



Research Paper

EFFECT OF HEAT TREATMENT PROCESS ON
TRIBOLOGICAL BEHAVIOR OF Ti-6Al-4V ALLOYSabry S Youssef^{1*}, Khaled M Ibrahim² and Mohammad Abdel-Karim¹*Corresponding Author: Sabry S Youssef, ✉ ssy00@fayoum.edu.eg

Titanium alloys are extensively used in various fields of engineering, medicine, aerospace and marine due to their excellent mechanical properties. Their usages are more pronounced today in the field of biomedical implants due to its superior bio-compatibility, corrosive resistance and high strength to weight ratio. Ti-6Al-4V alloy is high strength material that can be used in aerospace applications. Ti-6Al-4V has been found to have a better combination of high strength (UTS >1500 MPa) and good ductility (total elongation >10%) than many other Ti-alloys. It has been reported that the natural oxide film that forms on titanium and its alloys easily rub off, thus leaving the substrate in a state of high friction and low wear resistance. Thus, it was important to enhance the tribological properties of Ti-6Al-4V alloy through controlling the microstructure with no loss in mechanical properties by applying solution treatment at different temperatures above and below the beta transus. In this paper, therefore, the influence of the solution treatment at 1050 °C for 30 min with water cooling followed by aging treatment at 500 °C for 24 hr and then air cooled was investigated on the specimens from Ti-6Al-4V titanium alloy.

Keywords: Titanium alloys, Ti-6Al-4V, heat treatment, Solution treatment, Aging treatment

INTRODUCTION

Titanium (Symbol Ti, melting point: 1,670 °C, density 4.5 g/cm³) (Lütjering and Williams, 2003) is the fourth most abundant industrial metal in the earth's crust (0.61%), after aluminum (8.14%), iron (5.12%), and magnesium (2.10%) (Boehlar *et al.*, 2008). Although unalloyed titanium metal is soft and exhibits low strength, its alloys demonstrate

exceptional mechanical properties. The uses of commercially pure titanium are limited to applications where moderate strength, high corrosion resistance, and good weldability are desired (Thomann and Uggowitzer, 2000).

The remarkable properties of titanium alloys with regards their high strength, wear resistance, and low density are well known in aerospace related engineering circles.

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Beyond the aerospace sector, the usefulness of titanium alloys is also being realized in other industrial sectors that include petroleum refining, chemical and food processing, surgical implantation (biomedical industry), nuclear waste storage, automotive and marine applications (Kornilov, 1954).

Commercially pure titanium has an all-alpha structure and demonstrates superior resistance to corrosion but inferior mechanical properties as compared to titanium alloys. Compared with beta titanium alloys, alpha titanium alloys are superior in heat resistance and weldability but inferior in strength and workability (Weiss, 1963). Beta titanium alloys are alloys which are solution strengthened by adding beta structure stabilizers. An all-beta structure at room temperature can be obtained by rapidly cooling the specimen through solution treatment. Alpha phase precipitates in an all-beta structure by aging treatment. Alloys having a beta structure with precipitated alpha phase exhibit excellent strength. Two phase $\alpha+\beta$ alloys with a dispersion of the beta form in the alpha phase exhibit properties of each phase.

The Ti-6Al-4V in particular, is a general purpose $\alpha+\beta$ alloy that accounts for about 60% of all titanium production (Boyer, 1996; and Hughes *et al.*, 2004). This alloy is heat treatable and in annealed condition, it can replace many of the non-heat treatable alloys owing to its good strength and stability at

temperatures up to 400 °C (Thomann and Uggowitzer, 2000). The service temperature for this alloy is reported to be in the range of 400-483 °C (Thomann and Uggowitzer, 2000; and Hughes *et al.*, 2004) while operating at higher temperatures could result in titanium fire (Jaffery and Mativenga, 2009).

The aim of this work is to scutinize the effect of solution treatment at different temperatures above and below the beta transus on tribological behavior of Ti-6Al-4V alloy through controlling the microstructure with no loss in mechanical properties. The alloy was characterized after heat treatment using Metallographic investigation, tensile test, hardness test and tribological test.

EXPERIMENTAL PROCEDURE

The material used in this work was cast Ti-6Al-4V alloy, which was melted in induction Vacuum furnace. The chemical composition of the studied alloy is given in Table 1.

To remove the residual stress existing over the cast surface bars due to casting, 2.5 mm was removed from the surface by turning. The final dimension of the cast rods is then 25 mm diameter and 250 mm long.

Hot swaging was done at 900 °C to reduce the cast bars diameter from 25 mm to 8.5 mm in 11 steps. After swaging, the Ti-6Al-4V alloy was heat treated using two different regiems;

Table 1: Nominal Chemical Composition of Experimental Ti-6Al-4V Alloy

Nominal Composition	Average Chemical Composition, Wt. %						
	Al	V	C	O	Fe	Si	Ti
Ti-6Al-4V	6.37	4.12	0.02	0.25	0.24	0.02	Bal.

The first treatment was carried out by heating the Ti-6Al-4V alloy to 1050 °C for 30 min and then water quenched. After that, aging process was performed at 500 °C for 24 hr and finally air cooled.

The second treatment carried out by heating the Ti-6Al-4V alloy to 1050 °C for 30 min, cooled within a rate of 1 °C/min down to 800 °C, held at this temperature for 30 min and then water quenched. After that, aging process was performed at 500 °C for 24 hr and finally air cooled.

The as-cast and heat treated specimens were prepared by standard metallographic techniques which consist of polishing and etching in an etchant composed of 3 mL of HF, 30 mL of HNO₃, and 67 mL of H₂O. Then the specimens were investigated and photo micrographed using light microscope.

Vickers hardness test with applied load of 20 kg was used to determine the hardness values of the studied samples. All hardness measurements were performed in the middle part (core) of the specimens. The average Vickers hardness of the specimens was measured.

Tensile tests were conducted on an Instron model 5582 testing machine using crosshead speed of 1 mm/min. Threaded cylindrical specimens having gage length and gage diameter of 25 and 5 mm, respectively, were prepared for tensile testing. Three specimens were tested for each sample and the average was measured.

Wear test was carried using pin-on-ring tribometer testing machine. A cylindrical pin specimen was fixed against rotating stainless steel ring, with a surface hardness of 63 HRC.

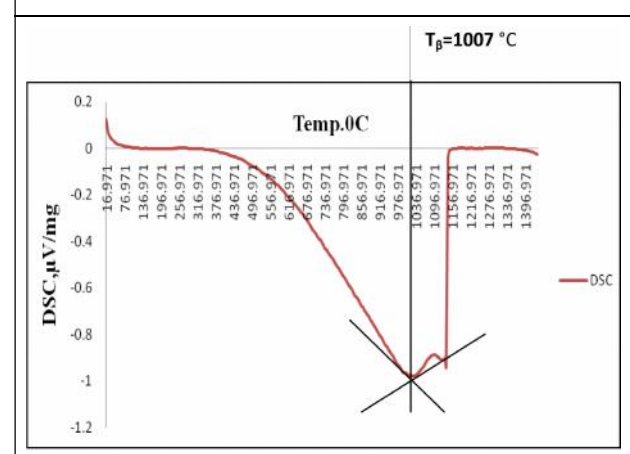
RESULTS AND DISCUSSION

Differential Scanning Calorimetry (DSC) for s-Transus Detection

In Ti-6Al-4V alloy, It has been shown that two endothermic peaks between begin and end of r/s phase transformation. The lowest temperature peak was interpreted as the phase transformation of secondary α transforming into β phase and the second temperature peak was identified as the phase transformation of primary α into β phase up to the beta transus temperature (Sha and Malinov, 2009).

In this study, the DSC test was used to determine the beta transus for the as cast condition of the studied Ti-6Al-4V alloy, as shown in Figure 1. It is noticed that the studied Ti-6Al-4V alloy exhibited a beta transus temperature of about 1007 °C.

Figure 1: DSC Curve of Cast Ti-6Al-4V



Microstructure Investigation

Ti-6Al-4V alloy is characterized to be sensitive to microstructural variations. Where many researches have been performed to obtain desired mechanical properties by controlling the microstructure through heat treatments (Morita *et al.*, 2005; and Cai *et al.*, 2011).

The microstructure of as-cast Ti-6Al-4V alloy is two-phased, consists of α and β solid solutions. The lamellae of the α phase are relatively regular and are mutually connected in a form of basket weave. Between these phases are thin areas of β phases. By the boundaries of the prior α grains, phase α can be formed which delimits these grains (Pinke *et al.*, 2012).

In this study, the microstructure of Ti-6Al-4V alloy in the as-cast showed a structure consists of equiaxed β -grains and various morphologies of α -phase, Figure 2a.

On the other hand, the swaged samples showed a structure consists of very fine equiaxed $\alpha + \beta$ structure because these samples were exposed to hot swaging process at 900 °C (i.e., at $\alpha + \beta$ temperature range), Figure 2b. Critical examination for this figure indicates that, the coarse as-cast structure, Figure 2a, is broken down into very fine equiaxed $\alpha + \beta$ structure, Figure 2b, by applying hot swaging process which is normally occurred by severe plastic deformation below the β -transus (~ 1007 °C).

The microstructures of the investigated Ti-6Al-4V alloy that was subjected to different solution treatments and then applied to consequently ageing treatment at 500 °C for 24 hrs are shown in Figure 3.

In this figure, a fine lamellar structure is formed when samples were rapidly quenched in water from 1050 °C in which this temperature is considered to be above the β -transus (~ 1007 °C) for the investigated Ti-6Al-4V alloy, Figure 3a. Fine Lamella (FL) microstructure shows different shapes of α , α -grain boundaries and colonies of α structure that consist of lamellar α colonies inside the prior β -grains. These colonies seem to be a continuous α or discontinuous α . However, the other samples that were quenched in water from 800 °C obtained a coarse lamellar structure as shown in Figure 3b. Coarse Lamella (CL) microstructure contains colonies of α within the prior coarse β -grains. On some selected β grain boundaries, α -plates nucleate and grow towards the β -grain interior (acicular α). This acicular structure of α was presumably formed during cooling from the β transition

Figure 2: Microstructure of the Investigated Ti-6Al-4V, a) As-Cast, b) Swaged

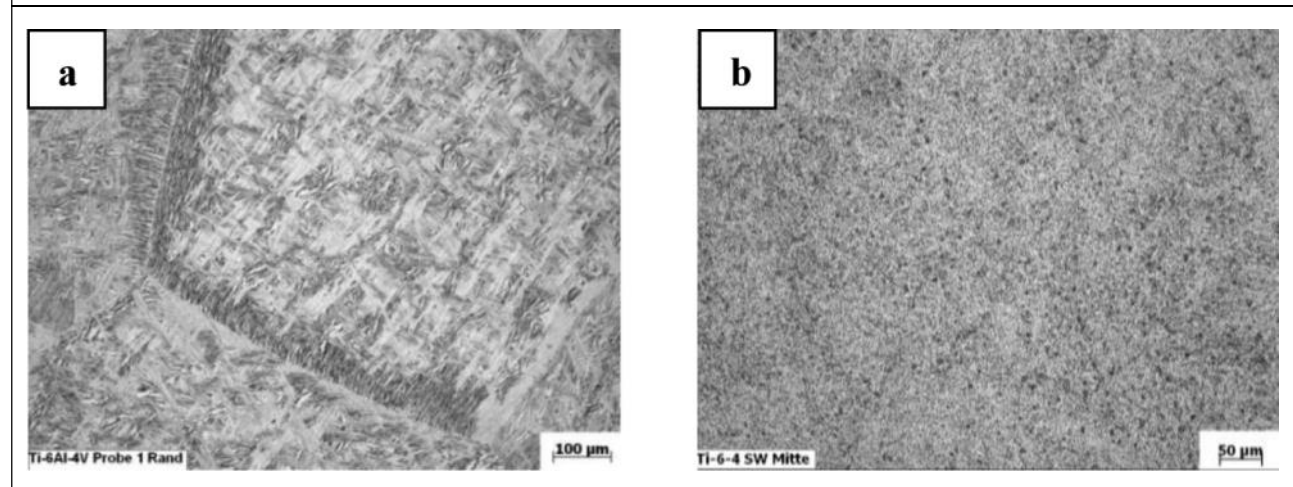
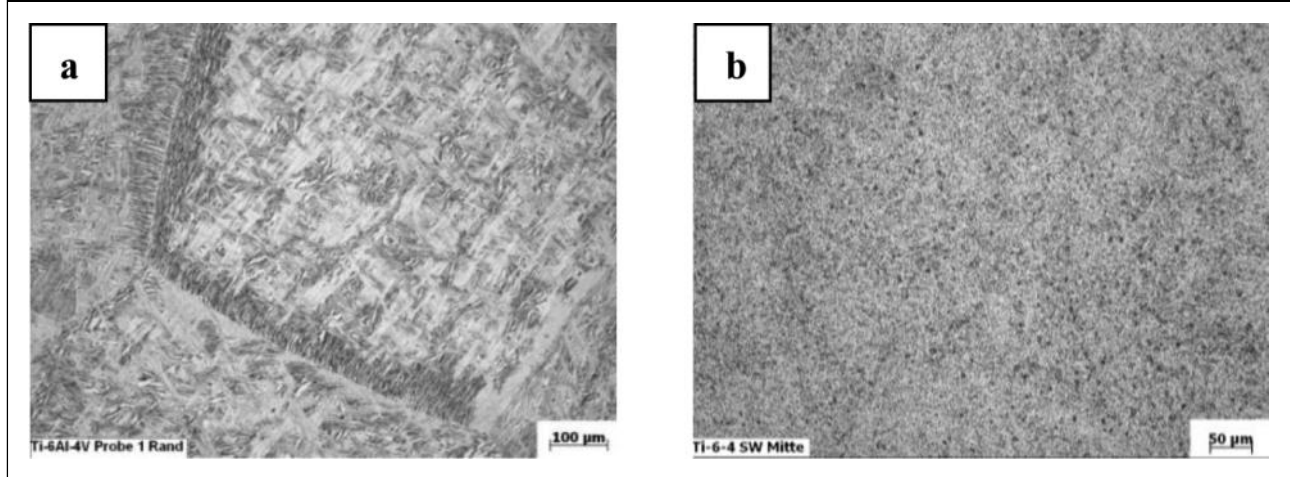


Figure 3: Microstructure of the Investigated Ti-6Al-4V, a) FL, b) CL



temperature (1050 °C) to 800 °C and form the coarse lamellar structure.

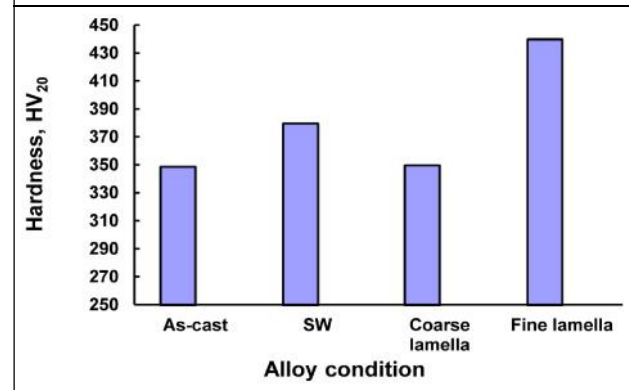
Mechanical Properties of Ti-6Al-4V Alloy

Hardness

Vickers hardness measurements were carried out to investigate the effects of applying hot swaging and heat treatment processes on the studied Ti-6Al-4V alloy. The results of hardness for the as-cast, swaged and heat treated conditions are shown in Figure 4. The as-cast condition obtained the lowest hardness value of 349 HV due to the presence of coarse s-grains and also existing of relatively heterogeneity in the r1+ s structure. The swaged samples obtained higher hardness compared to the as-cast condition due to the refining effect caused by applying severe plastic deformation on the cast structure, where the grain size decreased from 650 μm in the cast condition to 100 μm in the swaged one.

Solution treatments and subsequent ageing at 500 °C for 24 hrs lead to an increase in hardness comparing with the as-cast and swaging conditions.

Figure 4: Hardness Values of Ti-6Al-4V Alloy at Different Conditions



The increase of hardness can be ascribed to the decomposition of martensite structure, i.e., $r' \rightarrow 1r + s$. This is because, if the martensite structure is not formed after the solution treatment, the hardness during the aging treatment will rise probably as a result of precipitation of the fine r phase from s phase, i.e., metastable $s \rightarrow \text{fine } r + s$ (Pinke *et al.*, 2012).

The heat treated fine lamellar structure showed a hardness value reaches to about 440 HV. While, the heat treated coarse lamellar structure obtained a lower hardness value of 350 HV as compared to the fine lamellar

structure due to existing a higher amount of r -phase.

Generally, minimum hardness was obtained for the as-cast structure due to the coarse grain structure compared to the coarse lamellar, swaged and fine lamellar structure. On the other hand, maximum hardness was revealed for fine lamellar structure of about 440 HV.

Tensile Properties

The tensile properties of the investigated Ti-6Al-4V alloy were determined and showed that the as-cast structure obtained an UTS of 1050 MPa which is compatible for the $r+s$ structure with a coarse grain size, Figure 5. Moreover, the as-cast structure has a large degree of heterogeneity which has an influence on the tensile properties. By applying severe plastic deformation (swaging process) on the cast structure, the UTS increased to 1140 MPa due to increasing the dislocation density as well as having a very fine grain size.

In addition, solution treatments and subsequent ageing at 500 °C for 24 hrs lead to notable increase in the UTS in comparison with the as-casting condition. Hence, the heat

treated fine lamellar structure showed the maximum UTS (1300 MPa). Such increase can be ascribed to the formation of r' martensitic plates inside the $r+s$ structure. While, the heat treated coarse structure showed relatively lower UTS value (1000 MPa) compared to fine lamellar structure.

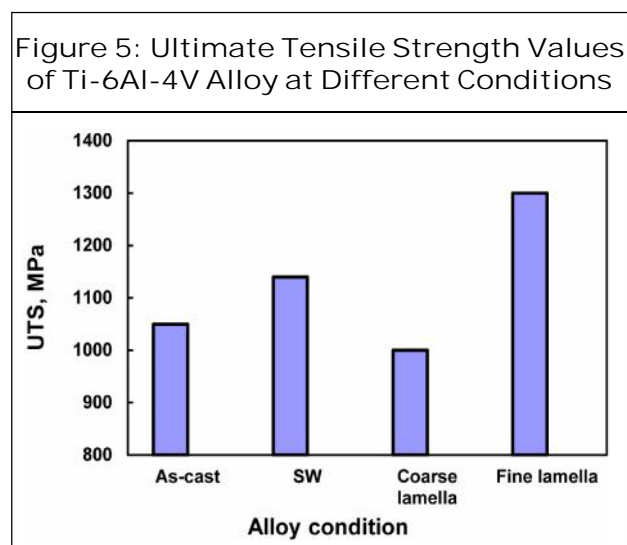
Wear Behavior of the Studied Ti-6Al-4V Alloy

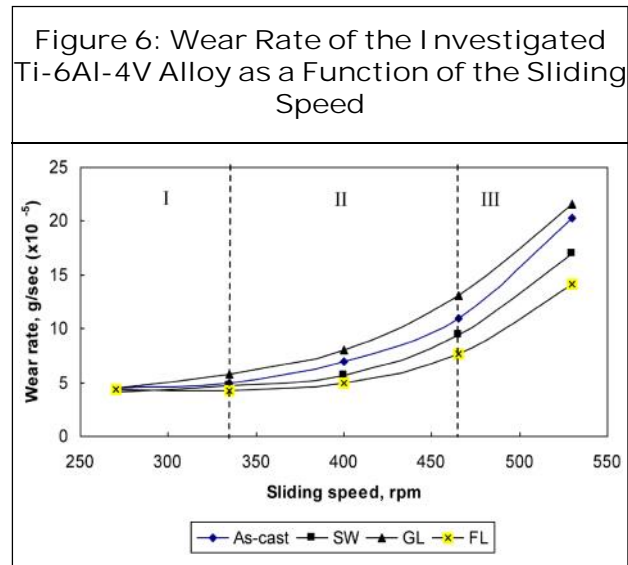
The wear test was conducted on the investigated Ti-6Al-4V alloy for all conditions (as-cast, SW, FL and CL) by using pin-on-ring Tribometer testing machine for determining the weight rate.

In order to correlate well between the different conditions of the studied Ti-6Al-4V alloy, the relation between sliding speed and wear rate is presented. Figure 6 illustrates the wear rate of the investigated Ti-6Al-4V alloy as a function of sliding speed. The estimated values of wear rates in all conditions indicated that the wear rates increase with increasing the sliding speed.

The heat treated samples with fine lamellar structure has smaller real area of contact as a result of increasing hardness. Since the wear rate is directly proportional to the real area of contact, where the heat treated samples with fine lamellar structure showed a lower wear rate. The influence of the sliding speed on the wear rate is different for all conditions.

The wear rate of the as-cast samples significantly changed with increasing of the sliding speed. Initially, wear rate is rapidly increased with increasing sliding speed up to 335 rpm, then showed a then showed a steady state condition till 465 rpm and finally rapid





increase with further increase of sliding speed of 530 rpm. This can be explained in three different stages as shown in Figure 6.

As clearly seen from this figure, the first stage (I) showed a very high amount of wear rate due to the first contact between the sample and the rotating ring. In such case, the wear rate will be high till coinciding the sample surface with the ring. Because the as-cast and the coarse lamella samples have a low hardness, they showed a high amount of wear rate in stage (I). With increasing the material hardness, the wear rate will be lower in stage (I). Just the samples are coincided with the ring; the wear rate will also increase significantly as defined by stage (II). With increasing sliding speed, the temperature of the sample will be also increased. In such case, a relative softening will be happened and then the wear rate will be increased. This phase is defined by stage (III), Figure 6.

The worn surfaces of some selected samples have been examined using SEM. The morphological analysis of the wear tracks confirms the above results. The SEM

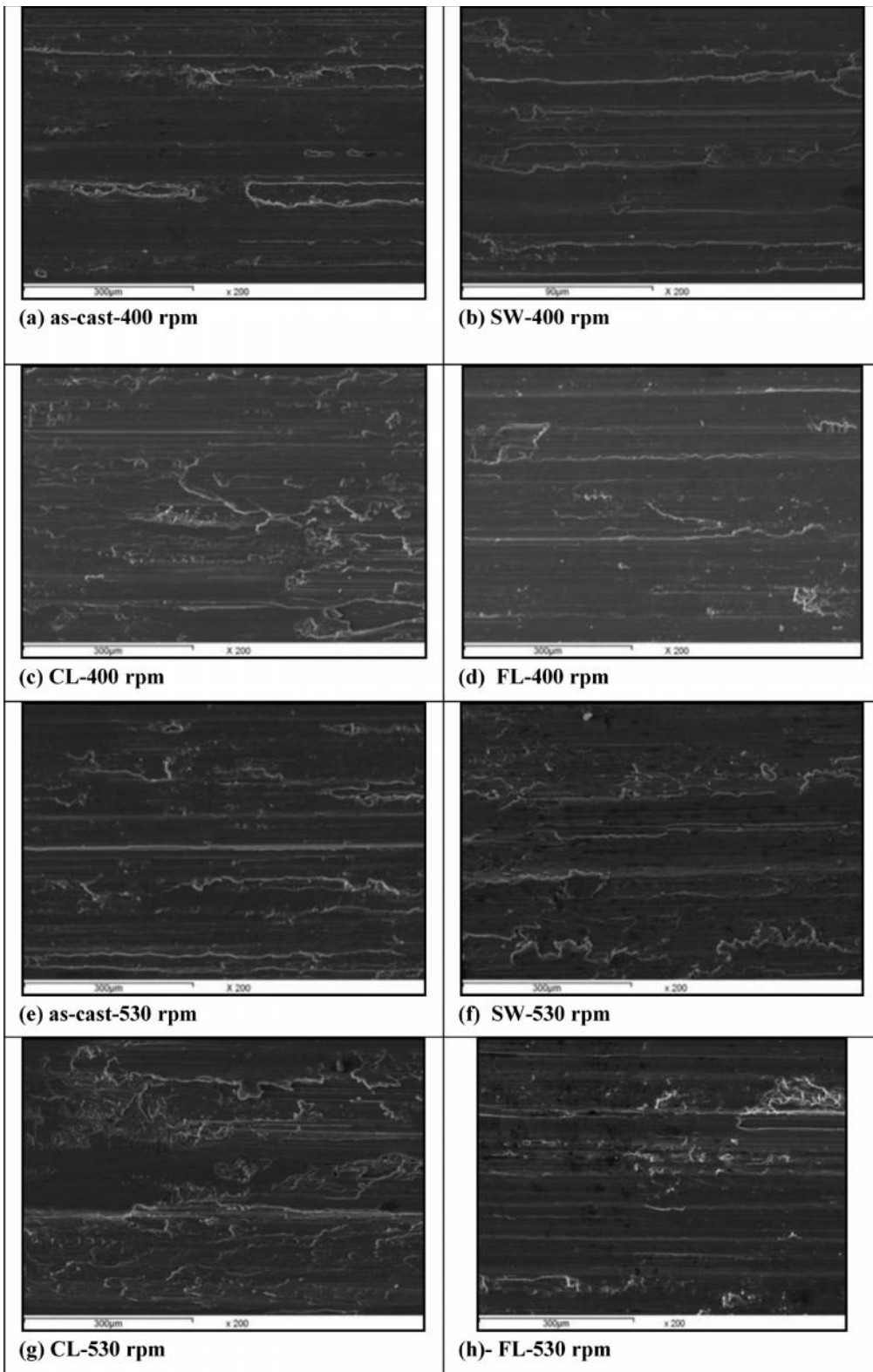
micrographs presented in Figure 7 show the typical worn surface morphologies of the specimens tested at the low, medium and high sliding speeds of 400 and 530 rpm, respectively. Some evidences of abrasion wear can be detected in all tested samples. Continuous sliding marks with plastically deformed grooves are also seen on the wear tracks independently of the sliding speed.

In addition, the extent of plastic deformation or ploughing is found to be higher in case of high sliding speed (530 rpm), Figures 7e-7h. The medium speed (400 rpm) showed moderate worn surface that can be described as tire track wear mode, Figures 7a-7d.

The removal of materials by adhesion is related to large wear loss and it appears that the wear rate can be determined by the contribution of adhesive wear to total wear. Thus, it can be observed that coarse lamella structure obtained the highest wear rate because it undergoes severe delamination, as shown in Figure 7g.

The SEM examination also shows that at least two wear mechanisms can be observed in this study. Lamination wear mechanism that can be seen at low sliding speed and it seemed to be more clearly for the heat treated samples with coarse lamellar structure because of its low hardness value, Figure 7c. While, delamination wear mechanism is more prominent when wear test is carried out at high sliding speed and its feature is more visible in case of low hardness, as shown in Figure 7g, for the heat treated samples with coarse lamellar structure.

Figure 7: Worn Surfaces of Ti-6Al-4V Alloy at Different Sliding Speeds



CONCLUSION

- The as-cast equiaxed $r_1 + s$ structure is broken down into very fine equiaxed $r + s$ structure by applying hot swaging at 900 °C.
- A fine lamella structure was obtained by rapid quenching in water from 1050 °C, and a coarser lamella structure was obtained by quenching from 800 °C.
- A more suitable combination of hardness, tensile properties, and wear resistance can be achieved by hot swaging and then rapid quenching in water from 1050 °C.
- The fine lamellar structure gave the lowest wear rate. While, the coarse lamellar structure showed the highest wear rate.
- Observation of the worn surfaces showed a lamination wear mechanism at low sliding speeds and delamination wear mechanism at high sliding speeds. 🌀

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