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Determination of friction coefficient on ZrN and TiN using lateral force microscopy (LFM)

Diego F. Arias, Diana M. Marulanda, Alejandra M. Baena, Alfonso Devia *

Laboratorio de Física del Plasma, Universidad Nacional de Colombia, Sede Manizales, Carrera 27 No. 64-60, Manizales, Colombia

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Abstract

The scanning probe microscopy (SPM) technique has been used in its atomic force microscopy (AFM) and lateral force microscopy (LFM) modes, in order to characterize morphologically and to determine the friction coefficient, respectively, for coatings of TiN and ZrN thin films. The measure on the friction coefficient shows that although these films have similar physical and chemical properties, they present a difference in their friction coefficient, showing that TiN presents a higher friction coefficient than ZrN. Using X-ray diffraction (XRD), the coatings were characterized structurally. Chemical states of the TiN and ZrN films were determined by X-ray photoelectron spectroscopy (XPS).

Keywords: Titanium nitride; Zirconium nitride; Friction coefficient; Lateral force microscopy

1. Introduction

In recent years, the micro-tribology has been converted in an interesting and fundamental area due to its multiple applications in the research and development of technological applications. In the technological area, when new micro-devices are developed, it becomes an essential science in the sense of understanding the origin of friction and wear. Theories of macro-tribology are not proper to explain the tribological and mechanical behavior which occurs in micro-machines or very high precision instruments due to their low charges and nanometric scale. Many classes of contact surfaces with a relative movement consist on thin films deposited over bulk materials. However, the properties of thin films are frequently different in comparison to bulk materials. Therefore, it is very difficult the use of experimental methods and theories which are traditional to study and to explain the tribological behavior of these thin films for special use [1].

Tribological properties in nano- and micro-scale between two solid sliding surfaces have a significant importance in the performance of micro-devices such as microelectromechanical systems (MEMS), whose manufacture has provoked a strong interest in nanotribology [2], and these properties provide crucial information to understand the origin and fundamental mechanism of friction and wear [3]. Through scanning probe microscopes such as the atomic force microscope (AFM) and lateral force microscope (LFM) or friction force microscope [4], to perform an experimental approach to the nanotribological regimen has been possible. LFM has a position sensitive photodetector (PSPD) which measures the lateral and vertical deflection of the cantilever simultaneously, detecting the friction and normal forces, respectively [5].

The measure of ultra low friction coefficients through the LFM technique has been studied extensively in the last decade. This measure depends on both intrinsic and extrinsic parameters of the film. These parameters are function of the molecular structure and they can depend on the atmospheric environment and the quantity of impurities such as oxygen and humidity. The intrinsic and extrinsic parameters can interfere on the surface and in the bulk of the solid lubricant and so will interfere with the friction coefficient measure [6]. More recently, this effect has been studied by tribochemistry, relating the dependence of the surface energy and dangling bond density to friction coefficient parameters [7]. The measure of the friction coefficient through the "pin on disk" technique requires a large area from the substrate and it can represent a limitation in order to understand the fundamental mechanisms of friction [8]. The use of this new technique provides more

* Corresponding author. Tel.: +57 68745410; fax: +57 68745410.
E-mail address: adevia@epm.net.co (A. Devia).

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information about the contribution of the crystal structure through the friction information in very small areas of surface [9].

In this paper, the friction coefficients of titanium nitride and zirconium nitride are determined. In order to assess the validity of the results, the friction coefficients of highly oriented pyrolitic graphite (HOPG), mica and silicon (1 0 0) are determined and compared to results reported in literature, which employ the same measure technique.

2. Experimental setup

2.1. Sample preparation

The deposition of thin films was performed in a non-commercial cathodic pulsed arc system [10] which consists of a cylindrical chamber made of stainless steel with 6.35 mm thickness and 30 cm long per 20 cm diameter. The chamber is equipped with a vacuum system which consists of a mechanical pump and a turbomolecular pump that allows reaching vacuum levels in the order of 10^{-6} mbar. The arc is generated when discharging a bank of electrolytic capacitors (54 mF and a maximum voltage of 450 V) by means of an RL circuit ($R = 0.54\ \Omega$ and $L = 2.3$ mH) among two faced electrodes. The substrate is the anode and the cathode is the target in the system. For the films growth, stainless steel 316 as substrate is used, whose fixed size is 3 mm thickness and 1.3 mm diameter. The sample is polished previously using sandpaper starting from sandpaper 100–1500 in order to finish using diamond suspension with granulometry 0.1 μm. Later on, they are cleaned using acetone or alcohol in order to eliminate grease. Deposition conditions are shown in Table 1.

Table 1
Deposition conditions

Deposition parameter	TiN	ZrN
Target	Ti	Zr
Pressure of N_2 (mbar)	1.3	0.52
Discharge potential (V)	320	340
Electrodes separation (mm)	7	7
Time of glow (min)	20	20

2.2. Structural and chemical analysis

In order to perform the analysis of the crystalline structure, the X-ray diffraction (XRD) technique was used, through a Bruker AXS difractometer, model D8 Advance, parallel beams geometry and a graphite monochromator. The data were obtained under conditions of a 3° grazing incident, with a range of 2θ from 30° to 80°, an increase of 0.02°, and a velocity of 8 s/step.

The XPS analyses were performed using a Thermo Vg Scientific ESCALAB 250 XPS/ISS, with an X-ray source of Al Kα, with a hemispheric energy analyzer between −10 and 1200 eV.

2.3. Morphologic study and measurement of friction coefficient

The morphologic study of the surface and the friction coefficient measurement were performed in a scanning probe microscope of Park Scientific Instruments (model AutoProbe CP) in the atomic force microscopy (AFM) and lateral force microscopy (LFM) modes, respectively. All the AFM images were obtained in a scanning area of 1 μm × 1 μm. The values of root mean square (rms) roughness and grain size were derived from AFM images, which were obtained from the average of the values measured in six random areas.

For the LFM case, a standard V-shape silicon nitride cantilever with integrated square-pyramidal tip was used, whose spring constants, normal (k_N) and lateral (k_L), can be determined by different methods [11,12], because the normal spring constants, even in the most accurate commercial cantilevers on the market, have uncertainties of more than ±50% [13], and the lateral spring constants are not given at all [14]. In LFM, the knowledge of the constants is an important factor [15]. In our work, they were evaluated based on the cantilever geometry that was determined through scanning electron microscopy (SEM), whose dimensions are shown in Table 2. The friction measures were performed with a scanning area of 0.5 μm × 0.5 μm and a scanning velocity of 3 Hz, calculating the average from the friction coefficients obtained in six different areas. The friction coefficient value is obtained from the slope of the normal force (F_N) against friction force (F_L) curve between the tip and the sample. The normal force is equal to the multiplication between the signal of normal voltage from the cantilever, the normal spring constant (K_N) and the normal sensitivity factor (S_N). The normal sensitivity factor (S_N) is obtained from the slope of the lineal section in the curve of normal deflection of the cantilever (V) against the probe-sample separation.

The normal spring constant (K_N) is given by the following equation:

$$K_N = \frac{Ewt^3}{2L^3} \cos\theta \left\{ 1 + \frac{4w^3}{b^3}(3\cos\theta - 2) \right\}^{-1} \quad (1)$$

where E is the Young's module, w the width of the cantilever leg, L the cantilever length, t the cantilever thickness, b the distance from the legs to the base, and 2θ is the angle between the legs [16].

The friction force (F_L) is calculated from the multiplication between the lateral deflection signal, the lateral spring constant (K_L) and the lateral sensitivity (S_L). The lateral voltage signal is taken as the half of the value between the forward and backward scan in the friction loop. The lateral sensitivity (S_L) is propor-

Table 2
Cantilever dimensions measured by scanning electron microscopy

Legs thickness (μm)	26.4
Distance between legs (μm)	169.2
Cantilever length (μm)	162.9
Angle 2θ between legs	55
Tip length (μm)	5