

Research Article

Characterization of Microstructure and Tribological Study on Heat Treated Titanium alloy (Ti-6Al-4V)

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Abstract

There is a growing demand for titanium. The aircraft industry is the driving force behind the demand for titanium. Titanium alloy exhibits an excellent combination of bio-compatibility, corrosion resistance, strength and toughness. The influence of diverse heat treatments on microstructural and tribological characteristics of Ti-6Al-4V alloy, which is used as implant material in biomedical engineering, Aerospace applications has been investigated on a pin-on-disc. Aim of the present study is to explore the possibility of heat treated Ti-6Al-4V alloy wear resistance improvement, examining the effects of different aging conditions on alloy microstructure and mechanical properties, as well as on alloy wear characteristics under different wear conditions on heat treated Ti-6Al-4V alloy using Design of Experiments.

Keywords: Titanium alloy, Wear, Heat treatment, Micro Hardness, Taguchi's Design of Experiment

1. Introduction

There is a maximum demand for titanium. The aircraft industry is the driving force behind the demand for titanium. The reason for this is that titanium has attractive properties that justify its use both economically and environmentally. Titanium alloys have superior strength-to-weight ratios. This implies that by substituting components manufactured from other metals in the aircraft with titanium components, a substantial reduction in structural weight can be achieved. Last few decades, because of their outstanding characteristics, titanium based materials are widely used in diverse industry branches, aerospace as well as in biomedical engineering (Niinomi, *Met al*, 2008). Biomedical and dental application of titanium based materials is in constant increase because of their lightweight characteristics, high corrosion resistance, excellent biocompatibility and good balance of mechanical characteristics in wide temperature range (C.R.Brooks, 1982). Cvijovic-Alagic *et al*, 2008 suggested that most widely used titanium based materials are Ti-6Al-4V alloy. According to Niinomi, *Met al*, 2004 and Capitanu, L, *et.al*, 2008, in recent years main concern, for further development of bio-metallic implant materials, mechanical and tribological characteristics may lead to further bone loss and degradation. One of the well-known heat treatments is solution treatment and quenching plus aging. Rodney B *et al*, 1994 Suggested that with the series of heat treatment, the tensile strength of Ti-6Al-4V alloy

will be increased. Peter .P and Mihalireger, 2002 have studied the influence of the solution treatment at 1050⁰C, 950⁰C and 800⁰C with water or air cooling followed by aging treatment at 550⁰C was investigated on the specimens from Ti-6Al-4V. They have found that heat treatment of casted Ti-6Al-4V alloy show that α martensitic structure is formed after water cooling from the solution treatment at 1050⁰C. They have also found that air treatment from each solution temperature lead to lamellar structure of $\alpha+\beta$ phases and the character of the formed microstructure has not changed after the aging temperature at 550⁰C. Kenneth G Budinski, 1991 has conducted experiment to study on most widely used titanium alloys such as grade 2 commercially pure titanium and the age hardenable Ti-6Al-4V. He conducted experiment on dry sand-rubber wheel test and pin on disc test to determine the best counter for these two titanium alloys for tribo system.

It is well known that the tribological property of Ti-6Al-4V alloy, such as wear resistance is highly dependent on its micro structural characteristics and mechanical property. Heat treatment variations are traditionally used to control the alloy microstructure and in turn to improve its mechanical property in order to achieve enhanced performance (Park, Y.J, *et.al*, 1998; Krol, S, *et.al*, 2001; Jovanovic, M.T, *et.al*, 2006; RehamReda, *et.al*, 2013; Mithunkuruville, *et.al*, 2008).. In terms of the wear behavior 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 has been investigated in the present work.

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The microstructure has been studied using metallurgical microscope and scanning Electron microscope, surface hardness has been studied using Vickers hardness tester and wear behavior has been studied using Pin on Disc wear testing machine. The effect of process parameters based on Design of Experiments on wear rate of heat treated titanium alloy has been presented.

2. Methodology

Figure 2.1 shows Microstructure of the primary [alpha] and transformed [beta] phases. The age hardening was carried out in Muffle furnace (figure 2.2) and wear tests were carried out by pin on disc wear testing machine illustrated in figure 2.3. The pin material was heat treated Ti-6Al-4V alloy. The disc material was EN-31 steel with a hardness of 640 HV. The pin specimens were cylinders with a diameter of 8 mm and a height of 20 mm. The disc specimens were cylinders with an outer diameter of 100 mm and a thickness of 8 mm. The difference in the mass measured before and after the test gives the wear of the specimen. The mass loss of the pin (specimen) was measured in an electronic weighing machine with a least count of 0.001 g. The ratio of mass losses was defined as wear rate.

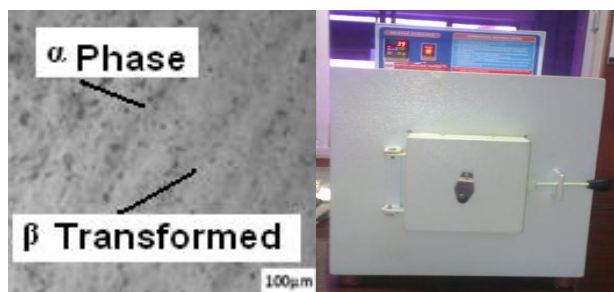


Figure 2.1Microstructure of the primary [alpha] and transformed [beta] phases **Figure 2.2**Muffle Furnace



Figure 2.3Experimental setup for Wear test on Pin on Disc

Ti-6Al-4V alloy is age hardened at 750°C, 825°C, 1000°C for aging time 30 min, 60 min and 90 min under room temperature, water and cryogenic quenching using Taguchi’s design of experiments based on L₉ orthogonal array. Table 2.1 shows selected Levels and Factors for micro hardness measurement. After getting the optimum

Micro hardness based on Taguchi’s design of experiments on age hardened Ti-6Al-4V alloy the specimen is carried out with wear analysis on pin on disc wear testing machine under different Load (L/N), Sliding Speed (m/s) and Sliding Distance (D/m) based on Taguchi’s design of experiments based on L₂₇ orthogonal array. Table 2.2 shows selected Levels and Factors for wear measurement followed by micro hardness. Micro structural analysis of age hardened and worn specimen was done using Trinocular Inverted Metallurgical microscope (figure 2.4).

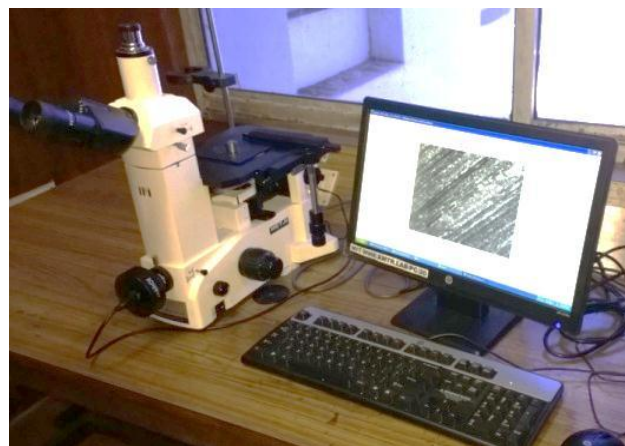


Figure 2.4Trinocular Inverted Metallurgical Microscope

Table 2.1Levels and Factors (Taguchi’s Methodology)

Levels	Aging Time (min.)	Aging Temperature (°C)	Solution Treatment
1	30	750	RQ
2	60	825	WQ
3	90	1000	CQ

Table 2.2Levels and Factors (Taguchi’s Methodology)

Levels	Load (L/N)	Sliding Speed (m/s)	Sliding Distance (D/m)
1	9.81	1.67	1500
2	19.6	2.51	5250
3	39.2	3.35	9000

3. Results & Discussion

3.1 Age hardened Ti-6Al-4V alloy Micro hardness measurement

The micro hardness measurements of Ti-6Al-4V alloy were done using Vickers indentation hardness tester. A load of 300 gf and an indentation time of 10 seconds were applied. Micro hardness measurement was done on each aged sample. In order to obtain good statistics, three indentations spaced 1 mm apart were made on each sample. The resulting values of micro hardness were then calculated automatically for individual indentations. The variations in the micro hardness with Aging Time (min.), Aging Temperature (°C) and Solution Treatment were then analyzed using L₉ orthogonal array to obtain optimum condition for micro hardness. Trinocular inverted Metallurgical microscope images of age hardened surface

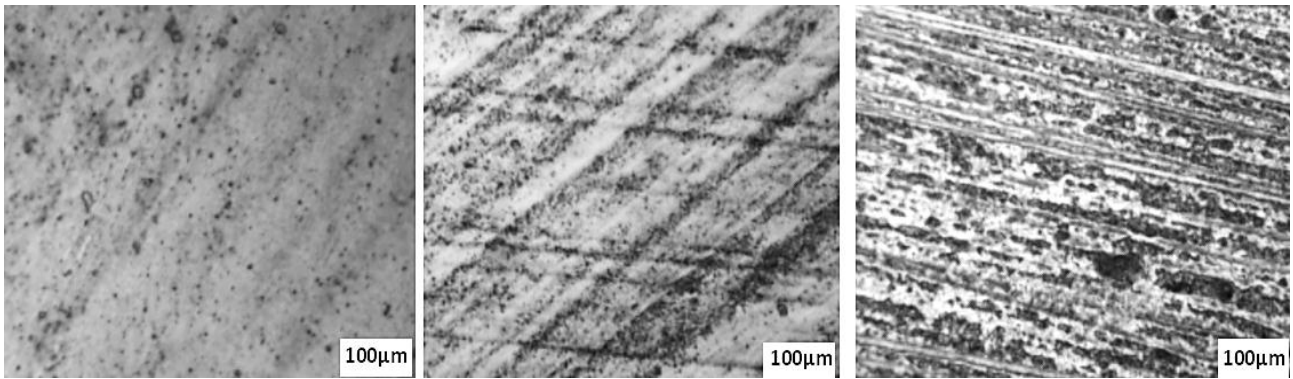


Figure 3.1 Trinocular inverted Metallurgical microscope images of age hardened surface under different aging condition (a) Room Temperature quenching; (b) Water quenching; (c) Cryogenic quenching

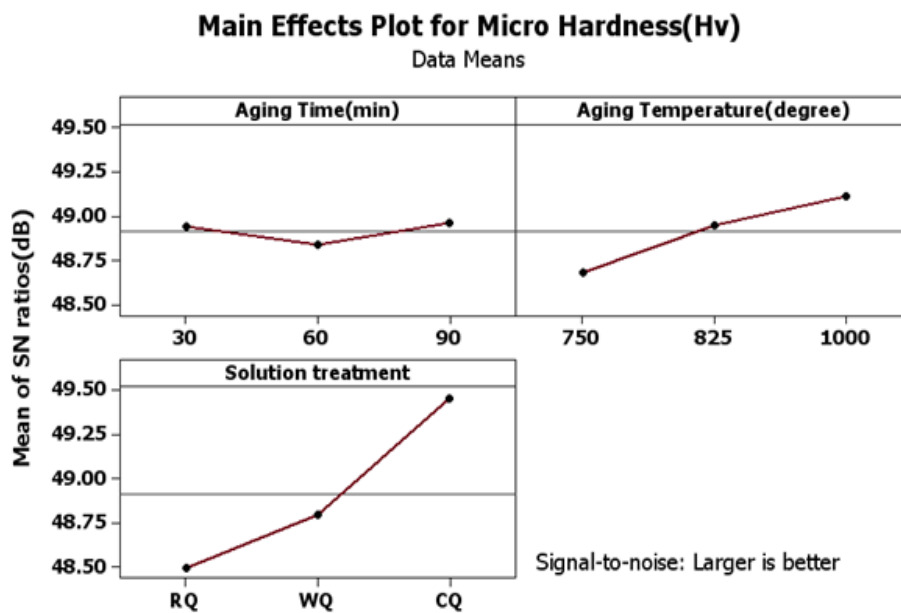


Figure 3.2 Mean S/N graph for micro hardness

Table 3.1 Response Table for Signal to Noise Ratios (Larger is better)

Level	Aging Time (Min.)	Aging Temperature (°C)	Solution Treatment
1	48.94	48.68	48.50
2	48.84	48.95	48.79
3	48.96	49.11	49.45
Delta	0.12	0.43	0.96
Rank	3	2	1

Table 3.2 Analysis of Variance for S/N ratios

Source	DF	Seq SS	Adj SS	Adj MS	F	P	Percent P (%)
Aging Time (Min.)	2	0.02569	0.02569	0.012846	6.47	0.134	1.47
Aging Temperature (°C)	2	0.28416	0.28416	0.142079	71.57	0.014	16.20
Solution Treatment	2	1.44463	1.44463	0.722314	363.56	0.003	82.33
Residual Error	2	0.00397	0.00397	0.001987			
Total	8	1.75845					100

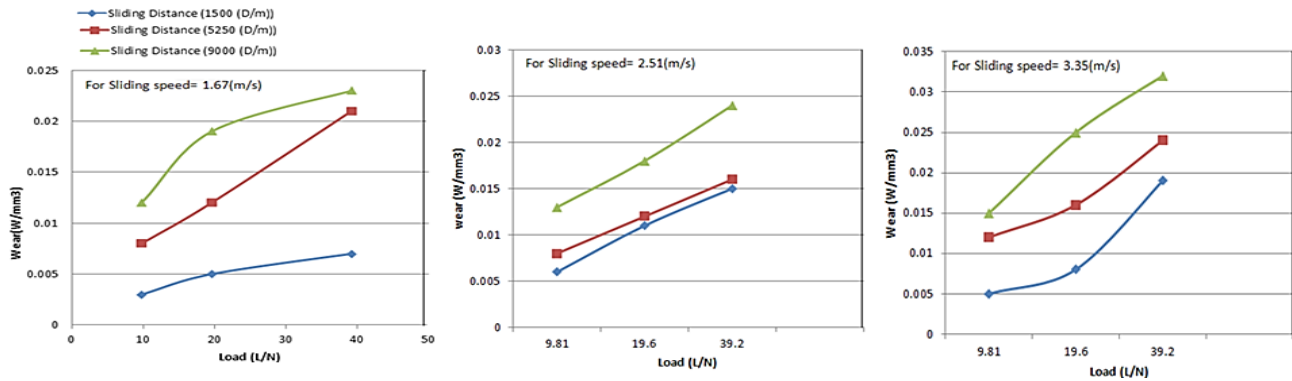


Figure 3.3 Variation of Wear with Load for different sliding distance and sliding speed

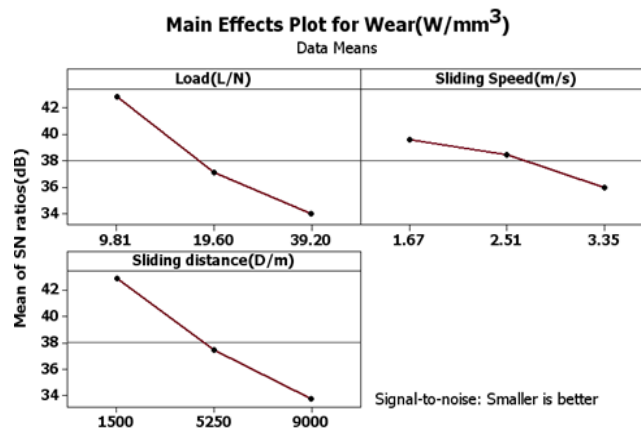


Figure 3.4 Mean S/N graph for wear (W/mm^3)

under different aging condition (a) Room Temperature quenching; (b) water quenching; (c) Cryogenic quenching; is shown in Figure 3.1.

From the main effects plot Figure 3.2 for micro hardness indicates the selection of higher aging time (90 min), higher aging temperature ($1000^{\circ}C$) and cryogenic quenching result the best combination to get higher micro hardness value under different age hardening conditions of Ti-6Al-4V alloy.

Table 3.1 shows the ranking of each age hardening parameter using the Response Table for Signal to Noise Ratios (larger is better) obtained for different parameter levels.

On the examination of the percentage of contribution (P%) of the different factors (Table 3.2), for micro hardness it can be seen that solution treatment has the highest contribution of about 82.33%, thus solution treatment is an important factor to be taken into consideration while age hardening Ti-6Al-4V alloy. It can be seen that Aging Temperature ($^{\circ}C$) (P=16.20%), Aging Time (min.) (P=1.47%).

3.2 Cryogenic quenched Ti-6Al-4V alloy wear measurement

Figure 3.3 shows experimental results wear rate behavior of the Ti-6Al-4V alloy under different wear conditions. Presence of less amount of wear is reported from the wear testing under 9.81 N load and 1500 (D/m) this is due to

protective oxide coating layer formed during cryogenic quenching of Ti-6Al-4V alloy and also due to acicular martensitic structure in its microstructure. The reduction of the shear strength of the material and increase true area of contact between contacting surfaces may also play role in higher wear rate for higher sliding speed. The variations in the wear under different Load (L/N), Sliding Speed (m/s) and Sliding Distance (D/m) were then analyzed using L27 orthogonal array to obtain optimum condition for wear and micro hardness.

From the main effects plot Figure 3.4 for wear indicates the selection of minimum Load (9.81N), lesser Sliding Distance (1500m) and lesser Sliding speed (1.67m/s) result the best combination to get lesser wear value for cryogenic quenched Ti-6Al-4V alloy.

Table 3.3 Response Table for Signal to Noise Ratios (Smaller is better)

Level	Load (L/N)	Sliding Speed (m/s)	Sliding Distance (D/m)
1	42.9	39.63	42.88
2	37.14	38.45	37.43
3	33.99	35.95	33.71
Delta	8.92	3.68	9.17
Rank	2	3	1

Table 3.3 shows the ranking of each wear parameter using the Response Table for Signal to Noise Ratios (smaller is

Table 3.4 Analysis of Variance for S/N ratios

Source	DF	Seq SS	Adj SS	Adj MS	F	P	Percent P (%)
(A)Load(L/N)	2	367.94	367.94	183.971	65.48	0.000	42.63
(B)Sliding Speed (m/s)	2	63.54	63.54	31.772	11.31	0.005	7.36
(C)Sliding Distance (D/m)	2	382.97	382.97	191.484	68.15	0.000	44.34
AXB	4	17.49	17.49	4.373	1.56	0.275	1.02
AXC	4	45.74	45.74	11.436	4.07	0.043	2.65
BXC	4	34.41	34.41	8.604	3.06	0.083	2.00
Residual Error	8	22.48	22.48	2.810			
Total	26	934.58					100

Table 3.5 Response Table for Signal to Noise Ratios (Larger is better)

Level	Load (L/N)	Sliding Speed (m/s)	Sliding Distance (D/m)
1	51.27	51.26	51.02
2	51.49	51.48	51.52
3	51.69	51.72	51.92
Delta	0.42	0.46	0.89
Rank	3	2	1

Table 3.5 Response Table for Signal to Noise Ratios (Larger is better)

Level	Load (L/N)	Sliding Speed (m/s)	Sliding Distance (D/m)
1	51.27	51.26	51.02
2	51.49	51.48	51.52
3	51.69	51.72	51.92
Delta	0.42	0.46	0.89
Rank	3	2	1

Table 3.6 Analysis of Variance for S/N ratios

Source	DF	Seq SS	Adj SS	Adj MS	F	P	Percent P (%)
(A)Load (L/N)	2	0.79717	0.79717	0.39859	61.29	0.000	14.65
(B)Sliding Speed (m/s)	2	0.95538	0.95538	0.47769	73.45	0.000	17.56
(C)Sliding Distance (D/m)	2	3.60553	3.60553	1.80276	277.20	0.000	66.29
AXB	4	0.04090	0.04090	0.01023	1.57	0.271	0.38
AXC	4	0.01262	0.01262	0.00315	0.49	0.747	0.12
BXC	4	0.10880	0.10880	0.02720	4.18	0.041	1.00
Residual Error	8	0.05203	0.05203	0.00650			
Total	26	5.57242					100

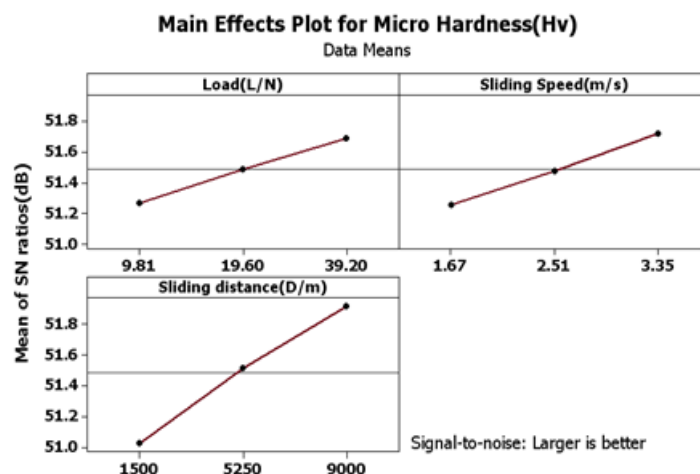


Figure 3.5 Mean S/N graph for Micro Hardness (HV)

better) obtained for different parameter levels. On the examination of the percentage of contribution (P%) of the different factors (Table 3.4), for wear it can be seen that sliding distance has the highest contribution of about 44.34%, thus sliding distance is an important factor to be taken into consideration. It can be seen that load (P=42.63%), sliding speed (P=7.36%), and interactions AxC (P=2.65%) have statistical and physical significance on wear. The interactions (AxB, BxC) neither present a statistical significance, nor a percentage of physical significance of contribution to the wear.

3.3 Cryogenic Quenched Ti-6Al-4V Alloy Micro Hardness Measurement of Worn Surface

From the main effects plot Figure 3.5 for micro hardness indicates the selection of high Load (39.2N), higher Sliding Distance (9000m) and higher sliding speed (3.35m/s) result the best combination to get higher micro hardness value for cryogenic quenched Ti-6Al-4V alloy due to the reason of work hardening taking place at very high Load, Sliding Distance and Sliding speed.

Table 3.5 shows the ranking of each micro hardness parameter using the Response Table for Signal to Noise Ratios (larger is better) obtained for different parameter levels.

On the examination of the percentage of contribution (P%) of the different factors (Table 3.6), for micro hardness it can be seen that sliding distance has the highest contribution of about 66.29%, thus sliding distance is an important factor to be taken into consideration. It can be seen that sliding speed (P=17.56%), load (P=14.65%). The interactions (AxB, AXC, BxC) neither present a statistical significance, nor a percentage of physical significance of contribution to the micro hardness.

4. Conclusions

The micro hardness were studied on Ti-6Al-4V alloy at age hardening temperature of 750°C, 825°C, 1000°C for aging time 30 min, 60 min and 90 min under room temperature, water and cryogenic quenching using L₉ orthogonal array based Taguchi's design of experiments. After getting the optimum Micro hardness based on Taguchi's design of experiments on age hardened Ti-6Al-4V alloy the specimen is carried out with wear and micro hardness of worn surface using pin on disc wear testing machine under different Load (L/N), Sliding Speed (m/s) and Sliding Distance (D/m) using L₂₇ orthogonal array based Taguchi's design of experiments. Based on the experimental and statistical study, the following conclusions can be drawn:

- The micro hardness was found to be higher for cryogenic quenching compared to room temperature quenching and water quenching.
- The wear rate was found to be minimum for lesser load, lesser sliding distance and lower sliding speed.
- From the main effects plot for micro hardness indicates the selection of higher aging time (90 min), higher aging temperature (1000°C) and cryogenic quenching result the best combination to get higher

micro hardness value under different age hardening conditions of Ti-6Al-4V alloy.

- On the examination of the percentage of contribution (P%) of the different factors for micro hardness it can be seen that solution treatment has the highest contribution of about 82.33%, thus solution treatment is an important factor to be taken into consideration while age hardening Ti-6Al-4V alloy. It can be seen that Aging Temperature (°C) (P=16.20%), Aging Time (min.) (P=1.47%).
- The micro hardness was found to be maximum for maximum load, higher sliding distance and higher sliding speed.
- The wear was found to be minimum for minimum load, minimum sliding distance and minimum sliding speed.
- On the examination of the percentage of contribution (P %) for micro hardness it can be seen that sliding distance has the highest contribution of about 66.29%.

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