Nanocharacterization of TiN films obtained by Ion Vapor deposition.

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Abstract. We evaluate and characterize the surface at the nanoscale level and take into account the temperature variation effect in the process of plasma ion deposition for H13 steel samples coated by Titanium Nitride (TiN). The interferometric microscopy and atomic force microscopy (AFM) were used to measure the film to analyze the variation of structural and morphological properties of nanofilms that depend on the temperature of substrate.

1. Introduction

Coatings of Titanium Nitride TiN, prepared by the method of physical vapor deposition PVD have been studied since the early 80’s [1]. The main purpose of these coatings is to provide to the industrial tools an increase of the resistance, improve wear properties and friction coefficient, hardness, toughness, resistance to oxidation among others, thus improving their useful life.

The structural and morphological properties of TiN films depend mainly on the process and the deposition conditions. During the deposition of the films there are different variables that need to be controlled directly because they affect the final properties of the coatings, among these are the variation of the substrate temperature, the substrate bias and nitrogen partial pressure.

With the advancement of nanotechnology it is necessary to innovate new microscopy techniques that facilitate the characterization of the morphology and mechanical properties of new materials. Techniques used nowadays are the interferometric microscopy and atomic force microscopy (AFM).

AFM makes on the sample a sweep with atomic distances by a very fine tip attached to the free end of a flexible support (cantilever) of the length from 100 to 200µm. Tip experiences a force when it approaches to the sample surface and from the deflections arising from this close contact the information about the physical characteristics of the studied area is obtained [2-3].

Interferometric microscopy is a technique that allows non-contact analysis of the surface topography of the sample to be tested. This technique combines the light reflected from the surface of the sample with a reference beam of the microscope itself forming interference fringes which are captured by a CCD camera.

The present work aims at thickness and topography nanocharacterization of the TiN films deposited by the method of physical vapor deposition PVD on H13 steel samples, using the techniques of AFM and interferometric microscopy.
2. Experimental Procedure

2.1 Preparation of films

The deposition of thin films on H13 steel samples was performed by using the PVD method in the MOSMET reactor. This reactor belongs to the group of Plasma Physics and Technology of the Universidad Industrial de Santander and this device implements a new technology that consist of a hybrid process based on simultaneous processing (ion implantation and deposition of metals).

H13 steel samples, used as substrate, were previously polished with 80-1500 grit sandpaper to achieve a mirror-like surface and then were cleaned with alcohol to remove impurities.

For the TiN film formation cathodic arc evaporation technique was used, which consists of the evaporation of Ti by electric arc discharge and simultaneous introduction of nitrogen into the reactor chamber, which interacts with evaporated titanium and finally the compound TiN is deposited on the substrate (H13 steel) [4 - 5].

To find the appropriate temperature for better adhesion of the films, tests at different temperatures were carried out and it was found that the ideal temperature for the deposition of the films was between 400ºC and 425ºC.

2.2 Film characterization

For the topographic surface characterization of TiN films interferometric microscopy technique and AFM were applied. The first of them was implemented to determine the thickness of the TiN films using white light interferometry with maxima detection technique. Images were collected with a 20x Mirau objective using an Nikon interferometric microscope [6-7-8].

Scanning Probe Microscope (Veeco CP-II AFM) working in contact mode, delivered the roughness and grain size.

3. Results and discussion

3.1 Interferometric microscopy

Figure 1. shows images of the thickness of the TiN films deposited at different substrate temperatures in samples of H13. These images were obtained using a interferometric microscopy.

![Figure 1](image1.png)

**Figure 1.** Interferometric Microscopy images of film thickness of TiN on H13 steel. (a) 400ºC (b) 425ºC

To determine the thickness of the films an average measurement of the heights produced by the film step on each surface was taken. Thickness value of 1.76µm for substrate temperature of 400ºC and 1.02µm for substrate temperature of 425ºC was obtained. These results have an uncertainty of 10 nm, due to the axial resolution of the system.

For the semitransparent films, interferometric methods can be used to measure the thickness.
3.2 Atomic Force Microscopy AFM
To determine the morphology and topography of the deposited films an analysis of a scanning area of 20x20µm using an AFM was made. Figure 2 shows three-dimensional AFM images of H13 steel samples coated with TiN on substrate temperature of 400°C and 425°C respectively.

![AFM images of the surface morphology of TiN films on H13 steel](image)

**Figure 2.** AFM images of the surface morphology of TiN films on H13 steel (a) 400°C (b) 425°C

The two-dimensional AFM images of Figure 3 show the difference in grain size of H13 steel samples coated with TiN at a substrate temperature of 400°C and 425°C. The films deposited at a substrate temperature of 400°C showed an average grain widths of 2.99µm with peak average heights of 705nm while the films deposited at a substrate temperature of 425°C had grain widths of 0.596µm and peak average heights of 90.11nm. The roughness of films deposited on substrate temperature of 400°C was 20.18nm and 301.46nm in average for temperature of 425°C.

![AFM images of the grain size of TiN films on H13 steel](image)

**Figure 3.** AFM images of the grain size of TiN films on H13 steel (a) 400°C (b) 425°C.

The lateral resolution of AFM is better than of an optical microscope, However the axial precision of AFM is comparable to an interferometric microscope when the area being studied is in the height range of tenths of microns.
4. Conclusions

The effect of substrate temperature on structural and morphological properties of TiN coatings produced by PVD method (arc evaporation) was studied. From the analysis of images obtained by AFM it is clear that grain size decreases with increasing substrate temperature, likewise the surface roughness of the samples also decreases with increasing temperature and a better surface coatings is produced at a temperature of 425ºC than those obtained at a temperature of 400ºC.

It was determined the thickness of the TiN films deposited at a substrate temperature of 400ºC is thicker than of the films deposited at a substrate temperature of 425ºC.

5. References

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