Tribological behaviour of graphite powders at nano- and macroscopic scales

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Abstract. With its high resistance, good hardness and electrical conductivity in the basal plans, graphite is used for many years in various tribological fields such as seals, bearings or electrical motor brushes, and also for applications needing excellent lubrication and wear-reducing properties. But thanks to its low density, graphite is at the moment destined for technologies which need a reducing of the weight combined with an enhancement of the efficiency, as it is the case in aeronautical industry. In this context, the friction and wear of natural (named graphite A) and synthetic (called graphites B and C) powders were evaluated, first at the macroscopic scale when sliding against steel counterfaces, under various applied normal loads. Scanning Electron Microscopy and AFM in tapping mode were used to observe the morphological modifications of the graphites. It is noticed that an enlargement of the applied normal load leads to an increase of the friction coefficient for graphites A and C; but for the graphite B, it seems that a “limit” load can induce a complete change of the tribological behaviour. At the same time, the nano-friction properties of these powders were evaluated by AFM measurements in contact mode, at different contact loads. As it was the case at the macroscopic scale, an increase of the nano-contact load induces higher friction coefficients. The determining of the friction and wear mechanisms of the graphite powders, as a function of both their intrinsic characteristics and the applied normal load, is then possible.

1. Introduction
As graphite is a multipurpose material which is used in metallurgic industry, transport or advanced technologies, its tribological behaviour was the subject of many works. The influence of the environment on its friction and wear properties was more particularly studied: it was observed that if its friction coefficient can be high in vacuum, it is greatly reduced when working under oxygen or water vapour [1,2]. This decrease of the friction coefficient was attributed to the passivation of the dangling bonds created during the sliding [3]. But if the environment plays a significant part in the tribological behaviour of graphite, the applied normal load is also of great importance.

This study lies within the framework of the previous context as it focuses on the influence of the applied normal load, on the tribological behaviour of graphite powders, natural or synthetic, which are intended to be used in brakes. The objective of this work is the elaboration of a classification of these powders, as a function of their friction performances under various applied normal loads, in order to determine the most adapted graphite for the application needed by the manufacturer supplier of these powders. In order to select the graphite with the optimal friction properties, tests were first realised at a macroscopic scale, on a classical pin on disc tribometer, with steel discs as counterfaces. Then measurements were carried out with Atomic Force Microscopy, on contact mode, to study the nanotribological behaviour of the three graphites.
2. Experimental device

Graphite pins

Three powders were tested: the graphite named A is a natural one whereas graphites B and C are synthetic; the grains size of these materials is 75 µm. These powders are compacted with a press to get small graphite pins of 5 mm diameter and 3 mm height.

Counterfaces

XC38 steel discs (44 mm diameter) are subjected to a polishing process in order to reduce their roughness to a value lower than 10 mm (Hv : 220 kg.mm⁻²).

Experimental conditions

The macroscopic experiments were realised on a home-made pin on disc tribometer, at the ambient during 40 min. The sliding speed was set at the constant value of 0.015 m.s⁻¹ (10 rpm), and various normal loads were applied: 30.4, 40.4, 45.4 and 50.4 N.

AFM measurements were carried out on a Nanoscope III – Digital Instrument machine, in contact mode, with a Si₃N₄ triangular tip (stiffness: 0.58 N/m). The scan frequency is 1 Hz, the size of the scanned zones varies from 2x2µm² to 5x5µm².

3. Results and discussion

3.1. Macroscopic tests

The friction coefficients obtained at the end of the tests are in the range of 0.10-0.16 (table 1), which is in accordance with the literature [4].

|            | 30.4N | 40.4N | 45.4N | 50.4N |
|------------|-------|-------|-------|-------|
| Graphite A | 0.10  | 0.12  | 0.14  | 0.15  |
| Graphite B | 0.11  | 0.15  | 0.13  | 0.14  |
| Graphite C | 0.10  | 0.12  | 0.13  | 0.14  |

The friction coefficients of graphites A and C increase when the applied normal load becomes higher; in the case of graphite B, it seems that there is a critical load (45.4N) which induces an opposite variation of the friction coefficient.

The behaviour of graphites A and C is a typical example of the linear increase of the friction coefficient with the normal load; this evolution can be explained as follows: the application of higher normal loads leads to more important local normal forces on the contact asperities; these latest are then distorted, even flattened. The contact area becomes consequently greater, inducing thus higher friction coefficient. The decrease of the friction coefficient observed for graphite B after sliding under 45.4N could probably be attributed to the combined influence of many parameters; one of them could be the hardness of the graphite. As a matter of fact, graphite B is the softer one, as its hardness is two times lower than those of graphites A and C; this has direct consequences on the transfer, induced by the sliding of the graphite pin on the steel disc, and finally on the friction. A study by Scanning Electron Microscopy of the topography of the transferred films formed under those various experimental conditions confirmed that the transfer obtained after the friction of graphite B, under the critical load, is quite different from the others, and favoured a decrease of the friction coefficient.

3.2. Nanoscopic tests

In contact mode, the AFM cantilever scanned the surface of the studied sample following a direction which is perpendicular to its axis. The characteristic value of this AFM mode is the TMR, that means “Trace Minus Retrace” which corresponds to the difference between the amplitude of the trace of the tip and the one of its retrace (figure 1).
This TMR is proportional to the friction force, and is given in volt. The force with which the tip is in contact with the surface (that corresponds to the applied normal load in macroscopic measurements) is also given in volt. It is possible, with a specific protocol of calibration, to obtain the friction coefficient ($\mu = \text{friction force} / \text{normal force}$), but this was not done here as it is only a preliminary study.

The selected areas for the friction measurements are composed of basal graphitic planes of similar size (figure 2).

**Figure 2.** AFM images of graphite (a) A ; (b) B ; (c) C, in contact mode under 3V (the left image is the heigh one, and the right image is the friction-retrace one)
Figure 3. TMR values (mV) as a function of the scanned distance under various contact loads, for graphite (a) A; (b) B; (c) C
Even if it is not possible, with this kind of results, to realise a precise quantitative study, general trends can be drawn. According to the diagrams of figure 3, it appears that the friction of graphite B seems to be more important than those of graphites A and C: the values of the TMR observed for B are more than three times higher than the ones of graphites A and C (these latest vary in the same range of values). Of course, the fact that the scanned surface for graphite B is 5x5µm² (instead of 2x2µm² for A and C) can play a part in these observed variations: the z range on this area is 600 nm (it is only 40nm and 50nm for graphites A and C), which indicates that the roughness is here more important; this can contribute to explain the higher friction of graphite B. However, even on the “flat” zone of graphite B, the TMR remains bigger than the values noticed on the smooth surfaces scanned on graphites A and C.

Even if graphite B presents a specific tribological behaviour at both nano- and macroscopic scales (compared with graphites A and C), the variations of the friction are quite different at the two scales, and the “nano-macro” link is not evident.

On the other hand, graphite B is the only one for which the increase of the TMR in clearly linked to the application of higher contact loads. For graphites A and C, the influence of the normal load is less markedly observable; it can be noticed, for example, that graphite A under a contact load corresponding to a voltage of 2V presents TMR values which are not far from those observed at 3V (sometimes they are even higher). The same phenomenon is noticed for graphite C.

It appears then that graphite B is distinguished from the two others, whereas graphites A and C have quite similar behaviours.

4. Conclusions

In this work, the tribological behaviour of graphite powders, natural and synthetic, was studied in order to classify them, and select the most appropriate for specific applications (braking,…). Macro and nanoexperiments were carried out at the same time. Tests realised on the pin on disc tribometer highlighted the particular behaviour of graphite B (existence of a critical load), whereas graphites A and C showed friction coefficient which classically increases when the applied normal load becomes higher. The influence of the contact load, at the nanoscopic scale, is less evident to prove, except for graphite B.

It seems that the tests which were realised at the moment, are not sufficient to clearly distinguish and classify these graphite powders; other experiments must be envisaged, both at the nano and macroscopic scales, to refine and better understand the tribological behaviour of these materials.

References

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