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Research Article

Surface Treatment of Ti-6Al-4V by Nitriding Heat Treatment Process

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Abstract

Titanium and its alloys are widely used as implant material, especially in hard tissue replacement for orthopedic and dental implant applications. However, its poor wear resistance requires proper surface hardening. Body implants are also subjected to loads at varying strain rates. Nitriding is one is method of increasing surface hardness. The samples were heat treated in a muffle furnace in the presence of potassium nitrate. Heat treatment was done in nine groups at the temperatures of 600, 650 and 700 ^{0}C during 2, 4, and 6 hr and then cooled in furnace for slow cooling. The highest hardness i.e. 438 is noted at 700 ^{0}C temperature, and 6 hr holding time. The mean of hardness is increased by increasing heating temperatures and increasing the holding time.

Keywords: *Ti-6Al-4V, Heat treatment, Nitriding, Furnace Cooling*

1. Introduction

The interaction between living tissue environment and implant completely depends on the surface properties of material. The property of implant to interact with tissue is termed as bioactivity. Bioactivity is defined as: A bioactive material is one that elicits a specific biological response at the interface of the material which results in the formation of a bond between the tissues and the material. Thus an implant material for hard tissue replacement should have appropriate mechanical properties with bioactive surface. Titanium and its alloys are widely used as implant material, especially in hard tissue replacement for orthopedic and dental implant applications because of its high strength-to-weight ratio and excellent biocompatibility. However, its poor wear resistance requires proper surface hardening. In some industrial applications, the tribological properties of the alloy surface become important; in such cases, the alloy surface is subjected to wear. Therefore, the use of the alloy in such situations becomes limited due to the poor tribological properties of the surface.

Nitriding of titanium and titanium alloys has been investigated for many years and is used effectively for protection against wear. Nitrogen has a high solubility in Ti so it strengthens the surface layer significantly. **B.S. Yilbas et al**. **(2006)** carried out laser gas assisted nitriding of Ti–6Al–4V alloy and formed nitride compounds and examined their concentration in the surface vicinity. They found that the nitride layer appears like golden color; however, it becomes dark gold color once the laser power irradiation is increased. **M. Karthega, and N. Rajendran (2010)** treated Ti–6Al–4V alloy with various concentrations (5 wt.%, 15 wt.% and 25 wt.%) of hydrogen peroxide (H_2O_2) and then heat treated to produce an anatase titania layer. The results revealed that titania layer with anatase nature was observed for all H_2O_2 treated Ti–6Al–4V alloy, irrespective of the H_2O_2 concentrations.

Dong-Bo Wei et al. (2012) prepared NiCrAlY coating by multi-arc ion plating. The results show that the chromising coating consisted of an outer layer of loose Cr deposition, an intermediate layer of compact Cr deposition and an inner Ti–Cr mutual diffusion layer. The multilayer oxide scales formed in the oxidation process, which has the better cyclic oxidation resistance compared with NiCrAlY thermal barrier coating. **L. Ge et al. (2013)** performed supersonic fine particle bombarding (SFPB) on Ti-6Al-4V alloy to form surface nanostructures. The effect of the surface nanostructures on the gas nitriding was investigated by varying the nitriding temperature and time. In comparison to the surface without SFPB treatment, the treated surface exhibited a thick nitrogen diffusion layer and high hardness under the same nitriding process.

Kaixuan Gu et al. (2014) investigated the effect of cryogenic treatment on wear resistance of Ti–6Al–4V alloy for biomedical applications by experimentally. Cryogenic treatments with the same soaking time of 24h at different temperatures of -80 ^{0}C, -140 ^{0}C and -196 ^{0}C were conducted and the treatments at the same temperature of -196 ^{0}C were then further given different soaking time of 3h, 48h and 72h to be investigated. By cryogenic treatment, the plowing in the worn surface was smoothed and shallowed, and the degree of plastic deformation in the subsurface was decreased. **S. Maya-Johnson, and D. Lopez (2014)** discussed the effect of the cooling rate during a forging process on the microstructure and corrosion behavior of a Ti–6Al–4V extra-low interstitial (ELI) alloy, which is commonly used as biomaterial. It

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was found that the clay as a cooling medium is a good candidate to obtain a proper microstructure and properties for biomedical applications, eliminating the requirement of subsequent heat treatment and reducing costs.

2. Experimental Procedure

Cold-rolled cylindrical plate of Ti-6Al-4V with a size of 50 mm diameter and 6 mm thickness was used in this study. The chemical compositions of Ti-6Al-4V are listed in Table 1.

Table 1: Chemical compositions of Ti-6Al-4V (Mass %)

Element	%
Al	6.12
Fe	0.13
V	4.23
C	0.04
O	0.12
H	0.014
Ti	Remaining

Heat Treatment

The samples were heat treated in a muffle furnace in the presence of potassium nitrate. Heat treatment was done in nine groups at the temperatures of 600, 650 and 700 ^{0}C during 2, 4, and 6 hr and then cooled in furnace for slow cooling. During heat treatment, the temperature was gradually increased from room temperature, about 15°C/minute to final temperature and hold final temperature for different period of time and then samples were furnace cooling.

Fig 1: Specimens for Experimentations

The figure 1 shows the specimens of Ti-6Al-4V used for experimentations. These specimens prepared by cutting on power hacksaw, grinding and by polishing. These specimens were dipped in potassium nitrate in a hardened mild steel container for nitriding of Ti-6Al-4V alloy. Then this container was placed in muffle furnace for nitriding as shown in figure 2.

Fig 2: Container of Potassium Nitrate in Muffle Furnace

Maintain the temperature of furnace for 2, 4, and 6 hr period of time. Firstly one specimen was dipped in container for a period of 2 hr, after 2 hr another one piece was put in container for next 2 hr period of time means total period of time 4 hr. Then after total 4 hr, put another one piece in furnace for another period of next 2 hr. Its means first specimens had total heating time 6 hr, second specimens had total heating time 4hrs and third specimens had total time 2 hr.

Fig 3: Slow Cooling of Specimens in Furnace

After completion of 6 hr, the container is bringing out from the muffle furnace. The specimens are removing from container and placed in furnace for slow cooling or furnace cooling.

Fig 4: Heat treated Specimens

The heat treated Ti-6Al-4V alloy in potassium nitrate at 600 ^{0}C, 650 ^{0}C, and 700 ^{0}C for period of 2, 4, and 6hr allowed cooling down in furnace for slow cooling. The microstructure of the top surface of the specimens heat treated at 700 ^{0}C during continues heating for period of 6 hr received an unintentional overshoot above the β transus temperature during the equilibrium stage resulting in an all β phase. Hence hardness is higher at this sample.

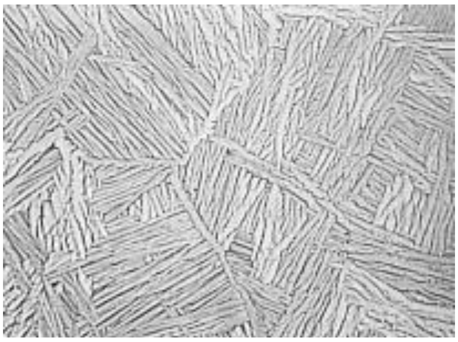


Fig 5: Micro surface structure of Ti-6Al-4V at 700 ^{0}C and 6 hr

3. Results and discussions

After all the measurements of hardness, it is required to study the effect of different heat treated parameters for hardness of Ti-6Al-4V alloy.

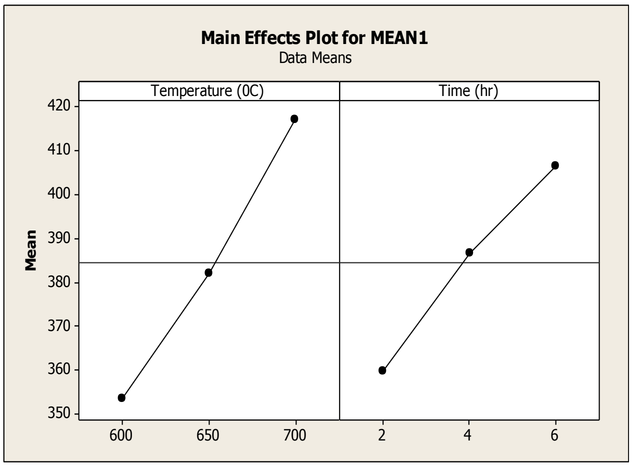
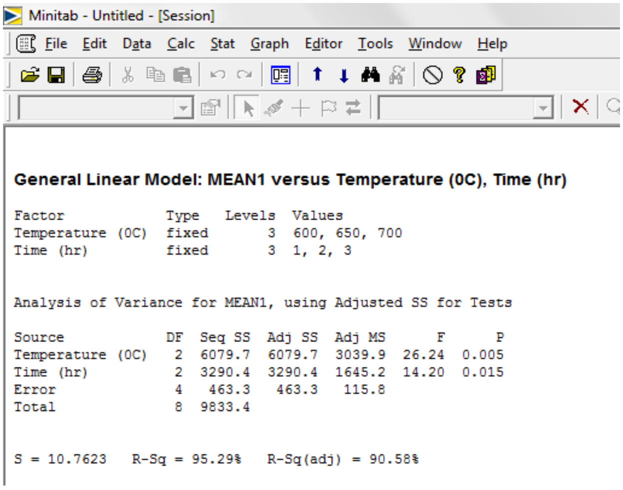


Fig 6: Mean value Graph for hardness

Figure 6 shows effect of various heat treatment parameters on the mean of hardness plotted utilizing the nitriding results obtained of Ti-6Al-4V. From the figure, it is observed that the mean value of hardness is increase by increasing the heating temperature from 600 to 650 ^{0}C and from 650 to 700 ^{0}C. The mean of hardness rises constantly by increasing the value of holding time from 2 to 4 hr and from 4 to 6 hr.

Analysis of Variance

The results were analyzed using ANOVA for identifying the significant factors affecting the performance measures. The Analysis of Variance (ANOVA) for the mean hardness at 95% confidence interval is given in below figure. The variation data for each factor and their interactions were F-tested to find significance of each calculated value. The principle of the F-test is that the larger the F value for a particular parameter, the greater the effect on the performance characteristic due to the change in that process parameter. ANOVA table shows that Temperature (F 26.24 value), and Time (F 14.20 value) are the factors that significantly affect the Hardness. Temperature has highest contribution to hardness.



General Linear Model: MEAN1 versus Temperature (0C), Time (hr)

Factor	Type	Levels	Values
Temperature (0C)	fixed	3	600, 650, 700
Time (hr)	fixed	3	1, 2, 3

Analysis of Variance for MEAN1, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Temperature (0C)	2	6079.7	6079.7	3039.9	26.24	0.005
Time (hr)	2	3290.4	3290.4	1645.2	14.20	0.015
Error	4	463.3	463.3	115.8		
Total	8	9833.4				

S = 10.7623 R-Sq = 95.29% R-Sq(adj) = 90.58%

Fig 7: ANOVA for Hardness

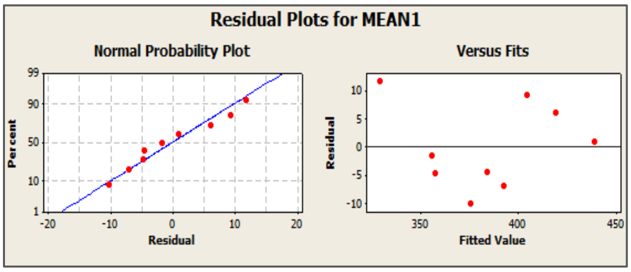


Fig 8: Residual Plots for MRR

The residual plots for hardness are presented in Fig. 8. It can be noticed that the residuals fall on a straight line, which means that the errors are normally distributed and the regression model is well fitted with the observed values. The Fitted value indicates that the maximum variation of -10 to 10, which shows the high correlation that, exists between fitted values and observed values.

Conclusions

Based on the experimental results and discussions the following conclusions are drawn for heat treatment of Ti-6Al-4V by nitriding as listed below:

- The highest hardness i.e. 438 is noted at 700^{0}C temperature, and 6 hr holding time.
- The mean of hardness is increased by increasing heating temperatures.
- The mean of hardness is increased by increasing the holding time.
- For higher hardness, the optimum parameters for nitriding are 700^{0}C temperatures and 6 hr holding time.

- The microstructure of the top surface of the specimens heat treated at 700 ^{0}C during continues heating for period of 6 hr received an unintentional overshoot above the β transus temperature during the equilibrium stage resulting in an all β phase. Hence hardness is higher at this sample.

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