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Improvement of tribological properties of Ti6Al4V alloy by nitrogen ion implantation

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Abstract:- Ion implantation is the most versatile and superior surface modification method. It has several advantages compared with other modification methods. In this study, the nitrogen was implanted by a process of ion implantation at 60 keV with different fluences of 1 x 10^{16}, 5 x 10^{16}, 1x10^{17} and 5 x 10^{17} ions/cm^2. Corrosion resistance of Ti6Al4V and ion implanted Ti6Al4V were investigated by an electrochemical test, at 37^0C in normal saline solution. ICP-AES studies were carried out to determine amount of ions leached out from samples when kept immersed in normal saline solution. The implanted samples showed variation in the corrosion resistance and microhardness with varying fluences. The sample implanted at 1 X 10^{17} ions/cm^2 showed an optimum corrosion resistance.

Keywords:- Titanium alloy; Nitrogen ion implantation; Electrochemical corrosion; Microhardness.

I. INTRODUCTION

Titanium and titanium alloy are widely used in dentistry and orthopaedics [1-4]. They provide high biomechanical properties and chemical stability in biological systems than other materials such as stainless steel and cobalt chromium alloy. Ti and Ti6Al4V alloy has better physical and mechanical properties and excellent corrosion resistance [5-6]. However, the widely used Ti6Al4V was found to release toxic ions Aluminium (Al) and Vanadium (V) into the body, leading to undesirable long term effects. It is reported that wear and corrosion are the main reasons for degradation of surgical implants such as hip and knee joint implants, which usually happens after 10–15 years of use [7]. Titanium is safe and bio-compatible in many in vitro and vivo studies because it forms a stable TiO_2 film which can release titanium particles under the wear in to the body environment. Thus, many attempt to reduce the corrosion, wear processes and better biocompatibility of titanium alloys have been performed by various surface modification techniques [8-9]. Ion implantation serves as a versatile tool for surface modification of biomaterials, though it is similar to the coating process, it does not involve the additional layer on the surface of sample. It creates alteration in surface properties of solids or the bulk properties of the underlying material and is independent of thermodynamic constrains. It has several advantages compared with other modification methods, low temperature treatment, no interface discontinuity as in film deposition and new metallurgical phases can be achieved with precisely controlled of ion fluences. Among the various ions to be implanted, nitrogen ion is the most suitable for biomedical applications. Nitrogen ion implantation improves the tribological properties such as hardness, corrosion resistance, wear resistance [10-12]. The nitrogen ion implantation improves the surface properties of Ti6Al4V. In this paper we had studied the corrosion behaviour of surface modified Ti6Al4V alloy by nitrogen ion implantation in normal saline solution conditioned by electrochemical method.

II. EXPERIMENTAL METHODS

The Ti6Al4V alloy was in the sheet form and sheet was cut into 15mm x15mm square samples with diameter 0.5mm. Prior, to study the Ti6Al4V samples were polished using silica carbide paper of 320,800,1000,1500,2000 and 2500 grit. Final mirror polish was carried out by 0.5µm grade diamond lapping in order to produce scratch free surface. The polished samples were subsequently cleaned in acetone, alcohol and de ionized water respectively. The samples were further subjected to ultrasonic cleaning in acetone for 20 minutes, rinse in de ionized water, dried and used for further studies. The elemental composition is shown in Table 1. The nitrogen ion implantation was done at LEIBF, IUAC, New Delhi, India. Nitrogen ion implantation on Ti at energy of 60 keV with different fluences of 1 X 10^{16}, 5 X 10^{16}, 1 X 10^{17} and 5 X 10^{17} ions/cm^2.

The electrochemical corrosion test was carried out using conventional three- electrode cell of 300 ml capacity by using Gamry-potentiostat/Galvanostat reference 3000, Tafel extrapolation method was used to calculate the corrosion rate. The cell was fitted with working electrode, saturated calomel electrode (SCE) as the reference electrode and the platinum as a counter electrode. The studies were carried out in normal saline solution at 37±1^{0}C with scan rate 1 mV/s and electrode potential was raised from -800 mV to 1000 mV. The solution was de aerated with pure argon (Ar) gas throughout the experiment. The critical parameters like Ecorr, Icorr, βa, βc and corrosion rate in mpy were calculated from the Tafel plots.

For the dissolution test, six samples of bare and nitrogen implanted Ti6Al4V alloy of surface area 1 cm^2 were immersed in 50 ml of normal saline solution in polypropylene bottles. The bottles were evacuated, tightly closed and incubated in thermostatic chamber at 37±1^{0}C. All bottles were shaken and rotated at the speed of 72 rpm. At the end of 4th, 8th, 16th, 32 and 64th weeks the solutions were analysed by Inductive Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) to determine the elemental concentration of Al and V leached out from the surface of samples.

Micro hardness measurement on six samples of each type were performed by mean of a micro hardness tester of indenter type Vickers for 5 seconds and test load of 10gm. Average micro hardness was calculated.

III. RESULT AND DISCUSSION

Table 1 Chemical composition wt. % of Ti6Al4V

C	Mn	Ni	Mo	V	Ti	N	Sn	Fe	Al
0.036	<0.001	0.008	0.004	4.08	89.56	0.008	0.011	0.25	5.97

The nitrogen implanted Ti6Al4V showed variations in the corrosion resistance with varying fluences. The sample implanted with fluence of 1X 10^{17} ions/cm^2 (sample R) showed an optimum corrosion resistance. The corrosion rate decreases with increase in fluences up to 1x10^{17}ions/cm^2 beyond which the corrosion rate increases i.e. for sample S with fluence 5x10^{17} ions/cm^2. At this high fluence 5 X 10^{17}ions/cm^2, ion bombardment may induce local variations in sputtering yield resulting in major changes in surface topography and induce defects that build up within sample results in increase in corrosion rate. Nitrogen implantation produces nitride layer on the surface of the sample which increases corrosion resistance. The order of corrosion rate obtained in normal saline solution is R < Q < S < P < 316L stainless steel < Ti6Al4V < NiTi. The corrosion rate of sample R was found to be 1.625e^{-3} mpy and E_{corr} was -59.94 mV in normal saline solution. Table 2 shows corrosion rate of nitrogen implanted Ti6Al4V alloy in normal saline solution. The result shows that nitrogen ion implantation increases the corrosion resistance of Ti6Al4V alloy in normal saline solution.

Table 2 Corrosion rate of nitrogen implanted Ti6Al4V NS solution

Sample code	Sample	Energy	Fluenc ions/cm^2	β_a V/decade	β_c V/decade	E_{corr} mV	I_{corr}	Corrosion Rate in mpy
A	Ti6Al4V	--	--	--	--	-763.0	1.603 µA	**1.00**
B	NiTi	--	--	--	--	-512.7	1.563 µA	**0.82**
C	316L SS	--	--	--	--	-419.4	1.128 µA	**0.51**
P	Ti6Al4V	60 KeV	1X10^{16}	489.2 e^{-3}	180.2e^{-3}	-246.2	10.55 nA	**8.717e^{-3}**
Q	Ti6Al4V	60 KeV	5X10^{16}	467.4 e^{-3}	333.0 e^{-3}	-95.91	3.181 nA	**2.022e^{-3}**
R	Ti6Al4V	60 KeV	1X10^{17}	283.5 e^{-3}	331.9e^{-3}	-59.94	2.556 nA	**1.625e^{-3}**
S	Ti6Al4V	60 KeV	5X10^{17}	315.0 e^{-3}	260.1e^{-3}	-143.8	4.688 nA	2.980e^{-3}

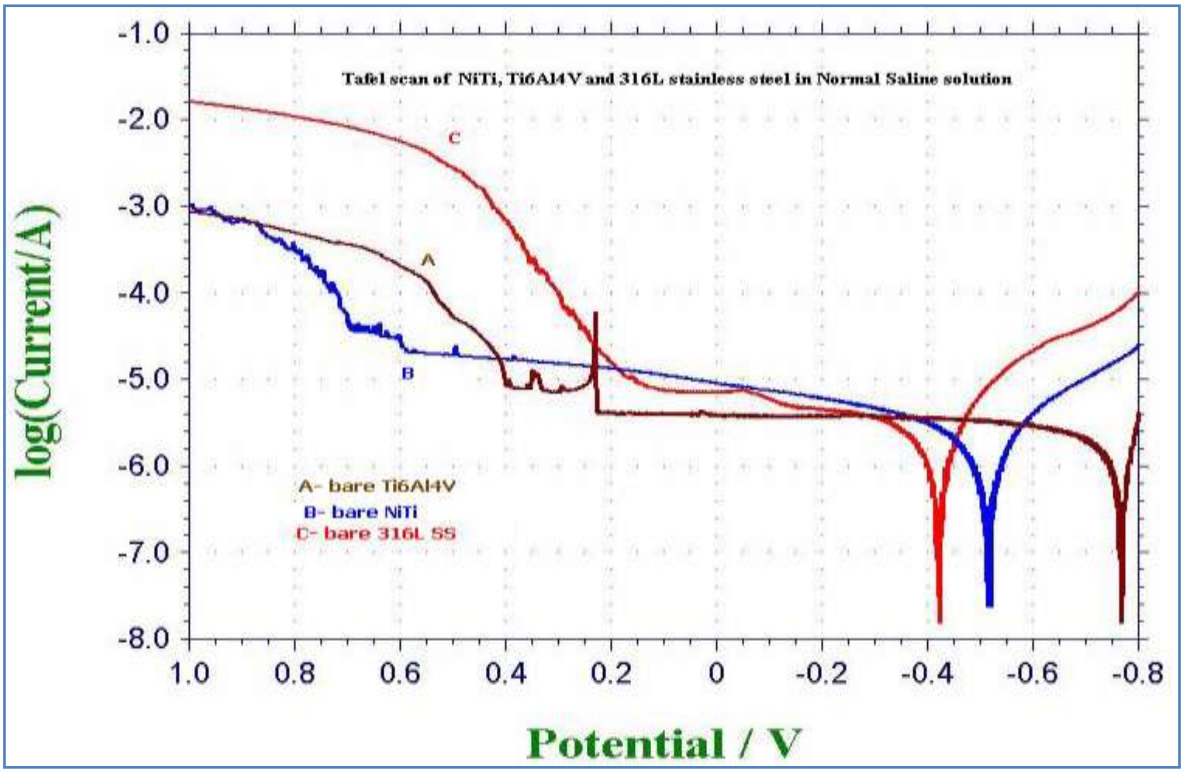

Fig.1 Tafel plot of bare TI6Al4V, NiTi and 316L stainless steel in normal saline solution.

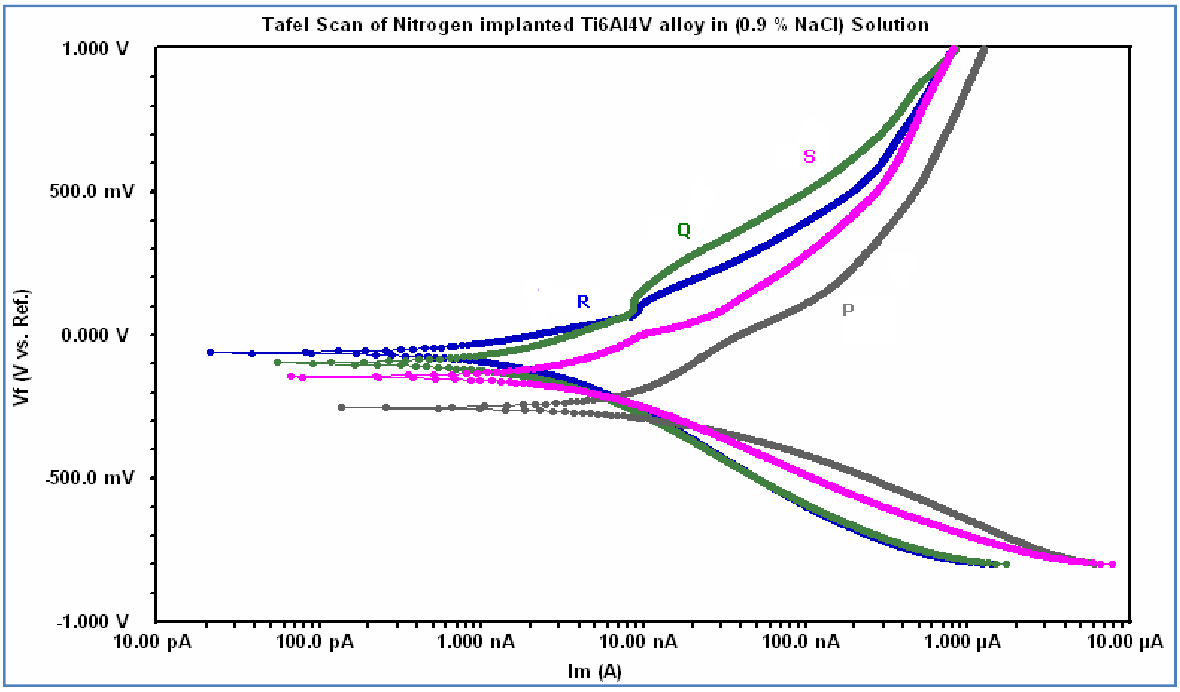

Fig.2 Tafel plot of nitrogen implanted Ti6Al4V alloy in normal saline solution.

The leaching out Al and V from the surface of unimplanted and nitrogen implanted Ti6Al4V alloy is shown Table 3. In dissolution test only sample- P showed Al ion leach out from the surface of sample after 64 weeks and it was found to be 24.15 ppb in normal saline solution where as all other nitrogen implanted sample showed Al and V ion concentration to undetectable level (ND). Nitrogen implanted sample showed improvement in elemental diffusion resistance.

Sample code	Specimen	4th week ppb	8th week ppb	16th week ppb	32nd week ppb	64th week ppb
A	V	ND	ND	ND	8.38	12.15
	Al	80.34	141.12	172.26	193.41	527.25
P	V	ND	ND	ND	ND	ND
	Al	ND	ND	ND	ND	24.15
Q	V	ND	ND	ND	ND	ND
	Al	ND	ND	ND	ND	ND
R	V	ND	ND	ND	ND	ND
	Al	ND	ND	ND	ND	ND
S	V	ND	ND	ND	ND	ND
	Al	ND	ND	ND	ND	ND

Fig.3 showed the micro hardness study of nitrogen implanted Ti6Al4V alloy. The result showed that the micro hardness increased as fluences increased. The sample S showed the highest micro hardness of 2950 Hv due to surface irradiation.

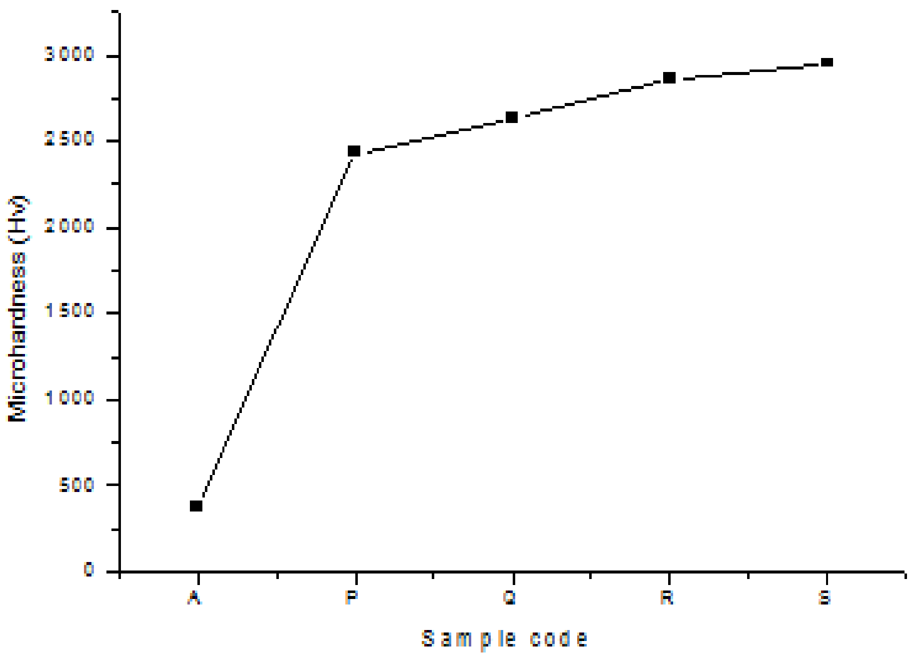

Fig.3 Micro hardness study

IV. CONCLUSION

The nitrogen implanted Ti6Al4V alloy showed improvement in corrosion resistance and elemental diffusion out resistance in normal saline solution. The nitrogen implanted samples showed variation in the corrosion resistance with varying fluences. The sample implanted at fluence $1x10^{17}$ ions/cm^2 showed optimum corrosion resistance. As fluences increased, micro hardness increased due to surface irradiation.

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