

Full Length Research Paper

Effect of heat treatment on dry sliding wear of titanium-aluminum-vanadium (Ti-6Al-4V) implant alloy

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Titanium and its alloys have very attractive properties that enable them to be used in the fields of aero space, biomedical, marine and also in many corrosive environments. The application of these alloys is more attractive today in the field of biomedical implant materials due to their superior biocompatibility and strength. These alloys have high coefficient of friction and poor abrasive wear resistance which results in the wear of the implant during its fixation in the body. Implant wear is a common phenomenon which results due to high friction between artificial implant materials when in contact with natural bone - this is much higher than healthy and natural joints that can withstand cyclic loads acting on them. The corresponding wear of the implant results in the accumulation of wear debris in the body tissues which results in inflammation, pain and loosening of implant resulting in shorter life period of the implant. Heat treatment of the alloy is one of the important techniques to improve the sliding wear properties of the alloy. The property of poor abrasive resistance can be altered by changing the microstructure of the alloy where the formation martensitic structure (acicular α or retained β) is resulted. The formation of martensitic structure in titanium alloy results in improved hardness value with a subsequent improvement in its sliding wear behavior. In this work, the implant material is subjected to heat treatment above its transformation temperature followed by slow cooling in furnace, air and water. These specimens were further aged and tested for dry sliding wear properties against hardened steel disc using a pin-on-disc apparatus using weight loss method with an optimal load of 50 N and a sliding distance of 500 m. An improvement of wear rate is reported under different heat treatment condition and an analysis of wear track of the specimens is done using scanning electron micrographs (SEM).

Key words: Biomaterial, heat treatment, martensitic structure, dry sliding wear.

INTRODUCTION

Titanium and its alloys have been extensively used today to replace the natural bone joint with artificial joints. The important correlation of bone substitute materials include biocompatibility with hard and soft tissue, an elastic modulus near that of bone and tensile ,compressive strength and fracture toughness equal to or greater than that of toughness equal to or greater than that of bone. In addition to fatigue strength, the wear resistance of the material should guarantee a safe operation of the implant

during the expected period of use (Boehrlart et al., 2008).

Degradation of artificial implant materials due to high wear rates can lead to unfavorable biological effects on bone density and implant fixation, resulting in shorter lifetime. Therefore, wear resistance is an important property while considering a suitable implant material. The wear resistance is directly proportional to the contact stress and friction at the part of contact and the type of heat treatment the material is subjected to. The type of heat treatment in a particular solution treatment of the implant results in the formation of acicular martensitic structure which greatly improves the hardness and wear resistance of implant.

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Wear resistance plays an important role whenever a material (a bone plate for fracture fixation) is attached to fractured bones of a different stiffness values and modules of elasticity; relative movements between the two different materials (bone and implant) and between the parts of multi-component systems must occur when a cyclic load is applied to the system. These relative movements causes a wear stress on the attachment devices (bone screws as well as on the eyelets). Therefore, high wear resistance is required for orthopedic implants to obtain biocompatibility and acceptability (Thomann, 2000).

Titanium exists in various allotropic forms. At low temperatures, it has a closed packed hexagonal crystal structure known as α , whereas above 883°C, it has a body centered cubic structure known as β . The α to β transformation temperature of pure titanium either increases or decreases based on the nature of the alloying elements. The alloying element such as aluminum, oxygen, nitrogen, etc., that tend to stabilize are called α stabilizers and the addition of these elements increase beta transus temperature, while elements that stabilize β phase are known as β stabilizers such as vanadium, molybdenum, niobium, iron, etc., and addition of these elements depress the β transus temperature. Some of the elements such as zirconium and stantium which do not have marked effect on the stability of either of the phase but form solid solutions with titanium are termed as neutral elements (Geetha et al., 2009).

Mitsuo (2008) reported that the application of stress by rapid quenching results in the formation of martensitic structure in steels which contain residual austenite in their microstructures. This phenomenon is called stress or strain induced martensitic transformation which enhances ductility or fracture toughness of steel. Deformation induced martensitic transformation also occurs in titanium where unstable β phase is retained at room temperature by rapid cooling such as water quenching from a high temperature near the β transformation temperature.

The draw back of extensive use of titanium alloys in hip replacement and other artificial joints is poor tribological properties such as poor abrasive wear resistance, poor fretting behavior and high coefficient of friction. The improvement of the above properties can be done with the help of four main mechanisms as suggested by Zhecheva et al. (2005). They are as follows:

- i. To induce a compressive residual stress.
- ii. To decrease the coefficient of friction.
- iii. To increase the hardness.
- iv. To increase the surface roughness.

The surface modification and change in microstructure can be obtained by heat treating the various samples at beta transus temperature (transformation temperature) where primary α changes from hexagonally closely packed crystallographic structure (α) to body centered

cubic crystallographic structure (β). These specimens were aged to complete the transformation of retained beta in order to achieve strengthening of the alloy.

Wear rate calculation by pin and rotating disc machine using weight loss method is one of the common techniques to evaluate dry sliding wear behavior of Titanium implant materials. Molinari et al. (1997) investigated on dry sliding wear mechanism of titanium-aluminum-vanadium (Ti-6Al-4V) alloy. In their experimental work, it has been found that the wear volume of the rotating specimens is reported as a function as sliding speed and the load applied on the pin. An increase in wear volume results with an increase in applied load. Alam et al. (2002), in their experimental work of dry sliding wear, reported that under a constant load of 45 N applied on pin, an increase of wear rate was identified up to a sliding distance of 500 m. There after, a steady state is attained; In this condition no appreciable change in the wear rate behavior of the alloy was observed.

Venkatesh et al. (2009) reported an improvement in various mechanical properties when $\alpha + \beta$ alloys were heat treated above the beta transus temperature and cooled by water quenching, air cooling and furnace cooling. Ajel et al. (2007) have studied the influences of heat treatment on Ti-6Al-7Nb implant alloy, where strength of the alloy is reported due to change in the alloy microstructure.

Loads acting on human joint vary considerably from joint to joint. For a particular joint, it varies with time during the loading cycle (Majumdar et al., 2008). It has been reported that stresses in the living area are of the order of 1 MPa. Gispert et al. (2007) used a normal pressure of 0.88MPa. In this work a load of 50 N with a contact pin diameter of 8mm is used to obtain a pressure of 1 Mpa which is considered to be a safe stress acting on the joint during the loading conditions.

It is evident that from this literature that the effect of various types of heat treatment influences the various changes in the respective microstructures which further results in obtaining tailor made mechanical properties and tribological properties of the implant alloy. In this work, dry sliding wear tests are conducted on Ti-6Al-4V implant alloy subjected to various heat treating conditions. These tests were conducted by considering an optimal load of 50 N with sliding distance of 500 m under a sliding velocity 1 m/s Microstructure evaluation along with Scanning electron microscope (SEM) analysis is done in support of the experimental work.

MATERIALS AND METHODS

The chemical composition (% by weight) of the alloy is as follows: 89.6% titanium, 6.29% aluminum, 3.95% vanadium, 0.09% iron, and 0.029% carbon.

The pins were cut according to the standard dimensions as shown in Figure 1 by wire EDM process employing a brass wire of nominal diameter of 0.3 mm. For Microstructure analysis all the cut specimens were mechanically polished via a standard

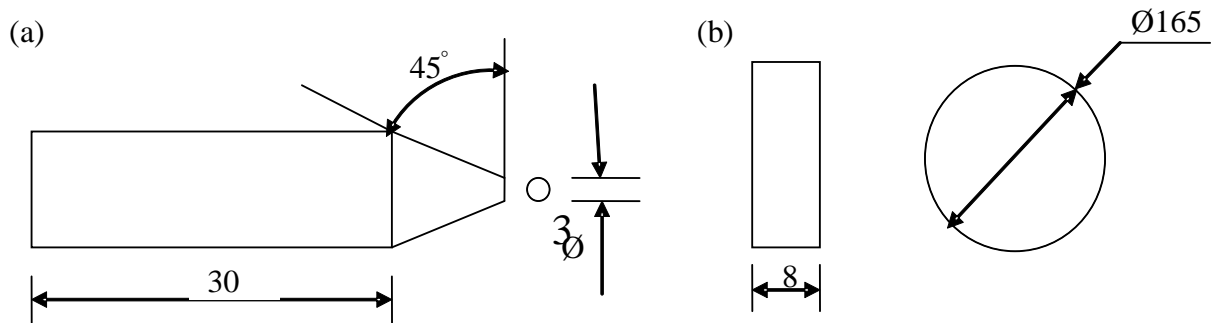


Figure 1. Dimensions of the (a) friction pin; (b) rotating disc in millimeters.

metallographic procedure to a final level of 0.3 μm alumina powder and etched with a solution of water, nitric acid and hydrofluoric acid (80:15:5 in volume). Scanning electron micrograph (SEM) analysis was also conducted to study the wear track behavior of various heat treated specimens. Hardness values were measured by Vickers micro hardness testing machine with a constant load of 0.5 kg. Average surface roughness (Ra) value of the wear track was measured by surface roughness testing instrument.

In this work, three samples of each condition were heat treated at 950°C for one hour in an argon controlled atmosphere for a period one hour. It is then aged at 550°C for a period of three hours in an air controlled furnace and subsequently cooled under various cooling environments such as cooling in a furnace, air and quenching in water. These specimens were then tested by DUCOM wear testing machine and the results are reported.

RESULTS AND DISCUSSION

Microstructure

Figure 2a represents the microstructure of the 'as received' alloy at room temperature showing equiaxed $\alpha + \beta$ matrix. Figure 2b reveals the formation of lamellar microstructure of α plates surrounded by prior β grain boundary which is due to the influence of slow cooling in the furnace. Figure 2c represents more number of primary α grains surrounded by needle like transformed or retained beta which is formed as a result of moderate cooling of the alloy in the air. Figure 2d represents acicular or needle like α in the matrix of primary α and β . Further, the scanning electron micrograph of quenched specimen shown in Figure 3 represents the presence of acicular (needle like) martensitic structure (α') in white globular primary α in $\alpha + \beta$ matrix. Equiaxed microstructures shown in Figure 2a often have high ductility as well as fatigue strength and are preferred for super plastic deformation, while lamellar microstructure shown in Figure 2b have high fracture toughness and show superior resistance to creep and fatigue crack growth. The formation of bimodal microstructure as shown in Figures 2d and 3 combine the advantage of both equiaxed and lamellar microstructures. The presence of acicular martensitic structure greatly improves the hardness values and also its ultimate tensile strength

(Leyens and Peter, 2003). The microstructures have been consistent with respect to the work done on this alloy by Jha et al. (2010) and Molinari et al. (2010).

Wear rate analysis

Figure 4 shows the wear rate behavior of the alloy under different heat treating conditions. Table 1 shows various properties associated with sliding wear of the implant alloy. Presence of high amount of wear is reported from the wear testing of 'as received' material. This corresponds to low hardness value which is Vickers hardness number (VHN) 311 as compared to quenched specimen which is having a hardness value VHN value of 380. High amount of hardness values in quenched specimen is due to the presence of acicular α' (martensitic structure) which was formed due to heat treatment above beta transus temperature followed by water quenching and aging. The wear rate as shown in Table 1 is also high as in the case of 'as received' material when compared to air cooling or water quenched specimens. The wear rate of furnace cooling specimen is greater than the water quenched and air cooled specimens due to the formation of lamellar α plate like structure where there is no presence acicular α' or retained beta as shown in Figure 2b. This is due to slow cooling of the specimen where complete transformation of body centered cubic crystallographic (β) structure to hexagonally closely packed (α) structure has taken place. This transformation to hexagonally packed structure limits plastic deformation of the furnace cooled specimen due to which higher wear rate is obtained

Weight loss and wear track analysis

Maximum amount of weight loss has been reported from 'as received' material as shown in Figure 5, where as less amount of wear is reported from both water quenched and air cooled specimens. The results commensurate with the hardness values and microstructure behavior of all the heat treated specimens.

Surface roughness values measured perpendicular to

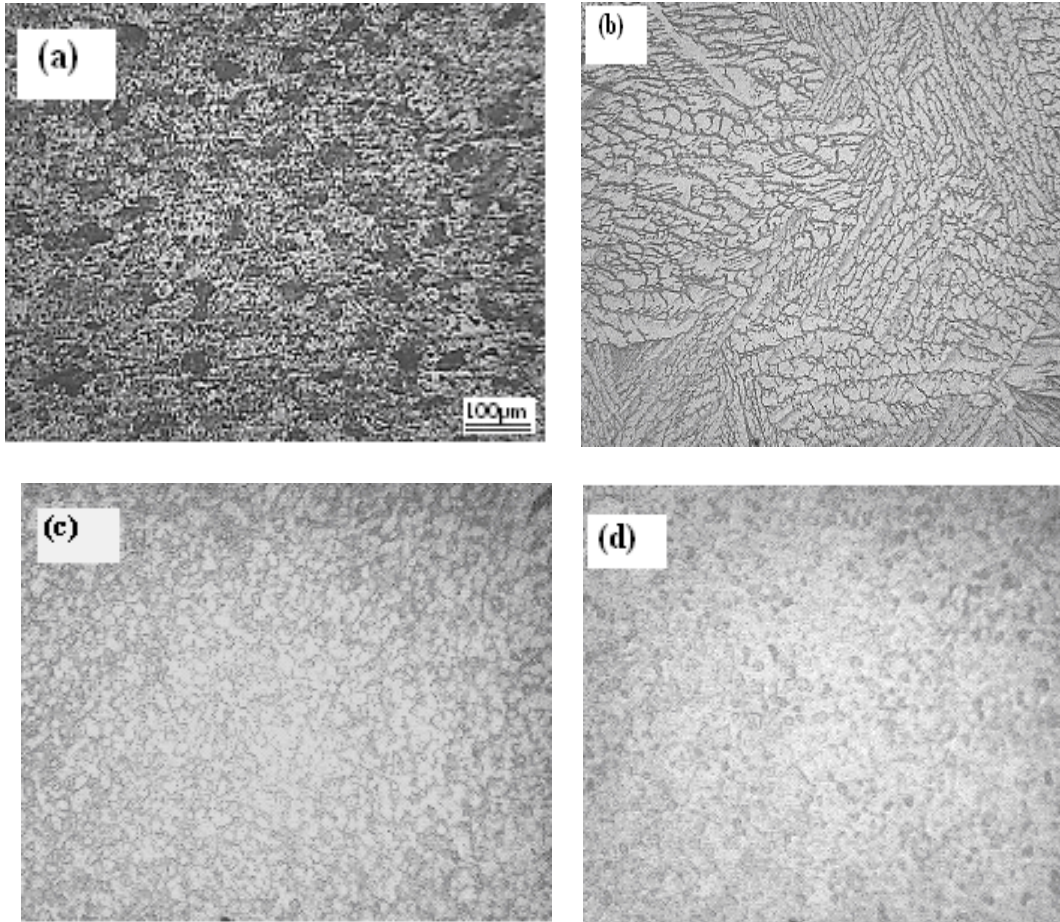


Figure 2. (a) Microstructure of 'as received' material; (b) furnace cooled and aged; (c) air cooled and aged; (d) water quenched and aged specimen at a Magnification of 100X.

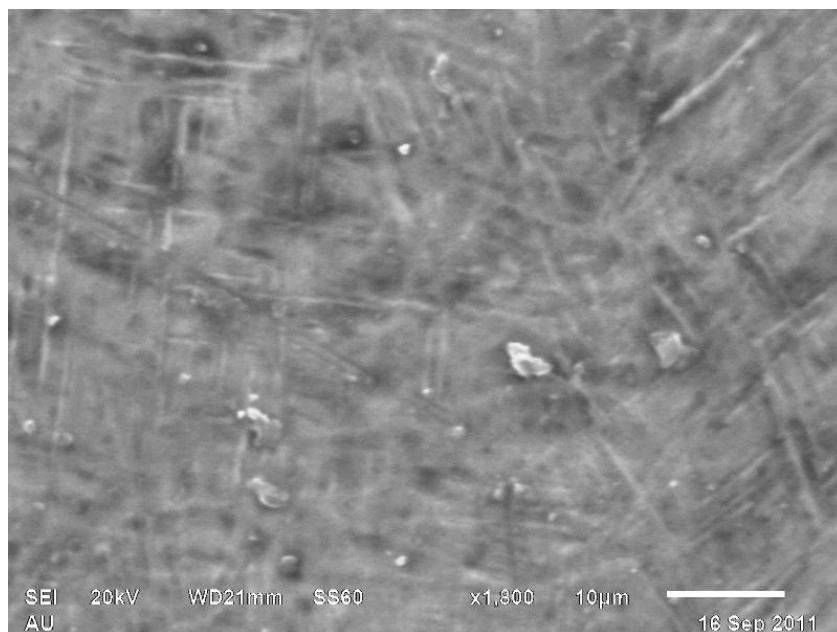


Figure 3. Scanning electron micrograph of quenched specimen.

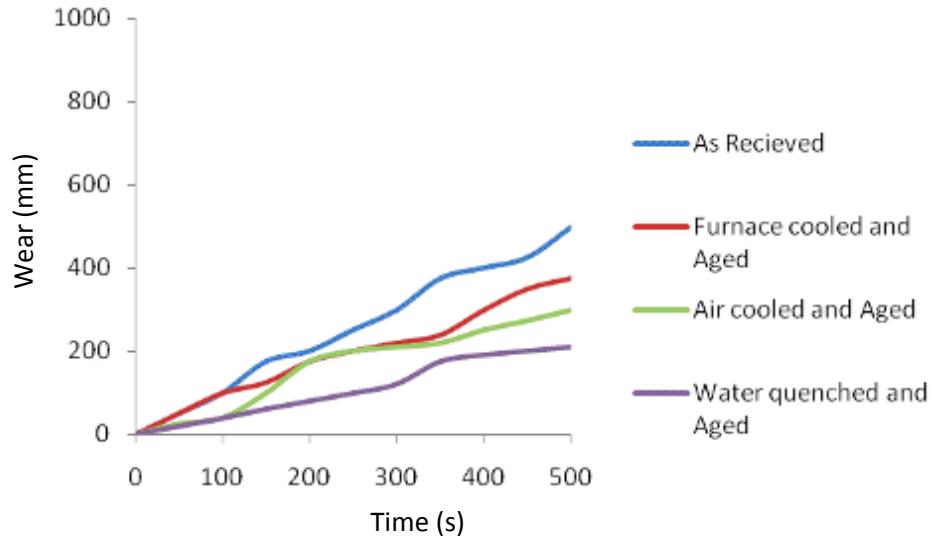


Figure 4. Wear behavior of the alloy under various heat treatment conditions.

Table 1. Wear properties under various heat treating conditions.

Heat treated condition	Wear track surface roughness (Ra)	Micro hardness (HV _{0.5})	Wear rate x 10 ⁻¹¹ m ³ /m
As received	2.11	311	1.954
Furnace cooled and aged	1.29	351	0.93
Air cooled and aged	0.858	340	0.186
Water quenched and aged	1.411	380	0.139

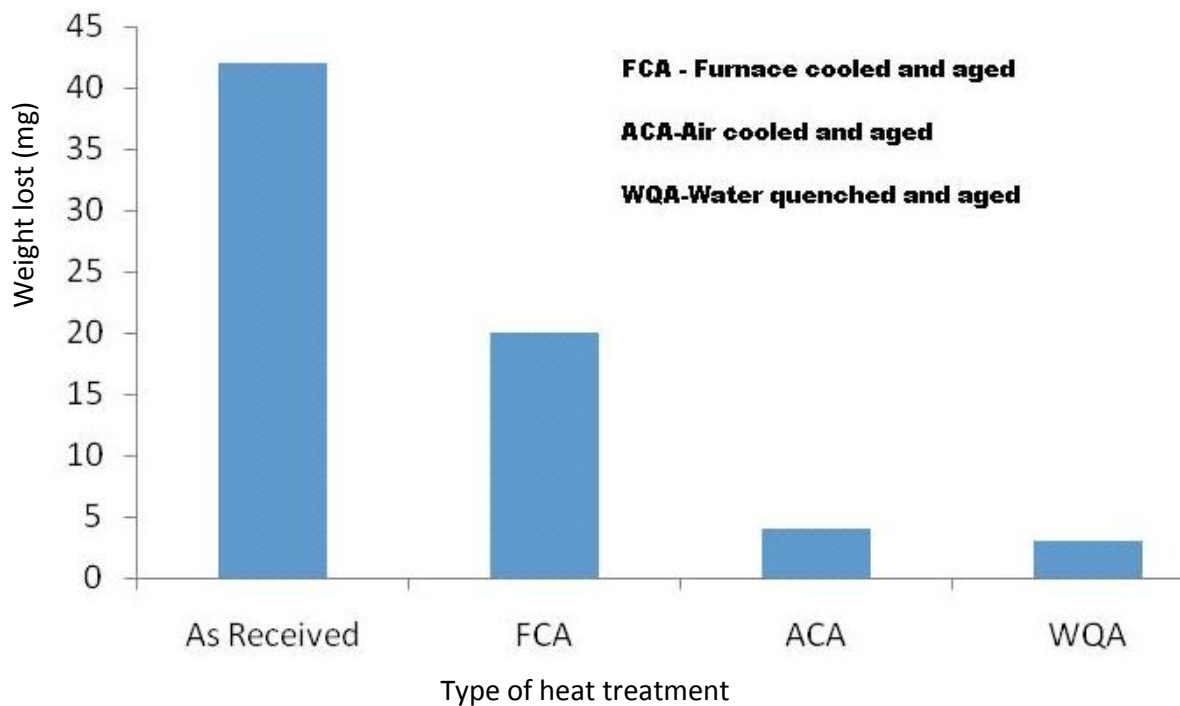


Figure 5. Weight loss of the alloy after heat treatment.

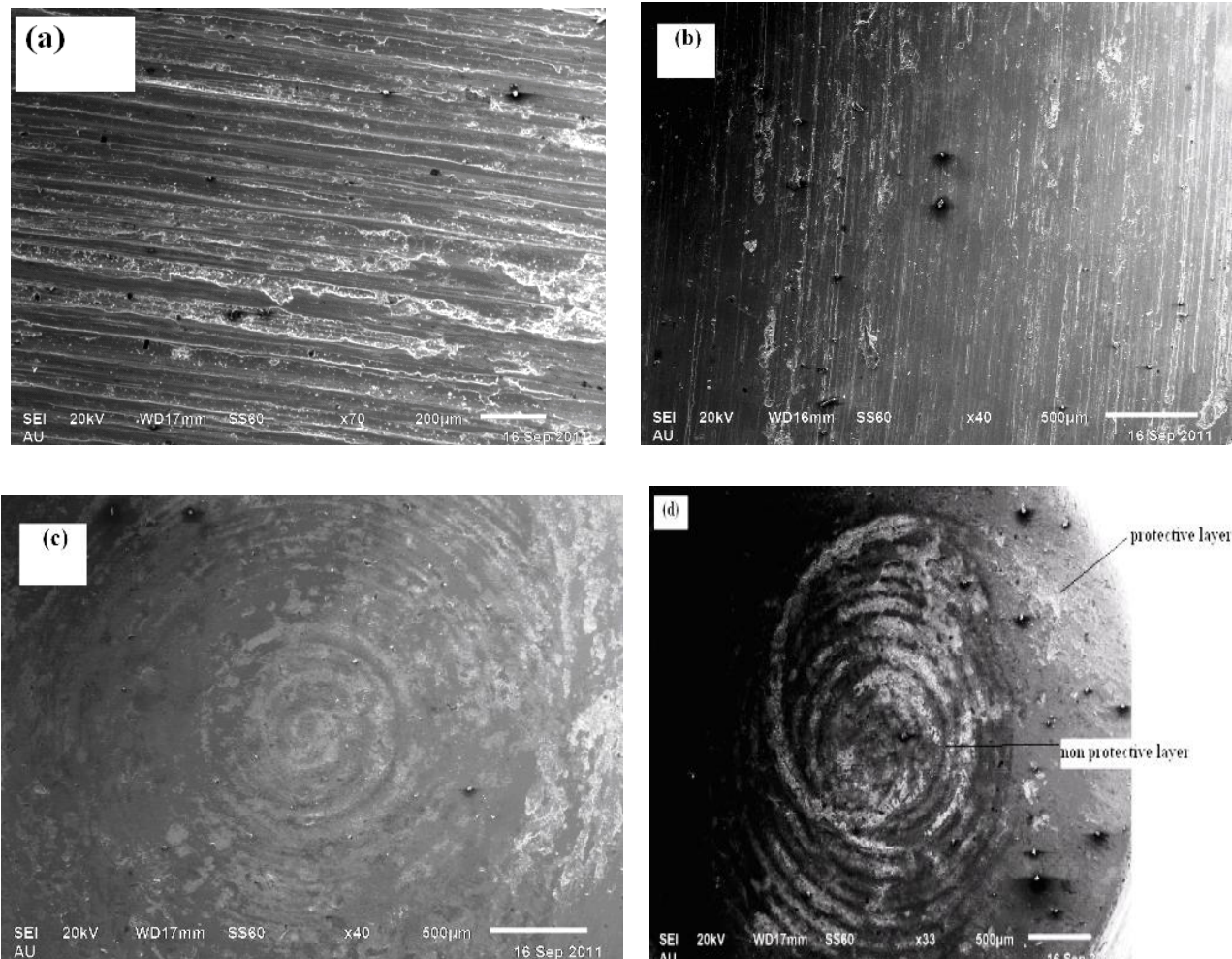


Figure 6. (a) Wear track of 'as received' material; (b) furnace cooled and aged; (c) air cooled and aged; (d) water quenched and aged specimen.

the wear track are given in Table 1. The wear rate of as received material is high due to low hardness of the specimen. This is due to coarse wear track developed during the testing procedure. The surface roughness value of water quenched specimen is moderately higher than that of the other heat treated specimens due to formation martensitic structure by heat treatment which resulted in an increase in the hardness value.

The wear track of 'as received' specimen as observed in scanning electron microscope (SEM) is coarse and thick with the presence of white titanium oxide globules as shown in Figure 6a. The wear track of furnace cooled specimen is moderately fine as compared to base metal specimen. Both the air cooled and water quenched specimens shown in Figure 6c and d accounts for the presence of protective layer coating. The coating is only formed due to faster rate of cooling, where the specimen is subjected to water quench or cooling in air. This coating, along with the martensitic (α') in the microstructure of the quenched specimen, play an important role in

limiting the wear rate when subjected to the above conditions. There is no presence of protective layer which can be observed from Figure 7a and b, when the implant alloy is heat treated at 600°C and simultaneously cooled by air and water. This is due to the fact that oxidation of the specimen has not occurred below the transformation temperature of the implant alloy. It is also clear that presence of retained beta or acicular alpha will take place only during heat treatment above transformation temperature with cooling in water and air medium. The corresponding wear track of these specimens shows very fine track layer of wear with no protective layer present. From the energy dispersive spectrometry analysis (EDS) shown in Figure 8a, a clear picture of the constituent elements is available. The protective layer mainly consists of carbon along with significant amount of oxygen which indicates that the specimen has undergone oxidation. It is evident from the literature that when titanium and its alloys are exposed to oxygen containing atmosphere, it results in the formation of an oxide

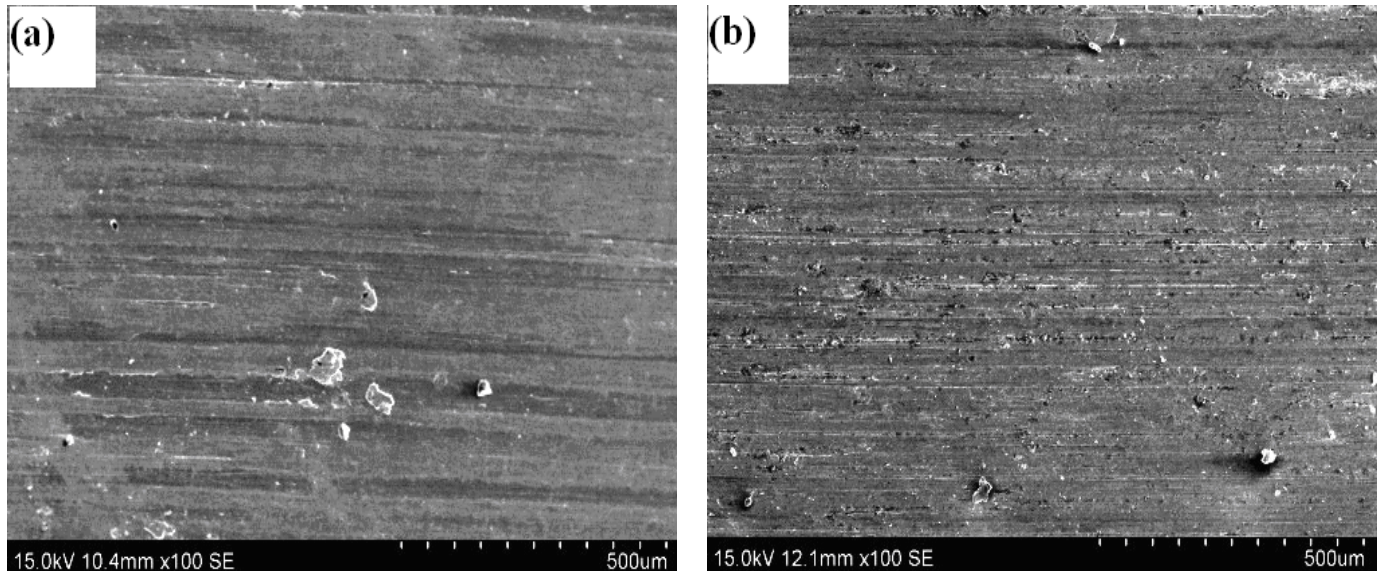


Figure 7. (a) Wear track of water quenched specimen heat treated at 600°C; (b) furnace cooled specimen.

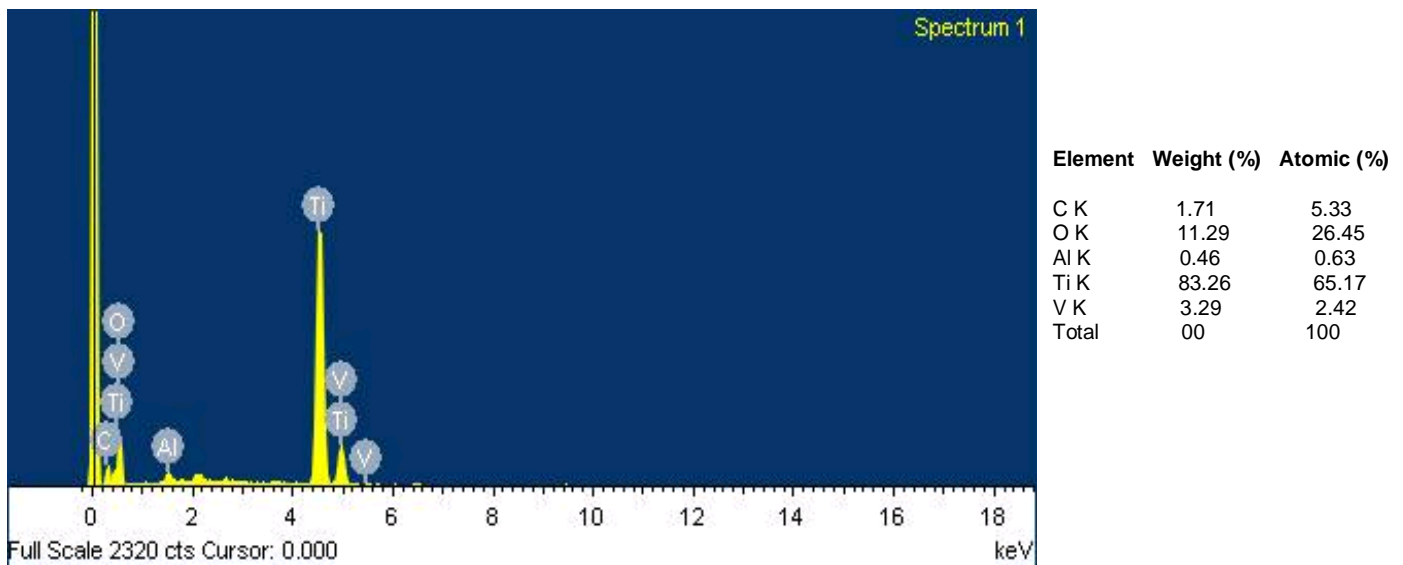


Figure 8a. EDS analysis of protective layer.

layer on its surface with an oxygen diffusion zone beneath it (Guleryuz and Cimenglu, 2008). This formation is more pronounced when the alloy is heat treated above transformation temperature and cooled rapidly in air or quenching by water. This formation further plays an important role in developing a protective layer which promotes remarkable advantage of the alloy while working in a friction and wear environment. The presence of protective layer is crucial in improving corrosive and wear resistance. The presence of carbides which would have formed during heat treatment also plays an important role in minimizing the weight loss as well as the

wear rate of the quenched specimen when in contact with the rotating disc. On the other hand, more amount of iron is detected from the non protective layer shown in Figure 8b, indicating adhesive wear of the alloy specimen while in contact with the sliding steel disc.

Conclusions

i. The wear rate of quenched specimen is very low due to the presence of protective oxide coating layer formed during heat treatment and also due to the presence of

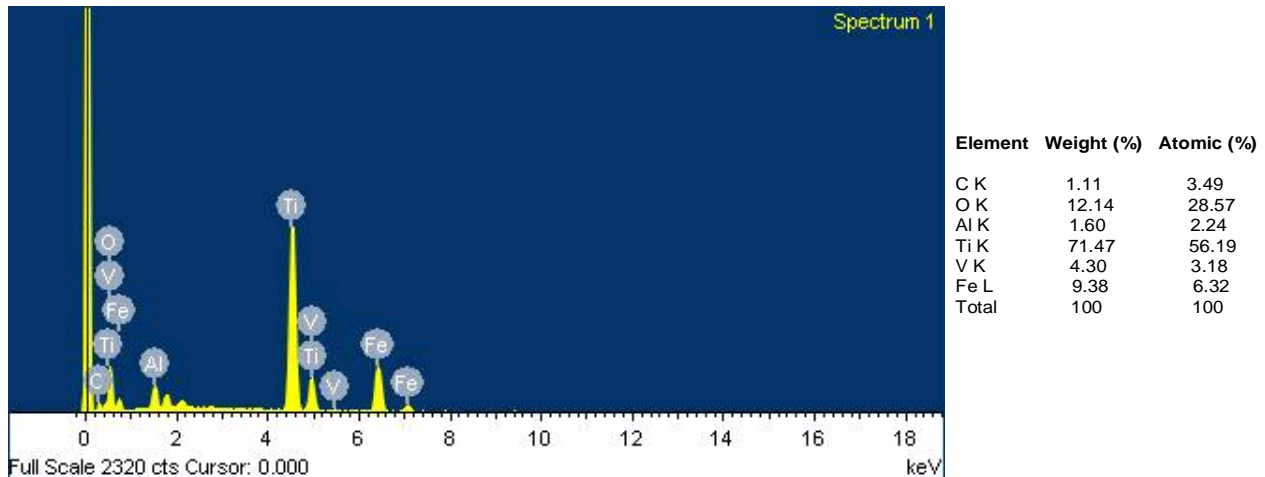


Figure 8b. EDS analysis of non protective layer.

acicular martensitic structure (retained β) in its microstructure.

ii. Finer wear tracks were observed when the specimen hardness is increased during rapid quenching and air cooling of the specimen below the transformation temperature

iii. Finer α grains in $\alpha+\beta$ matrix were observed in all the microstructures which are formed as a result of aging where complete transformation of retained β has taken place to α .

iv. Formation of protective oxide has taken place only in quenched and air cooled specimens, which indicates the presence of oxide layer at times of faster rate of cooling the alloy below the transformation temperature

v. Formation of a protective oxide layer plays an important role in improving corrosive resistance and wear resistance of the implant alloy.

vi. Ti-6Al-4V implant alloy due to its $\alpha+\beta$ binary alloy composition results in obtaining various dry sliding wear properties when it is heat treated above its transformation temperature and cooled through various medium such as furnace, air and water..

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