Evaluation of the stiffness and friction of Ti6Al4V ELI treated by glow discharge nitriding

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Abstract. In this study, an evaluation of the elastic-plastic surface hardening on Ti6Al4V ELI titanium nitride films obtained by glow discharge method was carried out by nanoindentation tests according to the standard ISO 14577. The nanotribological properties (metal-metal) were also evaluated using the pin-on-disc system Ti6Al4V surface deposition ELI with nitrogen, obtaining a correlation between the coefficient of friction of Ti6Al4V ELI treated by PVD and the Young's modulus of the respective substrate modified by PVD. To characterize the substrate for the characterization tests, scanning electron microscopy, atomic force microscopy and X-ray diffraction and contact angle were carried out. The results demonstrated that the substrates nitrided improved mechanical and tribological properties, hardness, Young's modulus and coefficient of friction, making the alloy Ti6Al4V ELI support axial loads in tension and compression.

1. Introduction
Nitriding of metallic materials is well known to enhance resistance to wear and corrosion, thereby reducing the coefficient of friction, and getting better tribological properties [1] technique. It is obtained by using different methods gaseous. For this work, the need for a suitable substrate, with acceptable hardness, in which there are no phase changes, the need to nitriding is suggested by the method of intense plasma discharge, which is a gas was raised (rich in nitrogen) in which an electric glow discharge generates nitrogen ions onto the desired substrate, favouring the diffusion in the substrate [2], in our case Ti6Al4V ELI alloy. Nitriding by physical vapour deposition (PVD for short) is a proprietary process engineering materials which ions of a material can be implanted into another solid, where Implanted ions are distributed in depth ranging between 200 and 2000 atomic layers (0.05µm 0.5µm) depending on the type of ion, the base material and the sputtering power. The maximum concentration, which for light ions such as carbon or nitrogen can exceed 50%, is located at a certain depth and then decreases smoothly [3]. The aim of this study was to provide the tools to analyse nanomechanical behaviour of nitriding by the method of intense plasma discharge and propose recommendations that lead to improved standards substrate.

2. Experimental
The material used was ELI alloy Ti6Al4V, its chemical composition is presented in Table 1. The dimensions of the specimens used were 12mm diameter and 2mm thick.

The samples were polished with silicon carbide paper, from 240 to 1200 and then from coarse and fine cloth polishing, of 0.3µm and 0.05µm with alumina slurry. Thereafter ultrasonic cleaning with
alcohol is performed for 15 minutes. The surface modification of Ti6Al4V ELI, nitriding was performed by intense plasma discharge method, this process for a NitrEos T700x900x1300 Tandem System, which has a working volume of 700mmx900mm and 700mmx1300mm and having a heating technology used hot wall and a system operating range 350 to 600°C, with 25 kwatt power source. 800V up to 10kHz, and pulse power of 70kWatt. Specimens worked at a temperature of 450, 480 and 520°C, and residence time of 4 hours. The characterization by x-ray diffraction (XRD) was performed in a brand diffractometer Bruker D8 Discover. Nanoindentation test was performed using a penetrator that this investigation was a nanoindenter Berkovich tip. For tribological tests, the contact pair used was metal-metal (Ti6Al4V ELI pin - disc treated Ti6Al4V ELI) for the respective calculation of the friction coefficient.

Table 1. Chemical composition of the titanium alloy Ti6Al4V obtained by fluorescence spectrometry energy dispersive X-ray.

| Alloy element | Al   | V    | Fe   | N    | Ti    |
|---------------|------|------|------|------|-------|
| wt (%)        | 6.102| 4.119| 0.183| 0.05 | 88.66 |

3. Results and discussion

XRD results showed possible changes generated by the nitriding treatment on the microstructure that manifest at very small scales. Figure 1 shows the appearance of nitrides at low temperatures, between 450 and 520°C. These directly affect the hardness obtained by the substrate and is confirmed by other authors obtained nitrides below 773K, ε-Ti2N and δ-TiN and which are clearly present to an atmosphere N/H=3/2, after treatment of more than 240min [4].

![Figure 1. XRD for samples treated at different temperatures.](image)

The effect of the intense plasma discharge in the composition on the surface of Ti-6Al-4V ELI, is a very complex function of the process parameters: treatment time, the proportion of nitrogen, sample temperature and pressure. Figure 2 shows the graphs nanoindentation with temperature variation. These curves are nonlinear; the download data are used to determine the mechanical properties based on the theory of indentation, in which the portion of the initial discharge depth-load curve represents the purely elastic recovery.

The theory developed to determine the hardness and rigidity of a material by testing nanoindentation is based on the material is ideally elastoplastic and the material deforms around the indenter [5]. From the depth-load curve, the elastic modulus and hardness are calculated by the following equations:
Where \( A \) is the contact area of the penetrator, and \( E_r \) is the reduced modulus, this is defined by:

\[
E_r = \frac{\sqrt{\pi}S}{2\sqrt{A}}
\]

(1)

\[
\frac{1}{E_r} = \left(\frac{1-n_m^2}{E_m}\right) + \left(\frac{1-n_i^2}{E_i}\right)
\]

(2)

Where \( E_m \) and \( V_m \) are the Young’s modulus and Poisson’s ratio of indented material respectively, and \( E_i \) and \( V_i \) saw the indenter. The hardness of the alloy \( H \) is defined as the maximum load \( P_{\text{max}} \), divided by the projected area of the indentation area under this load, that is, \( H = P_{\text{max}}/A_{\text{max}} \). Table 2 shows the results obtained in nanoindentation tests.

![Figure 2](image)

**Figure 2.** Nanoindentation curves for samples treated at different temperatures.

| Samples     | Nanoindentation hardness (GPa) | Young’s modulus (GPa) |
|-------------|--------------------------------|-----------------------|
| Untreated   | 3.9±0.2                        | 271.8±2.4             |
| 450° C      | 6.4±0.4                        | 197.7±8.1             |
| 480° C      | 9.8±0.4                        | 161.7±6.1             |
| 520° C      | 7.9±0.3                        | 145.5±2.4             |

Regarding the sample without nitriding, hardness increased until the treatment temperature of 480°C and a slight decrease in this tendency to 520°C is observed. However, the Young’s modulus decreases with temperature nitriding, which relate it to some authors the accommodation of the nitrogen in the structure assuming a decrease in the interatomic distances of Ti6Al4V ELI, decreasing its rigidity and preventing it when subjected high stress, decrease the delamination and debris [6,7].

Glow discharge nitriding, reducing wear and friction in the alloy Ti6Al4V ELI, leading to low mass loss. In Figure 3, the comparison of the friction coefficient variation ELI deTi6Al4V untreated with nitrided samples at different temperatures is observed.

versus time is observed in the sample of Ti6Al4V ELI, not treated by nitriding, with coefficient values about 0.8. Coefficient of friction values of about less than half, compared to the value given in the untreated sample (between 0.29 and 0.32) were obtained in all samples nitried, being less friction in the treated sample at 520°C. In Figure 4(a) and 4(b), the morphology of friction damage is observed from the pin-on-disc tests.

In these SEM images observed in the nitrided samples, no wear debris, contrary to the presence of such wear on the untreated sample nitriding.
Figure 2.3. Friction behaviour over time for different samples of Ti6Al4V ELI nitrided at different temperatures.

Figure 4. Images by scanning electron microscopy: (a) sample nitrided at 520°C and (b) not nitrided.

4. Conclusions
Nitriding by glow discharge in Ti6Al4V ELI allows decreasing the rigidity of the alloy to improve the tribological behaviour of mass loss by wear and friction, avoiding the presence of debris.

Acknowledgment
Our sincere thanks to COLCIENCIAS, for funding this project under contract no. 780-2011.

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