Effect of Hank's solution on sliding wear behaviour of Cr_3C_2-NiCr coated Ti6Al4V alloy

M. Raja Roy [#1], N. Ramanaiah [*2], B. S. K. Sundara Siva Rao [#3]

[#1]*Sr.Assistant Professor, Department of Mechanical Engineering, ANITS,Visakhapatnam,Andhra Pradesh,India*

[*2]*Professor, Department of Mechanical Engineering, AUCOE(A)Visakhapatnam, Andhra Pradesh, India*

[3]*Former Professor, Department of Mechanical Engineering,AUCOE(A)Visakhapatnam, Andhra Pradesh, India*

Abstract — *Ti6Al4V alloys are widely used in medical applications due to their bio compatibility and high strength. But, Ti6Al4V alloys are poor in wear. Poor wear resistance results in formation of wear debris in implants and causing pain and inflammation. In this work, Cr_3C_2-NiCr coatings were applied to improve the hardness and wear resistance. These are deposited on the substrate (Ti6Al4V) with 100µm, 200µm, 300µm and 400µm thickness using detonation spray(DS). Pin on disc wear tests have been carried out on these materials in simulated body environment (Hank's solution) with ASTM G-99 standard specimens. Improvement was observed in hardness and wear resistance when compared to substrate. Wear behaviour of the Cr_3C_2-NiCr coated Ti6Al4V alloy was studied using Taguchi design of experiments. Wear resistance improved and it was better in simulated body environment.*

Keywords — *Detonation Spray, Ti6Al4V, Surface Coatings, Wear, Hank's solution, Taguchi's orthogonal array, ANOVA.*

I. Introduction

Mechanical properties and bio-chemical compatibility makes Ti6Al4V alloy suitable for orthopedic implant applications [1]. Implants are subjected to action of sliding and rubbing contact of articulated surfaces during their service in body. Generally wear property can be defined as a source of damage to solid surface by progressive loss of material, due to relative motion between that surface and a contacting surface[2]. The property of poor wear resistance generates wear debris, when the artificial implant is in contact with the healthy and natural joint, the accumulated wear debris causes inflammation, pain and finally loosening of the joint[3]. Hank's solution [18] was used to conduct the wear test under simulated body environment.

Thermal barrier coatings are often deposited on metals to improve mechanical and tribological properties. Detonation spray(DS) is a thermal barrier coating technology expelling the melting or semi-melting state powder heated by the combustion of fuel in presence of oxygen to the surface of work piece at a high speed, which has been extensively used in many fields, such as aviation, space flight, petroleum, metallurgy and other chemical and machinery industries[4].This method gives an extremely good adhesive strength, low porosity and coating surfaces with compressive residual stresses.

The present research is carried out with the aim of determining the wear behavior of Cr_3C_2-NiCr coated [6] Ti6Al4V implant alloy. Detonation spray technique was used to deposit the coating and thickness was varied as 100µm, 200µm, 300µm and 400µm respectively to study the effect of coating thickness on wear resistance. Hardness of both substrates and coated specimens were found by conducting hardness test. Wear test was performed for different loads, speeds, sliding distances and coating thickness using pin-on-disc apparatus. Hank's solution was used for simulating body[19] fluid environment and a comparison is also made between sliding wear behaviour of Substrate and Coated material in wet condition.

The design of experiments (DOE) approach using Taguchi technique has been successfully used by researchers in the study of wear behavior[16]. The DOE process is made up of three main phases: the planning phase, the conducting phase, and the analysis phase. A 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 a signal to noise ratio to aid in the determination of the best process designs. The 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. The experimental results are analyzed using analysis of means and variance to study the influence of parameters [16-17]. A multiple linear regression model is developed to predict the wear rate of Ti6Al4V. 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 Cr3C2-NiCr using Taguchi technique.

II. EXPERIMENTAL WORK

A. Detonation Spray

Precisely measured quantity of the combustion mixture consisting of oxygen and acetylene is fed through a tubular barrel closed at one end. In order to prevent the possible back firing a blanket of nitrogen gas is allowed to cover the gas inlets. Simultaneously, a predetermined quantity of the coating powder is fed into the combustion chamber. The gas mixture inside the chamber is ignited by a simple spark plug. The combustion of the gas mixture generates high pressure detonation wave, which then propagate through the gas stream. Depending upon the ratio of the combustion gases, the temperature of the hot gas stream can go up to 4000^{0}C and the velocity of the shock wave can reach 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 held by the manipulator to form a coating. The high kinetic energy of the hot powder particles on impact with the substrate result in a build up of a very dense and strong coating [10-11].

B. Material and coating deposition

Ti6Al4V was used as substrate and its chemical composition is given in Table-I. Cr_3C_2-NiCr was used as coating material whose chemical composition is given in Table-II. In the present work, coatings are performed by 100µm, 200µm , 300µm , 400µm thick using detonation spray technique. Prior to coating, Optimum surface roughness was obtained through Grid blasting with Al_2O_3 grits for the best adhesion between coating and substrate. Figure-1 and Figure-2 shows the detonation spray and Grid blasting equipment used in the present work. The spraying process parameters for DS are listed in Table-III.

TABLE I
Chemical composition(Weight %) of Ti6Al4V

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

TABLE II
Chemical composition(Weight %) of Cr_3C_2-NiCr

Cr_3C_2	NiCr
75	25

TABLE III
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 1: Detonation Spray Process

Fig 2: Grid blasting

C. Hank solution

Hank's solution was prepared using high purity reagents. The chemical composition of the Hank's solution [20] is shown in Table-IV.

Table IV
Hank solution chemical composition

Component	(g/L)	Component	(g/L)
Nacl	8	Na_2HPO_4. $2H_2O$	0.06
KCL	0.4	KH_2PO_4	0.06
$NaHCO_3$	0.35	$MgSO_4.7H_2O$	0.06
$CaCl_2$	0.14	Glucose	1
$MgCl_2.6H_2O$	0.1	pH	6.8

D. Hardness

Hardness of substrate and coated material were found by IS 1586 test procedure as per BIS standards using Rockwell hardness tester. An average of five readings is reported.

E. Wear Test

The sliding wear tests were conducted on a pin on disc wear testing machine using Hank's solution according to the ASTM - G99 standards. Cylindrical specimens of size ϕ3mm and 30mm length made with substrate and coated material was used as test material. Chrome steel was used as counter face material. Figure-3 shows the pin on disc wear testing machine and Figure-4 Shows the specimens prepared for wear test.

Fig 3: Pin on Disc Wear Testing Machine

Fig 4: Cr_3C_2 - NiCr Coated Ti6Al4V Specimens

The wear test were carried out by taking the taguchi design of experiments by considering the load, speed, distance and coating thickness as factors and each factor is taken to four levels as shown in Table-V and Table-VI. Weight loss of the specimens was measured by using a balance with an accuracy of ±0.0001g.

Table V

Parameters 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 in Km	0.25	0.5	0.75	1
Coating Thickness in µm	100	200	300	40 0

Table VI

Taguchi Design of Experiments for wear test

Expt No.	**Load in N**	**Speed in m/sec**	**Distance in Km**	**Thickness in µm**
1	10	0.6	0.25	100
2	10	0.9	0.5	200
3	10	1.2	0.75	300
4	10	1.5	1	400
5	30	0.6	0.5	300
6	30	0.9	0.25	400
7	30	1.2	1	100
8	30	1.5	0.75	200
9	40	0.6	0.75	400
10	40	0.9	1	300
11	40	1.2	0.25	200
12	40	1.5	0.5	100
13	50	0.6	1	200
14	50	0.9	0.75	100
15	50	1.2	0.5	400
16	50	1.5	0.25	300

III. RESULTS AND DISCUSSION

A. Hardness

Rockwell hardness (HRC) values for substrate and coated material were found by IS1586 test procedure as per BIS standards and average of five readings is reported in Table-VII. Significant improvement in hardness is achieved through coating from 100µm to 400µm thickness.

Table VII

Rockwell hardness (HRC) values for substrate and coated material

Material	**Coating Thickness (Microns)**	**Rockwell Hardness (HRC)**
Ti6Al4V	Base metal	25.67
Cr_3C_2 - NiCr Coated Ti6Al4V	100	35.33
Cr_3C_2 - NiCr Coated Ti6Al4V	200	41.33
Cr_3C_2 - NiCr Coated Ti6Al4V	300	49.67
Cr_3C_2 - NiCr Coated Ti6Al4V	400	53.33

B. Wear Analysis

Wear test was carried out for the Ti6Al4V substrate using Pin-on disc wear testing machine and Percentage of weight loss was obtained as 9.77. Effect of Cr_3C_2 - NiCr coating on Ti6Al4V substrate was studying by varying thickness (100µm, 200µm, 300µm, 400µm) using Taguchi design of experiments. MINITAB software was used for analyzing Taguchi design. The experimental results were transformed into signal-to-noise (S/N) ratios. S/N ratio is defined as the ratio of the mean of the signal to the standard deviation of the noise. The S/N ratio indicates the degree of the predictable performance of a product or process in the presence of noise factors. The S/N ratio for wear rate using 'smaller the better' characteristic, 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 (wear and surface roughness) and n is the number of observations. The above S/N ratio transformation is suitable for minimization of percentage of weight loss.

C. *Sliding wear behaviour in Simulated body Environment*

Hank solution is used to simulated the body fluid and sliding wear behavior in wet condition was recorded by conducting experiments as per Taguchi orthogonal array. The experimental values were transformed into S/N ratios for measuring the quality characteristics using MINITAB software. The S/N ratio obtained for all the experiments are shown in Table-VIII and response to Signal to Noise ratios are presented in Table-IX . Maximum weight loss obtained was listed in Table-X.

Table VIII

Percentage of weight loss and S/N Ratio's for sliding wear behavior in simulated body fluid

Expt No.	L N	S m/sec	D m	Thick µm	% of Weight loss	S/N Ratio
1	10	0.6	0.25	100	0.036048	28.86241
2	10	0.9	0.5	200	0.093957	20.5414
3	10	1.2	0.75	300	0.074279	22.58263
4	10	1.5	1	400	0.028034	31.04633
5	30	0.6	0.5	300	0.211574	13.49074
6	30	0.9	0.25	400	0.011454	38.82083
7	30	1.2	1	100	0.087556	21.15424
8	30	1.5	0.75	200	0.056573	24.94788
9	40	0.6	0.75	400	0.118522	18.52405
10	40	0.9	1	300	0.050421	25.94773
11	40	1.2	0.25	200	0.045604	26.81994
12	40	1.5	0.5	100	0.098064	20.16978
13	50	0.6	1	200	0.197422	14.09209
14	50	0.9	0.75	100	0.128255	17.83849
15	50	1.2	0.5	400	0.078572	22.09461
16	50	1.5	0.25	300	0.140441	17.05014

Table IX

Response Table for Signal to Noise ratios- Smaller is better (% Weight loss)

Level	Load, N	Speed m/sce	Distance Km	Thickness Micron
1	25.76	18.74	27.89	22.01
2	24.6	25.79	19.07	21.6
3	22.87	23.16	20.97	19.77
4	17.77	23.3	23.06	27.62
Delta	7.99	7.04	8.81	7.85
Rank	2	4	1	3

Table X

Maximum Percentage of weight loss obtained for substrate and coated specimens in wet condition

	S.No	Percentage of weight loss
1	Ti6Al4V substrate	7.25
2	Cr_3C_2 - NiCr Coated Ti6Al4V	0.211574

D. *Analysis of variance*

ANOVA was used to determine the design parameters significantly influencing the wear rate (response). Table-XI shows the results of ANOVA for wear. This analysis was evaluated for a confidence level of 95%, that is for significance level of α=0.05. The last column of Table-11 shows the percentage of contribution of each parameter on the response, indicating the degree of influence on the result. It can be observed from the results that significant parameter for sliding wear in Hank's solution is Distance (26.73%) followed by load (23.10%), Thickness(21.36%) and Speed(15.95%).

Table XI

Analysis of Variance for SN ratios- Wear in wet condition (Hank solution)

Source	DF	Seq SS	Adj SS	Adj MS	F	P	Contribution
Load	3	149.24	149.24	49.75	1.80	0.320	23.10
Speed	3	103.05	103.05	34.35	1.24	0.431	15.95
Distance	3	172.67	172.67	57.56	2.08	0.281	26.73
Thickness	3	138.00	138.00	46.00	1.67	0.343	21.36
Residual Error	3	82.83	82.83	27.61			
Total	15	645.79					

E. Energy Dispersive X-Ray(EDAX) Analysis

Energy Dispersive X-Ray(EDX) Analysis a technique used for elemental analysis or chemical characterization of metals. SEM- EDAX of Cr_3C_2 - NiCr Coated Ti6Al4V before and after wear test in Hank's Solution are shown in Figure-5 and Figure-6. It is observed from the EDAX pattern is that, the coating elements are presented after the wear test. It is observed from the weight percentages that wear rate is less in the Hank's solution.

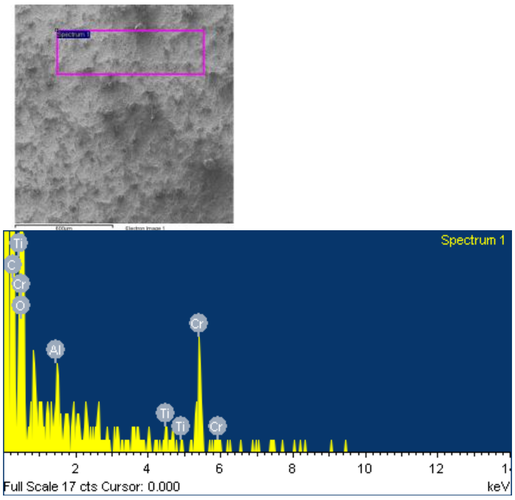

Fig 1 : SEM - EDAX of Cr_3C_2 - NiCr Coated Ti6Al4V before wear test

Table XII

Elemental Analysis before wear test

S.No	Element	Weight %	Atomic %
1	C K	43.49	67.15
2	O K	15.55	18.02
3	Al K	0.53	0.36
4	Ti K	1.34	0.52
5	Cr L	39.10	13.95
	Total	100	100

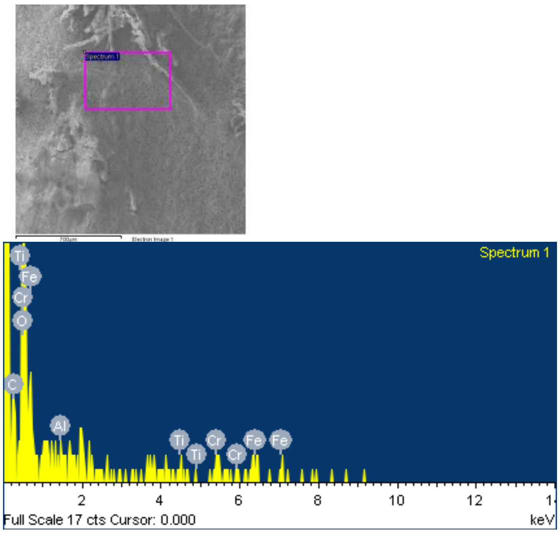

Fig 6 : SEM- EDX of Cr_3C_2 - NiCr Coated Ti6Al4V after wear test in Hank's Solution conducted with load- 30N, sliding distance -0.6 m/min, sliding distance 0.5 m and coating thickness 300 µm

Table XIII

Elemental Analysis after wear test

S.No	Element	Weight %	Atomic %
1	C K	38.40	64.34
2	O K	13.93	17.52
3	Al K	0.10	0.07
4	Ti K	0.81	0.34
5	Cr L	35.86	14.85
6	Fe L	10.90	2.88
	Total	100	100

IV CONCLUSIONS

A. Cr_3C_2 - NiCr coating on Ti6Al4V substrate was successfully employed using Detonation Spray technique.

B. Hardness of the substrate is improved from 25.67HRC to 53.33HRC

C. Percentage weight loss was decreased from 9.77 to 0.2115 for Substrate to Coated Specimen in wet condition respectively.

D. ANOVA results proved that Contributing factors for sliding wear in Hank's solution are Distance, Load, Coating thickness and Speed.

E. SEM- EDAX analysis proved that the presence of coating materials and substrate elements on the top surface.

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