Nanofriction of diamond coatings obtained by flame process

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Abstract. With its noteworthy physical and chemical properties, diamond is expected to find many applications in various industrial fields, particularly with the expansion of the new nanotechnologies. In order to optimise their use in these specific conditions, it is essential to better understand the very complex tribological behaviour of diamond coatings. This latest is studied from a macroscopic point of view, but essential facts which occur at a nanoscopic scale are still not well known. The mechanisms involved not only in the nucleation and growth of the diamond crystals, but also in the nanofriction of the coatings, must be understood. One way to reach this purpose is to analyse the crystals morphology and behaviour at a very small scale: atomic force microscopy (AFM) seems to be a suitable technique for this work. The crystals morphology is initially studied in tapping mode; the first observations indicated that secondary growth happened on the facets. Experiments in contact mode are then carried out: the observed behaviours are different on a facet, on a crystal, or on a part of the coating. The load applied to the tip seems to have an influence too as the friction force increases when the contact load becomes higher; moreover, these variations are clearly linked to the topography of the scanned surfaces. The purpose of this nano-experimental work is to get information to better understand the friction properties of the diamond coatings at a nanoscopic scale, and to establish, if possible, a potential correlation between the behaviour noticed at the macroscopic scale, and the one obtained with AFM measurements.

1. Introduction

The majority of the friction and wear tests realised in laboratory are carried out at a macroscopic scale, on devices allowing the application of normal loads on the order of several Newtons. But the wear rates of most of the components used in engines or machines are around the nanometer per hour, which is much lower than what can be simulated in laboratory. This is particularly the case for applications in nanotechnologies; it becomes then a necessity to develop methods allowing friction and wear measurements at a smaller scale, namely the nanoscopic scale. Atomic Force Microscopy stands thus out because of its abilities of studying nanoscopic properties of materials. Hard carbon coatings, which are promising films for nanoscopic applications, were tested thanks to this technique for few years: topography descriptions and roughness measurements were performed, through the tapping mode, on tetrahedral amorphous carbon films [1], on DLC films [2] or on diamond coatings obtained by Chemical Vapour Deposition [3].

The tribological behaviour of these films was also evaluated: scratch experiments realised on DLC coatings with a Berkovich diamond tip [4] indicated that the friction coefficient is modified when the contact load varies from 10 to 300nN. The same tests were carried out on carbon-doped boron nitride, and the friction and wear damage were reported to be strongly dependent on the normal load [5]. This load dependence of the friction coefficient was observed as well when a diamond tip slid against silicon
oxide surfaces, for contact loads lower than 0.4µN [6]: this phenomenon was attributed to the pronounced influence of the attractive forces in this loads range. Amorphous carbon films (a-C) sputtered on Si(100) substrates were studied by Surface Force Microscopy with a Berkovich diamond tip (contact load : 10-1200µN) [7]: an increase of the normal load leads first to a decrease of the coefficient of friction to a minimum value, then subsequently to an increase to a maximum value, after that it remains constant or slightly decreases; adhesion seems to be the dominant friction mechanism for loads lower than 50µN, whereas for the intermediate and high load ranges, both adhesion and ploughing occur. Measurements carried out by Friction Force Microscopy, with a diamond tip (normal load : 0-50µN), on polished natural diamond revealed that the friction force linearly increases with higher applied normal loads, and no load dependence on the friction coefficient was noticed [8]. The Lateral Force Microscopy is also proved to be a very interesting technique for friction coefficients measurements in the case of DLC films obtained by magnetron sputtering [9]: the contribution of contamination and/or of chemical bonds surface terminations can be studied as a function of the friction coefficients distribution.

Considering the possibilities of the Atomic Force Microscopy indicated by the previous work, the nano-tribological behaviour of diamond coatings (obtained by combustion flame process) is studied here, and more particularly the role of the contact load during friction.

2. Experimental device

2.1. The combustion flame process

The diamond coatings used in this study are obtained by the combustion flame process, a technique initially devised by Hirose and Kondoh [10] to remedy the financial and materials drawbacks of the Chemical Vapour Deposition. This method is based on the fact that a flame can be considered as a plasma in air, that means that it can play the role of a reaction chamber which gives the heat necessary to the creation of the radicals involved in the formation and the growth of diamonds. From a structural point of view, the main difference between these diamonds and those synthetized by CVD lies in the fact that the dangling bonds are here mainly saturated by oxygen, instead of hydrogen terminations. The advantages and drawbacks of the flame process as well as the conditions of deposition are given in [11]. The diamond coatings used for the AFM measurements are a mixing of {111} and {100} oriented crystals, deposited on triangular tungsten carbide plates; their grains size varies between 2 and 8 µm. Raman spectroscopy analyses revealed these films are constituted of sp³ hybridized carbon (lack of graphite).

2.2. AFM measurements

The measurements were carried out on a Nanoscope III–Digital Instrument machine, in the ambient (20°C, 40%RH); the first series was realised in tapping mode, with a Si₃N₄ cantilever, at a scan frequency equal to 1Hz (areas : 1x1µm² and 2x2µm²). Then the contact mode is used to determine the nanotribological properties of the coatings; the scan frequency remains the same, the selected cantilever is a Si₃N₄ triangular one with a stiffness of 0.58 N/m. The size of the scanned zones varies from 800x800nm² to 30x30µm².

3. Results and discussion

3.1. Tapping mode

This mode is first used in order to observe the topography of the films and the crystals morphology. As a mixing of {111} and {100} oriented crystals, the coatings do not present a homogeneous surface: there are different growth directions, twins and crystals overlapping are noticed (figure 1a). Moreover, crystalline structures due to a renucleation are observed; even if this so induced secondary growth is quite common in the film, it seems that this phenomenon could only developed on preferential facets (figure 1b): some surfaces of the crystals are subjected to this secondary growth, whereas the others remain smooth and unmarked. This could be linked to the surface energy which is probably different from one facet of the crystal to another. A similar fact is observed by Liu and al on
diamond films obtained by CVD [12]: the growth rate along a particular orientation is faster than on other facets, probably because of the prevalence of the pyramidal morphology in these films.

**Figure 1.** AFM images (phase) of the diamond coating (a) twins and crystals overlapping; (b) secondary growth on preferential facets

3.2. Contact mode

The measurements in this mode are realised on a *crystal facet* (800x800nm²), on a *crystal* (1x1µm²), and on *surfaces* of various sizes (from 20x20µm² to 30x30µm²), under various contact loads. On each selected zone, the Trace-Minus-Retrace (TMR) values are recorded as a function of the time, for every applied load (scanning direction: from the top to the bottom). In this preliminary study, the observations are qualitative and not quantitative.

The tests realised on small areas, that means on the *facet of a crystal* and on a *crystal* are presented on figures 2 and 3.

**Figure 2.** AFM measurements on a *crystal facet* – (a) topography image; (b) friction-retrace image; (c) TMR values as a function of the scanned distance, for different contact loads

**Figure 3.** AFM measurements on a *diamond crystal* – (a) topography image; (b) friction-retrace image; (c) TMR values as a function of the scanned distance, for different contact loads
The diagrams on figures 2b and 3b indicate that:

- an increase of the contact load leads to higher friction,
- the variations of the TMR at 0V, 1V and 2V present the same appearance: the TMR signal seems to follow the topography of the scanned surface.
- the topographical details are more visible when the contact load is bigger: the “sensitivity” of the TMR measurement seems to be more important at higher loads.

**Figure 4.** AFM measurements on the diamond coating on (a) 30x30µm²; (c) 25x25µm²; (e) 20x20µm² (friction-retrace image) - (b,d,f) TMR values as a function of the scanned distance, under different contact loads, for these various magnifications; section is taken along the red line on the corresponding friction image

Measurements on surfaces of bigger size are carried out as follows: a 30x30µm² area is scanned (figure 4a), then a zoom of 25x25 µm² is made on the left bottom area of the previous surface (figure
4c), and finally an other magnification is realised on the right bottom area of the last surface, leading to a 20x20µm² scanned surface (figure 4e). Here again, the friction becomes more important when the contact load is increased, and the previous observations are still valid (figures 4b,d,f).

In order to try to analyse these results in a quantitative way, the TMR plotted on a same area, but at various contact loads and for the three magnifications (red line on figure 4) are studied (figure 5). The TMR values clearly linearly increase when the contact load varies from 0V to 4V for the three magnifications (the correlation factors are very good in the three cases) ; however, the values obtained in the biggest area (30x30µm²) are slightly higher than those noticed for the 25x25µm² and 20x20µm² zones which are quite similar. This can be linked to the fact that these various analysed surfaces are scanned at the same frequency : optimal friction results can be obtained with an adequate combination of the scan frequency and of the magnification, more particularly here, for these rough coatings.

**Figure 5.** Influence of the contact load on the friction properties of the diamond coating (red line)

4. Conclusions

Diamond coatings obtained by flame process were observed by AFM, first in tapping mode in order to determine their topographical particularities, and also in contact mode to study their tribological properties at a nanoscopic scale. Observations in tapping mode indicate that renucleation and secondary growth occur on the crystals facets. When the contact load is increased during the measurements in contact mode, the friction increases too ; moreover, the TMR signal seems to follow the topography of the scanned surface.

Quantitative measurements must be carried out in order to complete this preliminary work, so as to better understand the friction properties of these diamond coatings at a nanoscopic scale.

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