



Tribological carbon-based coatings: An AFM and LFM study

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ABSTRACT

In this work some carbon-based coatings were studied by atomic force microscopy (AFM) and lateral force microscopy (LFM) techniques in order to evaluate their microstructure and friction properties at the micro and nanoscale. With this aim, four samples were prepared by magnetron sputtering: an amorphous carbon film (a-C), two nanocomposites TiC/a-C with different phase ratio ($\sim 1:1$ and $\sim 1:3$) and a nanocrystalline TiC sample. Additionally, a highly oriented pyrolytic graphite (HOPG) and an amorphous hydrogenated carbon coating (a-C:H) were included to help in the evaluation of the influence of the roughness and the hydrogen presence respectively. The topography (roughness) of the samples was studied by AFM, whereas LFM was used to measure the friction properties at the nanoscale by two different approaches. Firstly, an evaluation of possible friction contrast on the samples was done. This task was performed by subtraction of forward and reverse images and lately confirmed by the study of lateral force profiles in both directions and the histograms of the subtraction images. Secondly, an estimation of the average friction coefficient over the analysed surface of each sample was carried out. To take into account the tip evolution/damaging, mica was used as a reference before and after each sample (hereafter called *sandwich method*), and samples-to-mica friction ratios were calculated. The LFM was shown to be a useful tool to characterise a mixture of phases with different friction coefficients. In general, the friction ratios seemed to be dominated by the amorphous carbon phase, as it was impossible to distinguish among samples with different proportions of the amorphous phase (friction ratios between 1.5 and 1.75). Nevertheless, it could be concluded that the differences in friction behaviour arose from the chemical aspects (nature of the phase and hydrogen content) rather than surface characteristics, since the roughness (Ra values up to 5.7 nm) does not follow the observed trend. Finally, the Ogletree method was employed in order to calibrate the lateral force and estimate the friction coefficient of our samples. A good agreement was found with macroscopic and literature values going from ~ 0.3 for TiC to ~ 0.1 for pure carbon.

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1. Introduction

Tribology may be defined as the science that studies the phenomena taking place during movement of solids under contact. Under these conditions, a force which opposes to the movement appears (friction), which inevitably causes energetic losses. Due to that, wear of any of the materials under contact can happen. Thus, tribological properties cannot be defined according to specific materials, as may be done for mechanical properties for example [1]. The tribology deals with complex systems which depend on the sample tested, the counterbody (steel, alumina, sapphire...), environmental conditions (humidity, pressure, ...), test conditions (speed, load...) etc. Besides, in real (macroscopic) terms, the fric-

tion between two materials is dependent on the surface characteristics of the materials in contact (roughness, asperities) and the interaction between them (surface modification, chemical reactions, etc.) [2,3]. Possible situations can be the reactions between the counterbody and the sample (adhesion, transfer films) or between one or both of them and the surrounding atmosphere (corrosion, adsorption). Moreover, in many cases the tribological system becomes more complex due to the presence of debris of the materials under contact due to wear processes (*third bodies*), which can also be modified by chemical reactions. Of course, if dealing with processes including liquid lubricants the situation becomes even more complicated [4]. Carbon-based materials play an important role in mechanical and tribological applications [5]. Depending on the deposition method and the control of several variables, such as the amount of hydrogen or the sp^2/sp^3 ratio [6], these materials can show optimal hardness and low values of friction and wear under atmospheric pressure or vacuum conditions [2]. Moreover, if nanocrystals of a second phase are added, a so-called “nanocomposite” is obtained, which can show even better mechanical properties, such as increased hardness or toughness

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[7–9], while the tribological aspects are maintained or even improved.

The aim of this paper was to study the frictional properties of carbon-based materials at the micro- and nanoscale by using lateral force microscopy (LFM), whose probe size is in the order of magnitude or less than the roughness of the analysed surface. The small contact radius and applied load conditions (few nN) enabled to scan the surface of samples preventing undesired friction-induced surface modification phenomena, reducing the influence of the roughness and, consequently, to obtain the friction properties of the material. This goal was accomplished measuring the friction contrast and average lateral force values over the scanned area of the nanocomposite films. In the latter case, the influence of topography (roughness) and the hydrogen presence in the carbon matrix were investigated. The Ogletree method [10] was also attempted for calibrating the lateral response of the tip.

2. Experimental

2.1. Measurement of friction properties by LFM

The basis of the measurement of friction forces by means of an atomic force microscope is summarized in Fig. 1. Whereas the topographic information is obtained from the vertical displacements of the tip (typically, the displacement of the z-axis piezo), the frictional properties lead to the lateral torsion of the cantilever while scanning the sample in both directions (registered by the laser signals). However, as shown in Fig. 1, there are two possible causes for an increment of the lateral force on the tip: (i) the appearance of a topographic event with higher slope, or (ii) the existence of a material with higher friction coefficient. In order to evaluate the friction contribution to the total lateral force, the sub-

traction of lateral forces profiles recorded in both directions, forward and backward, is needed. By this procedure, the lateral forces originated from topographic effects should be cancelled (though some “topo” effects related to the “impact” of the tip when reaches an obstacle cannot be totally avoided) [11]. Notice that when a sum operation is performed instead of a subtraction the friction-related forces are cancelled and only topographic effects are obtained. This is an important concept that can be useful for calibration of the cantilever lateral spring constant by the Ogletree method [10].

The normal force (F_N) over the surface can be calculated according to the following equation:

$$F_N(\text{nN}) = [F_A(\text{nA}) + A(\text{nA})] \times \frac{k(\text{nN/nm})}{SR(\text{nA/nm})} \quad (1)$$

where F_A and A are the applied normal force and the adhesion expressed in nanoamperes, and k and SR are the spring constant and the sensor response of the tip, respectively. The F_A is a known parameter for each experiment, k is provided by the tip manufacturer and both A and SR can be determined by the software provided by the AFM manufacturer from load-displacement curves [12].

To get information about the lateral force over the tip (in nN) it is necessary to transform the electrical signal of the lateral force (in nA) by means of a lateral force conversion factor (β) which can be obtained by calibration of the lateral response of the system. This in situ calibration is not an easy procedure, and many options can be found in the literature (see as an example [13–17]). Most of them are based on the method developed by Ogletree [10], which employs a standard grating with well-defined profiles (slopes). Although for comparison among samples it may be sufficient to measure the lateral forces by the electrical response (in nA), the application of this method to calibrate the lateral deflection of the tip was also attempted.

In order to explore the friction contrast of the samples, the samples were scanned in contact mode and lateral force images (forward and backward) were recorded and subtracted after appropriate alignment. The cantilever scanned perpendicular to its long axis in order to obtain the maximum lateral deflection. This procedure yielded to a lateral force mapping where a friction contrast was identified by different colours. In some cases, the appearance of shifts between forward and reverse images due to non-linearity of piezos or by thermal effect, required the removal of a data portion and caused a non-square subtraction images [18].

The procedure for the estimation of the average friction coefficient is more complex. In order to get reliable information, the measurement of the lateral forces at different load is needed. In order to fulfil this requirement, the whole image was divided into 10 segments where the normal load was progressively increased (see Fig. 2). To get broader range of applied load, in cases where the value of adhesion was big enough, the additional force against the adhesion was also considered. This can be observed in the first step of Fig. 2b, whose value of total load is smaller than the adhesion value. From the subtraction of each pair of segments, one datum of lateral force was calculated, yielding thus 10 pairs of data “normal load-lateral force” for each image. The slope of the plot of lateral force versus normal load is proportional to the friction coefficient of each sample.

The same cantilever was used for all the measurements in order to avoid another variable than could invalidate the comparison among the frictional properties of the different samples. However, from sample to sample the tip can be deteriorated or its shape altered. To take into account this fact, one stable and reproducible internal standard (mica) [19] was measured between each two samples (this procedure has been therefore so-called “sandwich

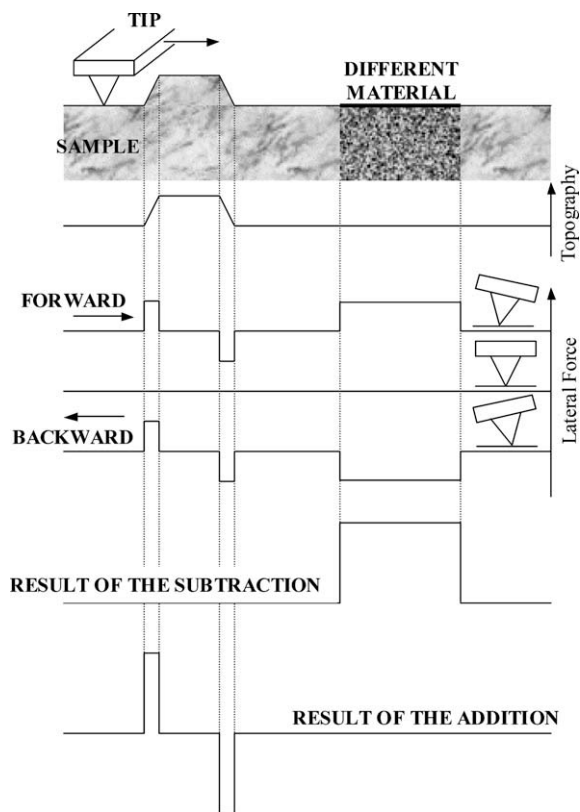


Fig. 1. Diagram of the tip behaviour (normal and lateral forces) for complete surface scanning cycle including subtraction and addition of both lateral force profiles.

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