showed in Figure 5.

4. CONCLUSIONS

The microstructure of Ti-6Al-4V ELI alloy varied with the heat treatment conditions. The solution treatment above the β transus and subsequent water quenching resulted in martensitic structure, while this alloy solution treated below the β transus and water quenched exibited primary α and untransformed β microstructure. The β solution treatment increased its hardness markedly. The appreciable change in microstructure and hardness appeared as a result of diverse solution treatment temperatures affected the wear resistance of this titanium alloy. Namely, increase in the solution treatment temperature improved its wear resistance in simulated body fluids. Conducted investigations revealed that applied load between 20 and 60 N has a large effect on the wear rate of heat treated Ti-6Al-4V ELI alloy. In both cases, the variation of the wear rate with applied loads was similar. As the applied load was increased, the wear rate increased. However, the effect was greater for (α+β) solution treated alloy than for β solution treated alloy. An increase in sliding speed, on the other hand, showed different trends. Increasing the sliding speed from 0.26 to 1.0 m/s was observed to decrease the wear loss of the β solution treated alloy, whereas increase that of (α+β) solution treated alloy. This result is difficult to explain, but may reflect the complexity of wear mechanism.

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