



## Evaluation of the Ti6Al4V Alloy Plasma Nitride for Orthopedic Implants: in Vitro Biocompatibility Study in Simulated Body Fluid

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### Abstract

In view of this work, plasma nitriding was executed on Ti-6Al-4V and the effect of nitriding by using glow discharge with 2 mbar of Ar+N<sub>2</sub> gas on corrosion resistance with different nitriding time and studied. The structure properties and the external appearance for alloys were performed with x-ray diffraction and optical microscopic. The investigation of XRD indicates the Ti alloys are polycrystalline with a cubic type (bcc). When the external appearance of the surface indicates was of  $\beta+\alpha$  structure which have been better technical properties on biomedical application. The biocompatibility investigation of nitride alloys *in vitro* medium containing human body fluid: we showed a layer of hydroxyapatite HAP which might be considered in good biocompatible ceramic with the expected ability for chemical bonding with bone.

**Key words:** plasma nitride, corrosion, biocompatibility, Ti alloys.

### 1. Introduction

Ti transiently allows a deficient shell in its electronic structure to power onward form solid explanation with might substitution aspect receive a magnitude factor inward  $\pm 20\%$ . In its rudimental form titanium gets an immense thawed point (1678°C), illustrate a hcp crystal arrangement  $\alpha$  up to the beta (882.5°C), reconstruct to a bcc structure  $\beta$  raised by this temperature. Ti alloys whatever has been broadly enforced in militant, biomedical and



chemical business [1]. This is primarily associated to its admirable corrosion defiance and outstanding biocompatibility. The Ti6Al4V and Ti6Al7Nb alloys have a bio-inert layer, whatever has low hardness and indigent wear resistance [2]. That one may reform its outer equity and attribute, a number of outer medication alteration operation were case [3]. So maintain accomplished quality in Ti alloys and advance the neurological properties of exterior expanse, assorted expanse conversion approach are hired, being plasma spraying, physical vapor deposition plasma nitriding [4]. Amid the assorted materials accessible in the retail, metallurgic biomaterials like titanium alloys are generally used are the instill surgery operation as a result of their good corrosion defiance. There are many reports in improving the bioactive coatings, as well as the enormous use of nitride on the surface of biomaterials [5].

Ti6Al4V has outstanding corrosion resistance to physical fluids and good comparability, due to its nitriding film passivation stability. Thus it is not only widely used in dental implants and bone wounds but also has become a paramount material for human body hard tissue substitutes [6]. Corrosion of humanoid mineral implant is demanding by virtue of it can negatively involve biocompatibility and habitual integrity [7].

There is handful senses tigger revision in joint replacements. Firstly, enduring going off society has carried an ever-increasing demand for materials individually for personal torso. It has been predicted 90% of community over the age of 40 experiences from retrogressive malady and the elderly people culture has heightened exceedingly newly [10].

## 2. Experimental

Affecting materials used in the present investigation were Ti6Al4V (all compositions are in weight %) as shown in table 1. All alloys were accepted in from about rods as regards aspect 2.5 cm diameter also cutting rod by diamond cutter (struers Denmaek) to pellet 0.5 cm thickness. They were procured from Anna University, India. The variety was than scrubbed in infuse water pursue by ultrasonic cleaning in ethanol. The materials used as regards present investigation were Ti6Al4V (all compositions were in weight %) as shown in table 1. All alloys were received in from of rods of aspect 2.5 cm diameter and cutting rod by diamond cutter (struers Denmaek) to pellet 0.5 cm thickness. They were procured from Anna University, India. The variety was washed in distilled water followed by ultrasonic cleaning in ethanol. The microstructures of the surfaces bare for electrochemical trial abide were again calculated using an optical microscope (Nikon type 120/Faban 50-1000X). The obtained Ti6Al4V samples were used as a substrate, and nitriding with 90% nitrogen gas. The substrate was not intentionally heated during the deposition. Sputtering on Ti alloy, the experiments process, adverse bias was added to boost the energy of particles; the sputtering parameters were listed in **Table (2)**.

**Table 1.** Nitriding condition for Ti6Al4V alloy

parameter	Condition
pressure	$5 \times 10^{-1}$ mbar
Substrate	Ti6Al4V
Sputtering	90%N <sub>2</sub> +10%Ar
Deposition time	15-25 hour
Applied voltage	600 volt
Discharge current	0.63 A
Inert electrode spacing	4 cm

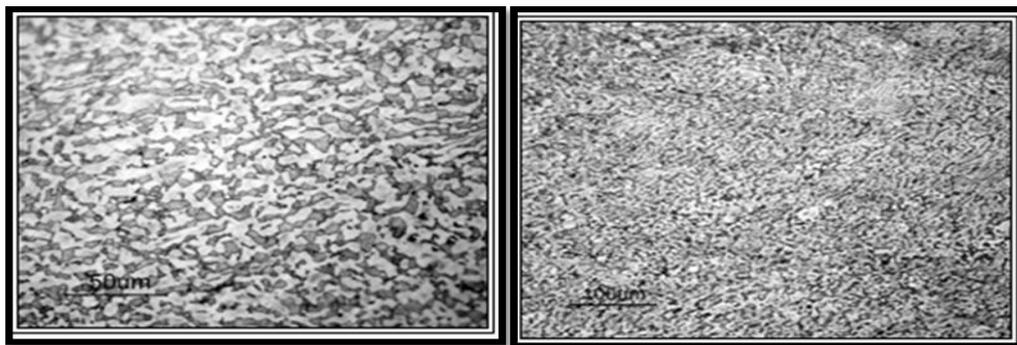
The obtained samples were examined by route of an X-ray diffraction (XRD) amount. In this research, X-ray diffractometer kind SHIMADZU, power diffraction arrangement along Cu-K $\alpha$  X-ray duct ( $\lambda = 1.54056 \text{ \AA}$ ) was worn. The X-ray checks were performed betwixt binary angles 30° also 80°. The electrochemical studies abide attend accepting a (Elektonic Germany Bank) Potentiostat, blend to a computer. The electrolyte was used for simulating human body fluid accustom, willing using laboratory, as shown in **Table 2**. The PH of quick fix was upheld at 7.4. of late prepared quick fix was nearly new for each analysis.

**Table 2.** Chemical composition for simulated body fluid

	CONSTITUENT	WEIGHT (gm/L)
1	NaCl	8.036
2	KCl	0.225
3	CaCl <sub>2</sub>	0.293
4	NaHCO <sub>3</sub>	0.352
5	K <sub>2</sub> HCO <sub>3</sub>	0.230
6	MgCl <sub>2</sub> .6H <sub>2</sub> O	0.311
7	Na <sub>2</sub> SO <sub>4</sub>	0.072

### 3. Result and Discussion

Affecting obtained images of the microstructures of samples tested are shown in **Figure (1)**. Etching solution contains 5ml HCl: 10ml HNO<sub>3</sub>:85ml H<sub>2</sub>O, impressed alloys in solution for 1 min then cleaned with dissolute water. We can observe two phases the base structure from beta-Ti and (beta+alpha) structure, that bcc structure. ( $\alpha+\beta$ ) structure has excellent applied properties in surgical implants. The typical topography of nitride intermediate layer TiN+Ti<sub>2</sub>N+ $\alpha$  (TiN), originated by glow discharge nitriding scheme, is shown in **Figure2**. The blanket is characterized by great solid, structural homogeneity, and well-being glue with a metal basement. Macroscopic samples of Ti6Al4V produced by these methods are shown in **Figure 3**.



**Figure 1.** microstructure of Ti6Al4V

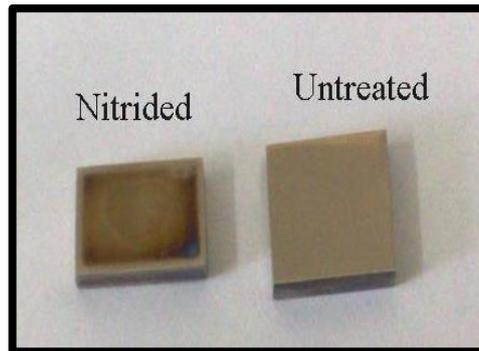


Figure 2. Ti6Al4V alloy before and after nitriding

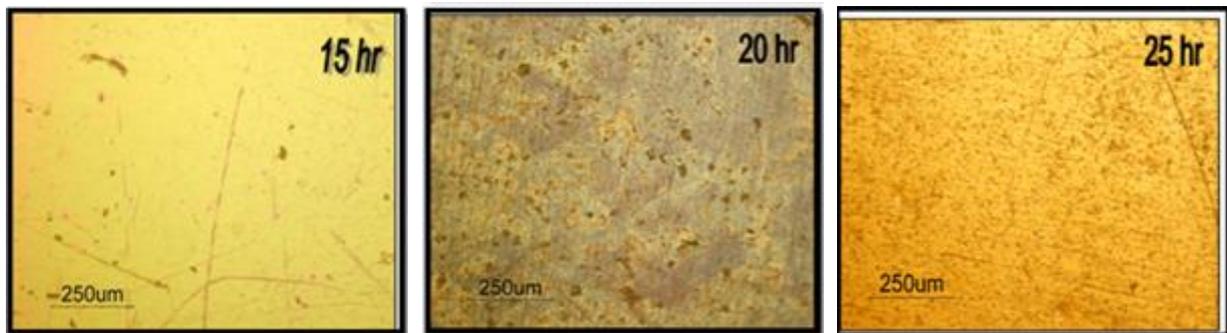
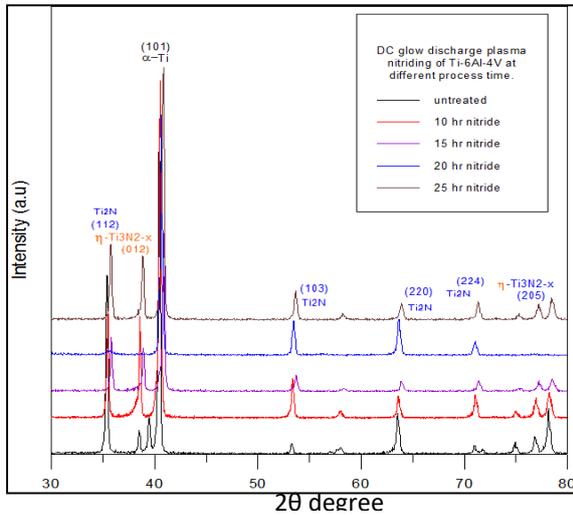
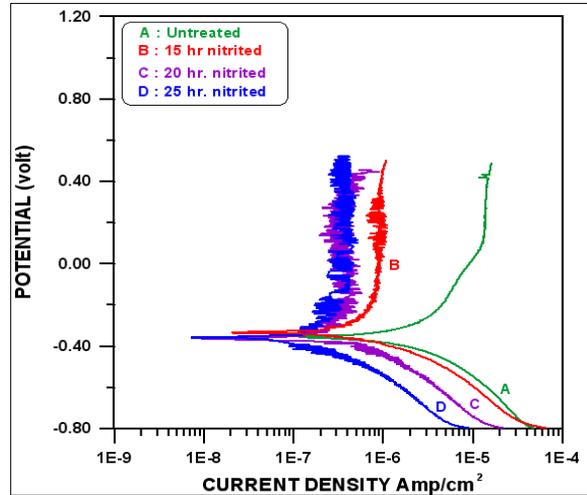


Figure 3. microscopy photo for alloys after nitriding 15, 20 and 25 hours

The XRD reasoning (see **Figure.4**) settled the habitation of commixture 3-zone expanse layer subsist of  $\delta$ -TiN,  $\xi$ Ti<sub>2</sub>N, and  $\alpha$ -TiN phase. It is admitted matter that the nitriding of metal expanse revises its mechanical estate [10] and corrosion resistance. Consequently, TiN+Ti<sub>2</sub>N+ $\alpha$ TiN the layers on titanium and their alloys reform their corrosion resistance. Apart from that, TiN+Ti<sub>2</sub>N+ $\alpha$ TiN layer feasible symbolize by biocompatibility and shortfall of cytotoxicity [9].it has also been established that the limited nitriding of Ti installment in nitride/Ti amalgam coatings revised the bond vitality of blanket on Ti substrate [8].



**Figure 4.** XRD of Ti6Al4V before nitriding



**Figure 5.** Potential curves for Ti6Al4V before and after nitriding

The potentiodynamic emission curves of the nanocrystalline Ti6Al4V alloys in SBF before and after nitriding are shown in **figure 5**. The corrosion potentials and corrosion current quantity of each specimen were resolute from the potention dynamic polarization curves by the Tafel extrapolation approach and are epitomize in Table 3. According to Table 1 and Fig. 3, the nanocrystalline Ti6Al4V alloy after 25 hours nitriding was more corrosion resistant ( $I_c=4 \times 10^{-7}$  Amp/cm<sup>2</sup>) than before nitride ( $I_c=0.19 \times 10^{-6}$ ). As reveal by a variation of the polarization curve (**Figure. 5**) to the right down of the microcrystalline Ti6Al4V curve betwixt untreated and nitriding at different times. Operation of nanocrystalline Ti-based materials also focused on other attentions on the biocompatibility of incorporate alloys; corrosion defiance is one of the director wealth an implant should possess. The success of an embed bet on the genuine review of this property, whatever is straight it related to its capacity to proceed along with the relentless body environment. Bad corrosion defiance in the carcass liquid will element the clemency of irreconcilable mineral ions from the fix, which is a duct aspect leading to allergic along with toxic attitude [10].

**Table 3.** Corrosion characteristics current densities ( $I_c$ ) and corrosion potentials ( $E_c$ ) of studied Ti alloys.

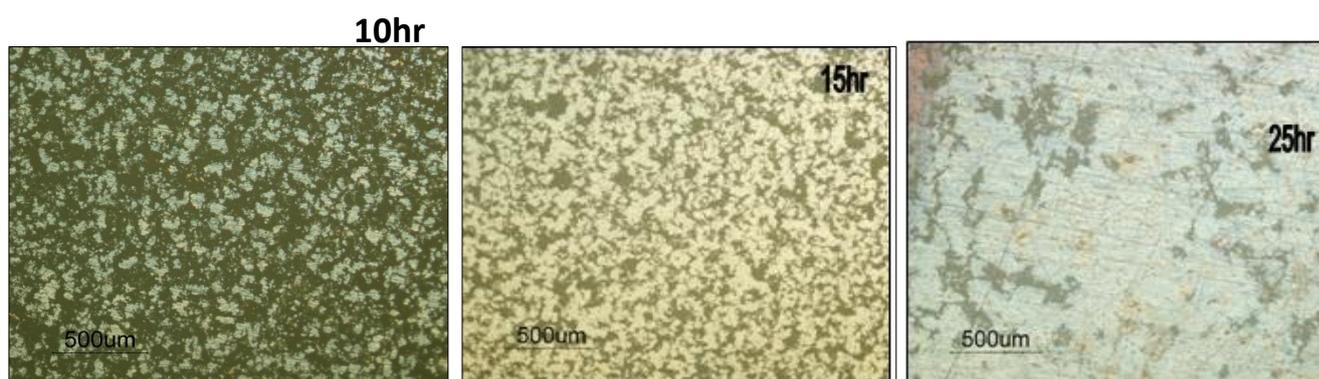
Nitride time(hours)	$E_{corr}$ (mv)	$I_{corr}$ (Amp/cm <sup>2</sup> )	Corrosion rate (mm/year)
untreated	-754.1	$0.19 \times 10^{-6}$	0.055
15	-360.281	$1.801 \times 10^{-7}$	$1.634 \times 10^{-3}$
20	-375.2	$1.65 \times 10^{-7}$	$1.503 \times 10^{-3}$
25	-334.36	$4 \times 10^{-7}$	$3.662 \times 10^{-3}$

**Table 4.** toxic metals in Ti6Al4V alloys before and after nitride

sample	Al (ppb)	V (ppb)
untreated	123	2.5
15 hr	62.5	0.058
20 hr	36.25	0.111
25 hr	26.25	0.098

Stimulated body fluid (SBF) laboratory prepared as a medium for biocompatibility of Ti6Al4V alloys before and after nitriding. Fig.4 shown toxic metals compounds in alloys and the effect of nitriding times after being impressed for one month. Annealed alloys at 400C for 4 hours after impressing the alloys in SBF, then used optical microscopy to showed the layer of Hydroxyapatite (HA) ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH}_2)$ ) on the surface of the alloys. Biocomposite materials accept been matured that one may associate bioactivity of ceramics. Hydroxyapatite (HA) is familiar for its deficiency and frail [11] but has an exceptional biocompatibility and bioactive substantial.

Though HA is coated on titanium, an renovation of the biomaterial chemical properties crop up from the perspective of potential bio-applications, the opportunity of HA surge in the corporeal habitat is more momentous that the fact of its presence on the surface of an implant. The bio-activity tests of inventing composite structure signify not e only that the HA layer dissolves, but it quickens the HA growth. It upholds reap familiar bioactivity ownership by conglomerate layers. fig. 6 showed micro optical scope for alloys after nitride at 15,20 and 25 hours; we can see the HA layer on the Ti6Al4v surface, which covered almost the surface point of for bone growth ability on the surface of treatment alloys.



**Figure 6.** Optical microscope photo for Ti6Al4V after impressed in SBF for one month

#### 4. Conclusion

XRD analyses show that a polycrystalline structure film with the cubic structure of Ti6Al4V and the nitride peaks show growth TiN+Ti<sub>2</sub>N+ $\alpha$ TiN and  $\delta$ -TiN,  $\xi$ Ti<sub>2</sub>N. XRD patterns are good in agreement with the standard patterns.

Advancement of implants with great corrosion and wear intransigence is of great emphasis for the durability of a biomaterial in our human scheme. The inability of an implant emerge to harmonize with the adjoining bone and other tissues by virtue of micro motions can also event in implant detachment.

Increase passivity effect on corrosion behavior and make alloys more compatibility to be used in the human body. Decrease the amount of (Al and V) metals after nitride and impressed for one month in SBF because of bioactive HA layer.

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