TRIBOLOGICAL BEHAVIOR OF Al_2O_3-TiO_2 COATED Ti6Al4V IMPLANT ALLOY IN SIMULATED BODY ENVIRONMENT

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***Abstract :** Ti6Al4V alloys are extensively used in medical applications due to their superior mechanical properties. But, Ti6Al4V alloys are poor in tribological properties such as wear and corrosion resistance. Poor wear resistance leads to formation of wear debris in human implants and causes inflammation. In this work, Al_2O_3-TiO_2 coating is applied to improve the wear and corrosion resistance. Al_2O_3-TiO_2 coating was deposited on the substrate with 100µm, 200µm , 300µm , 400µm thickness using detonation spray(DS). Implants are lies inside the human body; Hence wear, surface roughness and corrosion behavior was studied in the simulated body environment using Hank's solution. ASTM G-99 standard specimens are used for pin on disc wear tests. Experiments are carried out using Taguchi design. Grey relational analysis and response surface method are used for obtaining optimal experimental parameters. Improvement was observed in wear resistance for the Al_2O_3-TiO_2 coated Ti6Al4V alloy.*

***Keywords :** Detonation spray, Hank's solution, Wear, Corrosion, Taguchi Method, Grey relational analysis*

I. INTRODUCTION

Bio-compatibility and mechanical properties makes Ti6Al4V alloy appropriate for orthopedic implant applications[1]. Life of the implants materials depends on the wear resistance. Generally wear property can be defined as source of damage to a solid surface by progressive loss of material, due to relative motion between contacting surfaces[2-3]. Detonation spray(DS) coatings are often deposited on metals to improve tribological and corrosion properties. In this technique melted or semi-melted state powder by the heat due to combustion of fuel in presence of oxygen was expel on the surface of work piece at a high speed[4-5] and gives an extremely good adhesive strength and low porosity.

The present research is carried out with the aim of determining the Tribological and corrosion properties of Al2O3-TiO2 coated[6-10] Ti6Al4V implant alloy. Detonation spray technique is used to deposit the coating and thickness is varied as 100µm, 200µm , 300µm , 400µm to study the effect of coating thickness on Wear and corrosion resistance. Wear tests are performed for different loads, speeds, sliding distances and coating thickness using pin-on-disc apparatus[11-13]. Corrosion is measured using Potentiodynamic polarization test [14-15].

Taguchi technique for design of experiments (DOE) has been successfully used by researchers [16-17]. The DOE process consists of three main phases: the planning phase, the conducting phase, and the analysis phase. Major step in the DOE process is the determination of the combination of factors and levels which will provide the desired information. Analysis of the experimental results uses the signal to noise ratio to obtain the best process designs. The major aim of the present investigation is to analyse the influence of parameters like load, sliding speed, sliding distance and coating thickness on sliding wear of Ti6Al4V coated with Al2O3-TiO2. Grey relational analysis is used to convert multi response problem involving wear and surface roughness into single response problem to apply the Taguchi method[18-20].

1.1 Selection of orthogonal array

Most important step in the DOE process is the selection of orthogonal array based on number of factors and number of levels for each factor. The degrees of freedom for the orthogonal array should be greater than or at least equal to those for the process parameters. In present work four factors and four levels were considered. Therefore L_{16} orthogonal array was adopted for design of experiments.

1.2 Analysis of the Signal-to-Noise Ratio

Best process parameters can be obtained by the analysis of the experimental results using signal to noise ratio. Taguchi technique is a powerful design of experiment tool for acquiring the data in a controlled way and to analyze the influence of process variable over some specific variable which is unknown function of these process variables and for the design of high quality systems. Taguchi creates a standard orthogonal array to accommodate the effect of several factors on the target value and defines the plan of experiment. Signal-to-noise ratio for smaller is the better characteristics given by Taguchi which can be calculated as logarithmic transformation of the loss function, is given as :

$$S/N = -10 \times log\left(\frac{\sum(Y^2)}{n}\right)$$

Where y is the observed data and n is the number of observations.
The aim of the present work is to find the influence of experimental parameters like load, speed, distance and thickness to minimize the sliding wear behavior of Al_2O_3-TiO_2 coated Ti6Al4V in simulated body environment by employing the Taguchi's orthogonal array and Grey relational analysis.

2. EXPERIMENTAL WORK

2.1 Detonation spray

Oxygen and acetylene combustion mixture is fed through a tubular barrel from one end. A blanket of nitrogen gas can cover the gas inlets to prevent the possibility back firing. At the same time, fixed quantity of the coating powder is send into the combustion chamber. The Gas mixture inside the chamber is ignited by spark plug. The combustion of the gas mixture creates high pressure detonation wave, which then transmit through the gas flow. Depending upon the ratio of the combustion gases, the temperature of the hot gas stream can go up to 4000^0C and the velocity of the shock wave can reach up to 3500m/sec. The hot gases generated in the detonation chamber travel down the barrel at a high velocity and in the process heat the particles to a plasticizing stage and also accelerate the particles to a velocity of 1200m/sec. These particles then come out of the barrel and impact the component to form a coating. The high kinetic energy of the hot powder particles on impact with the substrate result in a buildup of a very dense and strong coating [10].

2.2 Materials and coating deposition

Ti6Al4V was used as substrate and its chemical composition is given in Table 2.1. Al_2O_3-TiO_2 was used as coating material whose chemical composition is given in Table 2.2. In the present work, coatings are performed by 100µm, 200µm , 300µm , 400µm thick using detonation spray technique. Prior to coating, Substrate was blasted with Al_2O_3 grits. Optimum surface roughness was obtained through Grid blasting for the best adhesion between coating and substrate. Fig.2.1 and Fig.2.2 shows the detonation spray and Grid blasting equipment used in the present work. The spraying process parameters for DS are listed in Table 2.3.

Table 2.1 Chemical composition (Weight %) of Ti6Al4V

Ti	Al	V	Fe	Cr	Mo
Bal	6.53	3.85	0.08	0.01	0.03

Table 2.2 Chemical composition(Weight %) of Cr_3C_2-NiCr

Al_2O_3	TiO_2
87	13

Table 2.3 DS parameters for Cr_3C_2-NiCr deposition

Oxygen flow rate(slph)	850
Acetelene Fuel(slph)	2440
Nitrogen flow rate(slph)	12
Spray distance	120mm
Gun speed	10mm/sec

Fig.2.1 Detonation Spray Process

Fig.2.2 Grid blasting

2.3 Hank solution

Simulated body fluid environment was created using Hank's solution. It was prepared using high purity reagents; their chemical composition is given in Table 2.4.

Table 2.4 Hank solution chemical composition

Component	Nacl	KCL	NaHCO3	CaCl2	MgCl2.6H2O
(g/L)	8	0.4	0.35	0.14	0.1
Component	Na2HPO42 H2O	KH2PO4	MgSO4.7H2O	Glucose	pH
(g/L)	0.06	0.06	0.06	1	6.8

2.4 Surface roughness measurements

Coating material surface roughness before and after the wear test were measured by using Talysurf instrument shown in Fig.2.3. An average of five readings is reported for all experiments.

Fig.2.3 Talysurf Surface roughness instrument

2.5 Wear testing

Pin-on-disc wear testing machine is used to study the sliding wear behavior. Ti6Al4V cylindrical pins of 6mm diameter and 30mm length with Al_2O_3-TiO_2 coating are used as test material. Levels for experimental parameters such as load, speed, distance and thickness are as shown in Table 2.5. Specimens are prepared as per ASTM G-99 standards as shown in Fig.2.4. Mass of each specimen was measured with an accuracy of ±0.0001g and the average of three readings was recorded. Pin on disc experimental set up was shown in Fig.2.5. Experiments are carried out as per Taguchi's design in the simulated body environment created using Hank's solution. Wear of the material is studied by considering the mass loss during the wear test.

Fig.2.4 Al_2O_3-TiO_2 Coated Ti6Al4V Specimens

Fig.2.5 Pin on Disc Wear Testing Machine

Table 2.5 Parameter for wear test

Factors	**Levels**			
	1	**2**	**3**	**4**
Load in N	10	30	40	50
Speed in m/s	0.6	0.9	1.2	1.5
Distance Km	0.25	0.5	0.75	1
Coating Thickness in µm	100	200	300	400

3. RESULTS AND DISCUSSION

3.1 Sliding wear behavior of Al_2O_3-TiO_2 coated Ti6Al4V in simulated body environment (Hank's solution)

Taguchi design of experiments and response of sliding wear behavior of Al_2O_3-TiO_2 coated Ti6Al4V in simulated body environment were shown in Table 3.1. Mass loss and surface roughness are considered as response parameters during the wear test. Grey relational grey relational grade and S/N ratio's were obtained.

Table 3.1 Taguchi design of experiments for sliding wear of Al_2O_3-TiO_2 coated Ti6Al4V in simulated body environment

E.No.	Load, N	Speed, m/sec	Distance, Km	Thickness, µm	Mass loss, mg	Surface Roughness, µm	Grey Relational grade	S/N Ratio of Grey Relational grade
1	10	0.6	0.25	100	0.88	3.78		
2	10	0.9	0.5	200	0.99	3.78	0.7061	-3.02267
3	10	1.2	0.75	300	0.94	3.83	0.6347	-3.9486
4	10	1.5	1	400	1.25	3.38	0.63745	-3.9110
5	30	0.6	0.5	300	1.53	2.99	0.57665	-4.7817
6	30	0.9	0.25	400	1.48	3.14	0.59015	-4.5807
7	30	1.2	1	100	1.76	4.07	0.5618	-5.0083
8	30	1.5	0.75	200	1.89	3.47	0.39335	-8.1044
9	40	0.6	0.75	400	1.82	2.93	0.4398	-7.1348
10	40	0.9	1	300	1.93	3.43	0.56105	-5.0199
11	40	1.2	0.25	200	1.82	3.81	0.44105	-7.1102
12	40	1.5	0.5	100	2.01	4.03	0.4083	-7.7804
13	50	0.6	1	200	2.05	2.59	0.3665	-8.7185
14	50	0.9	0.75	100	2.18	4.02	0.67855	-3.3683
15	50	1.2	0.5	400	1.95	3.26	0.3512	-9.0889
16	50	1.5	0.25	300	2.03	4.26	0.46635	-6.6257

Main effects plot representing the combined optimum factors of mass loss and surface roughness was shown in Fig. 3.1. From the main the effects plot optimum levels of load, speed, sliding distance and thickness were shown in Table 3.2

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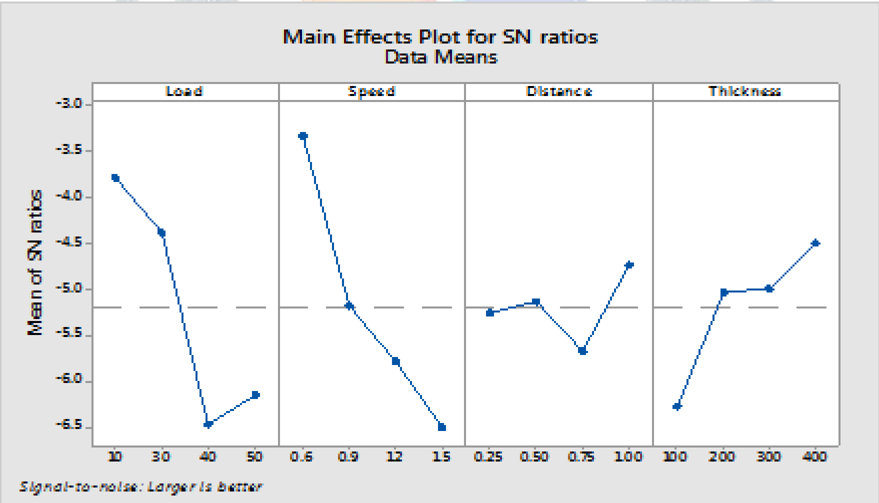

Fig. 3.1 Main effects plot for sliding wear of Al_2O_3-TiO_2 coated Ti6Al4V in simulated body environment

Table 3.1 Optimum levels for sliding wear of Al_2O_3-TiO_2 coated Ti6Al4V in simulated body environment

S. No.	Experiment Factors	Optimum level
1	Load	10N
2	Speed	0.6m/sec
3	Sliding distance	1Km
4	Coating Thickness	400µm

3.2 Response surface method - Sliding wear test of Al_2O_3-TiO_2 coated Ti6Al4V in simulated body environment

Mathematical models for the mass loss and surface roughness have been developed using response surface method (Equation-1 and 2) to study the sliding wear behavior in simulated body environment. Experimental values, predicted values and % error are presented in Table 3.2 for mass loss and surface roughness values are shown in Table 3.3.

$$Mass\ loss = 1.5 - (0.045 \times Load) - (0.779 \times Speed) - (6.9 \times Distance) + (0.0171 \times \text{Thickness}) + (0.00143 \times \text{Load} \times \text{Load}) + (0.285 \times Speed \times Speed) + (2.04 \times Distance \times Distance) - (0.000015 \times \text{Thickness} \times \text{Thickness}) + (0.0021 \times Load \times Speed) + (0.0067 \times Load \times Distance) - (0.000019 \times Load \times \text{Thickness}) + (4.16 \times Speed \times Distance) + (0.0089 \times Speed \times Thickness) \quad \ldots..(1)$$

$$Ra = 5.43 - (0.275 \times Load) + (0.37 \times Speed) - (12.3 \times Distance) + (0.037 \times \text{Thickness}) + (0.0044 \times \text{Load} \times \text{Load}) - (1.323 \times Speed \times Speed) + (5.3 \times Distance \times Distance) - (0.000026 \times \text{Thickness} \times \text{Thickness}) + (0.0921 \times Load \times Speed) - (0.0562 \times Load \times Distance) - (0.000100 \times Load \times \text{Thickness}) + (7.7 \times Speed \times Distance) - (0.0213 \times Speed \times Thickness) \quad \ldots..(2)$$

Table 3.2 Experimental, Predicted values of mass loss and percentage error for sliding wear of Al_2O_3-TiO_2 coated Ti6AL4V in simulated body environment

Expt No.	Load N	Speed m/sec	Distance Km	Thickness µm	Mass loss gms	Predicted Mass loss gms	% Error
1	10	0.6	0.25	100	0.88	0.89105	-1.25568
2	10	0.9	0.5	200	0.99	0.88715	10.38888
3	10	1.2	0.75	300	0.94	0.97955	-4.20744
4	10	1.5	1	400	1.25	1.16825	6.54
5	30	0.6	0.5	300	1.53	1.5255	0.294117
6	30	0.9	0.25	400	1.48	1.4202	4.040540
7	30	1.2	1	100	1.76	1.7562	0.215909
8	30	1.5	0.75	200	1.89	1.8435	2.46031
9	40	0.6	0.75	400	1.82	1.7191	5.543956
10	40	0.9	1	300	1.93	1.89435	1.847150
11	40	1.2	0.25	200	1.82	1.8139	0.335164
12	40	1.5	0.5	100	2.01	2.04975	-1.97761
13	50	0.6	1	200	2.05	2.0562	-0.30243
14	50	0.9	0.75	100	2.18	2.145	1.605504
15	50	1.2	0.5	400	1.95	1.9381	0.61025
16	50	1.5	0.25	300	2.03	1.9915	1.896551

Table 3.3 Experimental, Predicted values of surface roughness and percentage error for sliding wear of Al_2O_3-TiO_2 coated Ti6Al4V in simulated body environment

E. No.	Load, N	Speed, m/sec	Distance, Km	Thickness, µm	Surface roughness, µm	Predicted Surface roughness, µm	% Error
1	10	0.6	0.25	100	3.789	3.75107	1.0010
2	10	0.9	0.5	200	3.78	3.89527	-3.0494
3	10	1.2	0.75	300	3.836	3.82083	0.3954
4	10	1.5	1	400	3.381	3.52775	-4.3404
5	30	0.6	0.5	300	2.99	3.21152	-7.40865
6	30	0.9	0.25	400	3.146	3.22732	-2.58488
7	30	1.2	1	100	4.071	4.13248	-1.51016
8	30	1.5	0.75	200	3.474	3.387	2.5043
9	40	0.6	0.75	400	2.933	2.88937	1.4874
10	40	0.9	1	300	3.434	3.53797	-3.0276
11	40	1.2	0.25	200	3.811	3.88193	-1.8611

12	40	1.5	0.5	100	4.034	4.24525	-5.2367
13	50	0.6	1	200	2.597	2.80272	-7.9214
14	50	0.9	0.75	100	4.026	3.95512	1.7605
15	50	1.2	0.5	400	3.268	3.55088	-8.6560
16	50	1.5	0.25	300	4.269	4.282	-0.3045

3.3 Wear tracks of Al_2O_3-TiO_2 coating for sliding wear test in simulated body environment

Wear tracks of the substrate and Al_2O_3-TiO_2 coated specimens in simulated body environment (Hank's solution) at optimum experimental parameters are shown in Fig.3.1 and Fig.3.3 respectively. Fig.3.2 shows severe debri formation and wear tracks which attributes to maximum mass loss on substrate where as Fig.3.3 shows few tracks on Al_2O_3-TiO_2 coated specimen which refers to minimum mass loss.

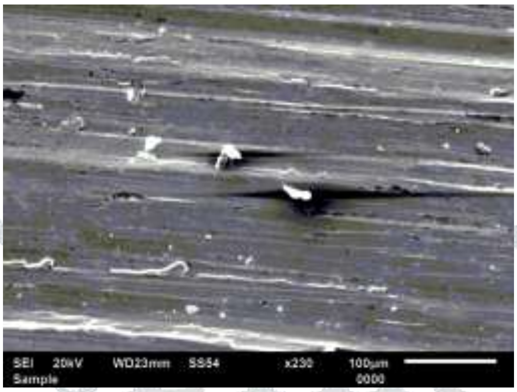

Fig. 3.2 Wear tracks on Ti6Al4V substrate

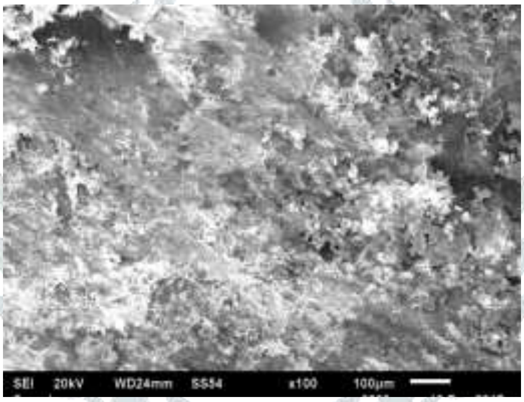

Fig.3.3 Wear tracks on Al_2O_3-TiO_2 coated specimens at optimum parameters

3.4 Confirmation test of sliding wear in simulated body environment for Al_2O_3-TiO_2 coated Ti6Al4V

Confirmation test was carried out at the optimum parameters. It is observed that, mass loss in experimental and predicted are 1.103mg and 0.771mg respectively. Experimental, predicted surface roughnesses are 4.724µm and 5.604µm respectively. Mass loss and surface roughness of the Ti6Al4V substrate are 60.13mg and 3.38µm respectively. Wear test results shows that wear resistance of Al_2O_3-TiO_2 coated Ti6Al4V is high due to less mass loss. Surface roughness is observed to be high for Al_2O_3-TiO_2 coated Ti6Al4V due to formation of wear debris.

4. CONCLUSIONS

1. Optimum experimental parameters obtained with Grey relational analysis for sliding wear test of Al_2O_3-TiO_2 coated Ti6Al4V in simulated body environment are load : 10N, speed : 0.6m/sec, sliding distance : 1Km, Coating thickness : 400µm.
2. Response surface mathematical models are obtained for mass loss and surface roughness.
3. Experimental and predicted values of mass loss (1.10mg and 0.77mg) and surface roughness (4.72µm and 5.60µm) are good in agreement.

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