Characterization of the structure of the coating of multilayers using AFM and Interferometric Microscopy.

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Abstract. Ti / TiN films were deposited on H13 steel and silicon substrates with different deposition voltage, by means of the cathodic arc evaporation (CAE) technique, this process was carried out by nanolayers deposition, requiring a detailed survey on growth films, for the properties characterization such as grain size, thickness and roughness of the film was used the atomic force microscopy (AFM) techniques and Interferometric Microscopy. Obtaining a the films growth when varying the deposition voltage.

Key words: AFM, Interferometric Microscopy, Thin Films, Nanometry.

1. Introduction

The H13 steel is a used material for industrial tools that are under some form of weathering; but this material has hardness variations, thermal and chemical stability, and corrosion resistance [1-2]. To improve its performance against these variations, this steel can be coated with different compounds by deposition techniques, an example is the cathodic arc evaporation (CATHODIC ARC EVAPORATION-CAE) [1-3].

In these days the titanium (Ti) and the compound titanium nitride (TiN) coatings are commonly used in industry to form multilayers, especially when they are deposited on various steel substrates, the main characteristics of these compounds deposited by this technique is its high hardness, good wear resistance and corrosion [4-5].

There are various microscopy techniques that facilitate the characterization of the morphology and mechanical properties of deposited films, among these techniques are the atomic force microscopy (AFM) and Interferometric Microscopy.

Atomic Force Microscopy is based on the interaction between a very fine tip and sample surface [4-6]. This cause a relative movement between tip and sample by a fine sweep based on piezoelectric materials. Keeping the interaction tip - sample steady, three-dimensional maps are obtained with a nanometric resolution in all the three axes [4-6]. The Interferometric Microscopy is a no contact technique, used for the characterization of surfaces in three dimensions. Interferometric objectives have a beam splitter that sends a portion of the light to the sample surface and part to a mirror, the reflected light of these two surfaces are recombined to form interference bands that are collected on a CCD camera. [7]
The objective of this study is to characterize some properties of the Ti / TiN films deposited on H13 steel and silicon substrates by the technique of cathodic arc evaporation (CATHODIC ARC EVAPORATION-CAE), those properties are characterized by the microscopy techniques of Atomic Force (AFM) and Interferometric Microscopy.

2. Experimental Procedure

Ti / TiN layers were deposited in H13 steel and silicon substrate using the technique of cathodic arc evaporation (CATHODIC ARC EVAPORATION-CAE), using the MOSMET reactor in the Plasma Physics Laboratory, University Industrial of Santander.

Before the film deposition, the H13 steel substrate was subjected to a process of polishing sandpaper from the 80 to 1600, the deposition technique is a shock in the form of electric arc generated to evaporate and ionize a material (cathode), the voltage difference between the substrate and the reactor chamber causes the ions to be focused, accelerated and projected by a magnetic field to the part to be coated. This technique has the advantage that the protective films fabricated on the substrate surface do not present adhesion problems; because of the high voltage no interface between film and substrate is formed.

To make a coating such as Ti / TiN the target material is Ti, which is evaporated by arc discharge in the presence of nitrogen gas (N2) located in the reactor chamber, the nitrogen reacts with titanium ions to form the deposited layer, there is also a very important feature in the deposition process: the substrate surface must be at a proper temperature that is maintained due to the kinetic energy of ions when they collide with the workpiece.

The morphological and surface study of the multilayered Ti / TiN was performed using the atomic force microscopy and interferometric techniques; using an Veeco Digital Instruments CP-II AFM Atomic Force Microscope (AFM) in contact mode, through which the topography and the surface morphology of the films was established, this analysis includes topographic roughness grain size and surface characteristics of homogeneity as well as the thickness of the films of different H13 steel and silicon samples, the Interferometric Microscope used is a Carls Zeiss Nikon with Interferometric objective, it was used to measure the thickness of the Ti / TiN films deposited on H13 steel and silicon substrate. We used 3 samples of H13 steel and 2 samples of silicon, silicon samples were deposited at different voltages to analyze the film growth.

The presented data for each substrate is the average obtained by examining each sample in different areas in order to observe the uniformity of the film.

3. Results and discussion

The morphological and surface characteristics of the Ti / TiN multilayers were determined with different techniques, whose results and analysis are presented below.

3.1. Analysis of Interferometer Microscope

The results of the thickness analysis of the Ti / TiN films for each substrate using the Interferometric microscope are presented in Table A1.
Tabla A1. Valores del espesor de las películas de Ti/TiN para cada sustrato utilizando el Microscopio Interferométrico.

| Muestras         | Espesor (nm)     |
|------------------|------------------|
| Acero H13        | 572.0-610.2      |
| Silicio 0.0 V    | 84.6-103.5       |
| Silicio 60 V     | 394.6-463.4      |

A Ti / TiN film growth by increasing the deposition voltage was established, this growth was determined by the thickness analysis of the two silicon samples deposited at different voltages. These films show a rise to 77%.

The literature suggests that the deposition under heavy ion bombardment promotes the development of a dense film with high structural quality with good toughness. [8]

The Image1 displays the thickness of the Ti / TiN films for each substrate homogeneity watching films on H13 steel substrate, and which do not jagged as displayed in the silicon substrate 0.0V image.

Imagen 1. Espesor de la películas Ti/TiN de diferentes sustratos utilizando el Microscopio Interferométrico. a) Sustrato de acero H13, b) Sustrato de silicio de 0.0V, c) Sustrato de silicio 60V

3.2. Atomic Force Microscope (AFM) Analysis

For each of the samples a scan area of 60µm x 60 microns was taken at a 0.5Hz frequency. The topographic analysis of the result obtained by Atomic Force Microscope found the value of average roughness. The film thickness and grain size in width and height values are in Table A2 for each substrate.
Table A2. Values of roughness, thickness and grain size in width and height of the films of Ti/TiN for each substrate using Atomic Force Microscope (AFM).

| Muestras      | Rugosidad (nm) | Espesor (nm) | Tamaño del grano |
|---------------|----------------|--------------|------------------|
|               |                | Ancho (nm)   | alto (nm)        |
| Acero H13     | 480-520        | 580-638.5    | 625.3-831.5      | 406.5-507.7 |
| Silicio 0.0 V | 47.8-56.2      | 59.17-105.45 | 222.9-335.5      | 54.28-105.6 |
| Silicio 60 V  | 346.0-379.0    | 421.6-490.7  | 471.1-520.1      | 197.6-262.2 |

An increase in the thickness of the films is determined by a voltage variation, as shown in the values found in the silicon samples, this growth is up to 75%.

Likewise, a relationship between the average roughness and thickness is precised and these values have variability between 22% and 30% for both H13 steel and silicon substrates.

The average grain size of the samples depends on the growth conditions of the film and the substrate surface. For samples in the analysis, we see in detail this relationship, as there is a grain size growth by increasing the deposition voltage this growth is 35% for the width of grain and 49% for the height of the grain.

In the Image2. Shows the average roughness, grain size and thickness of each substrate films.

![Image2](image2.png)

Imagen 2. Espesor de la películas Ti/TiN utilizando Microscopio de Fuerza Atómica (AFM) a)Sustrato de Acero H13, b) Sustrato de Silicio de 0.0 V, c) Sustrato de Silicio 60.

In the images the films deposited on H13 steel and silicon of 0.0 V substrates are displayed, it presents more uniformity because the film is smooth.

In Imagen.2 changes in the heights and widths of the samples by a voltage variation are shown. It is also observed that films with defined widths and heights are on the H13 steel substrate; this is because the steel before the deposition was polished and cleaned properly, so that the films had a better grip.
4. Conclusions.

Ti / TiN Multilayers were deposited on H13 steel and silicon samples using the cathodic arc evaporation (Cathodic ARC EVAPORATION-CAE) technique, silicon samples were deposited with a voltage variation, and H13 steel samples deposited at a temperature of 425 °C, thus obtaining an average value of film thickness, roughness and grain size, a proportionality between the voltage of deposition and growth of the multilayers was observed, the growth is 75% observed with Interferometric Microscope observed and 73% with the Atomic Force Microscope (AFM).

Depending on the conditions of film growth and the substrate surface, properties that depend on the topography were established as on the average grain size and roughness for different samples, there was an increase of the grain size, when the deposition voltage is increased this growth is 35% for the width of grain and 49% for high grain, also a relationship between average roughness and thickness of the multilayers presenting a variability between 22% and 30% for the two substrates was established.

Comparing the value of Ti / TiN film thickness on different substrates using the two characterization techniques: A difference between the values is observed, which depends on the accuracy of microscopes, resulting in an accuracy of 10nm in the Interferometric Microscope and 0.1 nm. In the Atomic Force Microscope (AFM).

5. References

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