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Friction and wear behaviour of silicon carbide/graphene composites under isooctane lubrication

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Abstract

The tribological performance of silicon carbide (SiC)/graphene nanoplatelets (GNPs) composites is analysed under oscillating sliding tests lubricated with isooctane, looking to explore their potential as components for gasoline direct injection (GDI) engines. High graphene filler contents (20 vol.% of GNPs) are required to substantially reduce the friction coefficient of SiC ceramics, attaining decreases on friction up to 30% independently of the applied load. For all materials and testing conditions a mild wear regime is evidenced. SiC/20 vol.% GNPs composite also enhances the wear resistance up to 35% at low load, but the addition of GNPs produces a deleterious effect as the load augments. The tribological behaviour depends on the formation and destabilization of a solid lubricant carbon-based tribofilm and strongly correlates with the mechanical properties of the tested materials.

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1. Introduction

The automotive industry needs to accomplish the new energy and eco-friendly targets trying to reduce both the fuel consumption and the harmful emissions to the atmosphere, the latter objective requested by the European emission standards Euro 6 [1]. Gasoline direct injection (GDI) engines can fulfil those goals but pressure levels up to 80 MPa are required [2], which jointly with the poor lubricity of gasoline compel to replace the metallic sliding components by more wear resistant materials such as advanced ceramics. As a matter of fact, Pfister et al. [3] found a good performance with high mechanical efficiency using a self-mated textured SiC cam/pusher system within a high-pressure 3-piston prototype pump for gasoline. Besides, the addition of carbon nanotubes (CNTs) into silicon nitride ceramics (Si_3N_4) to produce composite cams also exhibited an excellent mechanical efficiency and low cyclic variations and noise emissions when tested against steel pushers [4]. It seems that CNTs provided a better distribution of the local stresses beneath the tested surfaces and promoted lubrication by tribochemical reactions with the steel pushers.

At present, the incorporation of graphene nanofillers to ceramic materials has mostly showed a clear benefit in the enhancement of their tribological properties for all the studied ceramics (see [5] for a review). In this way, reductions in the friction coefficient between ~10 to 70% and improvements in the wear resistance of ~50-100%, as compared to their respective monolithic reference ceramics, have been achieved [5].

Most of these studies have been carried out under dry testing conditions; whereas scarce works on lubricated systems have been reported [6,7]. In this way, Si_3N_4 /graphene composites tested under isooctane [6] and water [7] lubrication showed a similar tribological trend, i.e., the addition of graphene fillers led to an enhanced wear

1 resistance of the Si_3N_4 ceramics of ~ 60%, while the friction response was hardly
2 improved.
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5 In general, the better tribological response of ceramic/graphene composites is
6 commonly explained by the formation and stabilization of an adhered carbon-based
7 protective tribofilm to the worn surface and, under severe testing conditions, also to the
8 higher fracture toughness exhibited by the composites that would limit the material
9 removal. In the case of silicon carbide (SiC) ceramics, one of the most demanded
10 engineering ceramics due to its excellent combination of properties, like good
11 mechanical strengths at elevated temperature, high thermal conductivities, and great
12 oxidation and wear resistances [8], only one study devoted to the tribological properties
13 of SiC/graphene composites has been found [9]. In that work, some of the present
14 authors established that the wear resistance of SiC ceramics under dry testing conditions
15 significantly enhanced (72%) with the addition of 20 vol.% of graphene nanoplatelets
16 (GNPs) due to the presence of a wear protecting tribofilm, which was mainly formed by
17 pulled-out, exfoliated and crushed GNPs during the sliding motion.
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20 Based on these previous works, here we present for the first time the tribological
21 properties of SiC/GNPs composites under oscillating sliding tests lubricated with
22 isooctane. This configuration simulates the conditions of high-pressure pumps for GDI
23 engines. The friction coefficient (μ) and the wear resistance of the composites have
24 been determined as a function of the GNPs content, analysing in detail the wear
25 mechanisms and the role played by the mechanical properties of the tested materials on
26 their tribological responses.
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2. Materials and Methods

Four different materials were fabricated: a reference monolithic material -labelled as SiC- and three SiC-based composites containing 5, 10 and 20 vol. % of GNPs (type N006, Angstrom Materials Inc.), which were identified as SiC5GNP, SiC10GNP and SiC20GNP, respectively. The experimental procedure is described elsewhere [9]. In brief, SiC/GNPs powders were processed by mixing and sonicating two isopropyl alcohol suspensions independently prepared, one containing GNPs dispersed using a sonication process and another one containing an attrition milled ceramic slurry comprising β -SiC powders (BF-17A, H.C. Starck) plus 5 wt.% of Y_2O_3 powders (Grade C, H.C. Starck) and 2 wt.% Al_2O_3 powders (SM8, Baikowski Chimie). The dried and sieved powder compositions were, afterwards, spark plasma sintered (SPS, Dr. Sinter, SPS-510CE) under vacuum atmosphere (4 Pa) at 1800 °C for 5 min, applying an uniaxial pressure of 50 MPa. Fully dense ($\geq 99\%$ of the theoretical value) disc specimens of 20 mm diameter x 3 mm height were obtained.

All composites exhibited a good dispersion of GNPs within the ceramic matrix, the nanoplatelets being preferentially oriented with their basal plane perpendicular to the SPS pressing axis. As an example, Figure 1a illustrates this GNPs alignment in the fracture surface of the composite containing the largest amount of graphene fillers (SiC20GNP). It is important to remark that the monolithic SiC, with no filler additions, contains ~3-4 vol.% of graphene multilayers in-situ grown during the SPS process of the ceramics [10]. In order to elucidate the role played by the GNPs in the tribological behaviour of the SiC composites, all materials were processed to achieve comparable matrix characteristics; in particular, similar matrix grain size ($\sim 0.6 \mu m$) and aspect ratio (1.4). The mechanical characterization of the composites was previously reported [9], and Table 1 summarizes some of the main mechanical parameters.

Figure 1. a) SEM micrograph of the fracture surface of SiC20GNP composite and b) scheme of the tribotest configuration.

The specimens were polished to obtain a mean roughness (R_a) value below 10 nm (Table 1), which was measured with a stylus profilometer (Dektak XT, Bruker). Then, they were ultrasonically cleaned, first in acetone for 2 min and, afterwards, in ethanol for 2 min, and dried with compressed air. In order to simulate the tribological conditions in GDI systems, linear reciprocating ball-on-plate tests were carried out with a UMT3 tribometer (CETR) under lubrication with isooctane. The balls were commercial Si₃N₄ spheres (10.3 mm diameter, SN101C, Saint Gobain) and the plates the developed materials. All tests were performed on the surface parallel to the basal plane of the GNPs, as shown in Fig. 1b, using applied loads ranging from 50 to 180 N (Hertzian pressures in the range of 2.1-3.3 GPa at the very beginning of the tests), stroke length of 2.5 mm, frequency of 20 Hz ($0.1 \text{ m} \cdot \text{s}^{-1}$) and total sliding distance of 360 m. The experiments were conducted at $25 \pm 3 \text{ }^\circ\text{C}$ and a relative humidity below 20%.

A stylus profilometer (Dektak XT, Bruker) was employed for measuring three 2D wear track profiles, as stated the ASTM G133 standard [11]. Friction coefficient, μ , was continuously registered during the tests, estimating the steady-state (μ_{ss}) value within

the 200-360 m interval. The wear volume of the plates (W_V) was assessed using the following equation:

$$W_V = \Delta x \cdot A + \pi \cdot \frac{d^2}{8} \cdot W_l \quad (1)$$

where Δx is the nominal stroke, A is the cross section area beneath the 2D curves, and d and W_l are the width and depth of the wear tracks, respectively. Eq. (1) contains the sum of two terms, one according to the recommendations of ASTM G133 standard, which only considers the central zone of the wear tracks, and another one that estimates the W_V in the two edges of the track [12]. The wear rate (W_R) was estimated as follows:

$$W_R = \frac{W_V}{F_N \cdot l} \quad (2)$$

where F_N is the normal load and l is the total sliding distance. All the tribological data shown in this work represent the average of, at least, three valid tests.

Table 1. Mechanical [9] and roughness parameters of the different materials: Young modulus (E), hardness (H) fracture toughness (K_{IC}), mean roughness (R_a).

Material	GNPs (vol.%)	E (GPa)	H (GPa)	K_{IC} (MPa·m^{1/2})	R_a (nm)
SiC	0	506 ± 46	27.6 ± 1.0	3.2 ± 0.4	3.8 ± 0.8
SiC5GNP	5	310 ± 23	24.3 ± 2.1	4.8 ± 0.4	6.5 ± 0.6
SiC10GNP	10	233 ± 38	14.8 ± 3.1	5.9 ± 0.4	8.0 ± 2.0
SiC20GNP	20	120 ± 28	9.8 ± 1.1	4.1 ± 0.1	9.0 ± 2.0

Scanning electron microscopy (SEM, TM1000 and S-4700 models, Hitachi)

observations were done on both unworn and worn surfaces. Micro-Raman spectroscopy

(Alpha3000 WITec GmbH) was also employed to characterize the tested plates using

the 532 nm laser wavelength excitation on scanned maps of $50 \times 50 \mu\text{m}^2$ (25×25 pixels), recording one spectrum per pixel and 1 s of acquisition time.

3. Results and discussion

Friction data of the different tested materials are shown in Figure 2. From the dynamic evolution (Figs. 2a-c) and the steady-state values (Fig. 2d), it is clear that only the composite with the highest GNPs content (SiC20GNP) exhibited a substantial reduction in the friction coefficient (~ 0.12) as compared to the reference material (~ 0.17), reaching a decrease of about 30% independently of the applied load (Fig. 2d). This improvement in the friction response is at the high end of the data range reported for other ceramic/graphene composites (0-37%) [5], with the unique exception of one work where $\text{B}_4\text{C}/6 \text{ wt.}\%$ GNPs composites tested in air reached maximum reductions on friction of 66% [13]. In contrast, lower GNPs additions (SiC5GNP and SiC10GNP) did not significantly modify the friction behaviour of SiC ceramics, showing similar μ_{ss} values than those attained for SiC (0.16-0.19), which kept almost constant with the load. Interestingly, a similar behaviour was previously reported by some of the present authors on Si_3N_4 ceramics containing 4.4 vol.% GNPs tested under the same isooctane lubricated conditions [6]. Therefore, at first glance, it seems that under the current testing conditions, large graphene filler contents (well above 10 vol.%) are required to enhance the friction response of these tribosystems during the whole process. Furthermore, the present results evidence for the first time that the incorporation of graphene fillers can considerably reduce the friction of ceramics under external lubrication (isooctane or water).

Figure 2. Dynamic friction (μ) evolution at: a) 50 N, b) 100 N and c) 180 N, and d) steady-state friction coefficient (μ_{ss}) for the different tested materials.

In terms of the wear behaviour (Figure 3), the wear resistance of SiC ceramics improved at 50 N in about 35% when adding 20 vol.% of GNPs (SiC20GNP), reaching minimum W_V and W_R values of $1.5 \times 10^{-3} \text{ mm}^3$ and $8.3 \times 10^{-8} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, respectively.

However, composites with lower GNPs contents exhibited an almost negligible improvement in the wear resistance (just 13% for SiC5GNP) or even produced a detrimental effect ($\sim 30\%$ less resistant for SiC10GNP) as compared to the reference material. As the applied load augmented, W_V and W_R progressively increased with the amount of GNPs, SiC being up to one order of magnitude most wear resistant than SiC20GNP. Notwithstanding, it should be mentioned that considering all tested materials and the whole range of applied loads, W_R values were in the order of 10^{-7} to

$10^{-6} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, which is characteristic of a mild wear process. These values are far beyond the typical values of tribotechnical systems [14], allowing SiC/GNPs composites suitable for being part of components in automotive systems.

Figure 3. a) Wear volume (W_v) and b) wear rate (W_R) of the plates as a function of the applied load.

The wear tracks were studied by SEM and micro-Raman spectroscopy to identify the wear mechanisms occurred in the different materials (Figures 4-6). In the latter case, the worn surfaces were analysed following a similar procedure as described in a previous work [9]. In brief, the distribution of SiC- and carbonaceous-rich areas before and after the tribotests were revealed by creating false coloured Raman maps where the most intense Raman bands recorded for SiC and GNPs in the spectra (Fig. 5a) were merged. In particular, the SiC transverse optical mode -TO- at $\sim 796 \text{ cm}^{-1}$ (coloured in blue in the Raman map) and the carbon G-band at $\sim 1591 \text{ cm}^{-1}$ (coloured in red) were selected.

SEM observations evidenced that while the worn surfaces for all materials appeared almost imperceptible when tested at 50 N due to their lower W_v values and very smooth surfaces (Table 1), an increment in the applied load allowed distinguishing some wear

phenomena (Fig. 4). In fact, some abrasion grooves jointly with microfracture and grain pull-out were identified in all materials. A tribolayer was not observed on the SiC, SiC5GNP and SiC10GNPs surfaces but it was clearly visible in the SiC20GNP one.

Figure 4. SEM micrographs of the worn surfaces tested at 180 N for: a) SiC, b) SiC5GNP, c) SiC10GNP and d) SiC20GNP. The arrows point wear phenomena.

From the Raman point of view, similar maps were observed for SiC and SiC5GNP when comparing their respective untested and 180 N tested surfaces (Figs. 5b,c). As it was previously mentioned, the reference material contains ~3-4 vol.% of graphene multilayers in-situ grown during the SPS process (red features in the Raman map). Raman data would confirm the absence of adhered debris on the tracks for both materials and, hence, of tribofilm formation, as SEM observation also pointed out (Figs. 4a,b). The intensity ratio between Raman D and G-bands (I_D/I_G), commonly employed as a tool to estimate the crystallinity degree of the graphene species, in both materials

slightly increased after the tribotests, from 0.35 to 0.69 for SiC, and from 0.28 to 0.74 for SiC5GNP. These data would mean that graphene sheets suffered certain damage during the sliding tests, as they could be pulled-out and crushed to some extent. However, they were not able to form a debris layer, being probably discharged out of the wear track.

Figure 5. a) Micro-Raman spectrum of the untested SiC20GNP composite showing the SiC (TO and LO) and GNP (D, G and 2D) bands. b-d) False coloured Raman maps for

1 untested (left column) and 180 N (right column) tested surfaces corresponding to: b)
2 SiC, c) SiC5GNP and d) SiC20GNP. Features in blue and red are ascribed to SiC and
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4 GNP, respectively. I_D/I_G values are also included within the maps.
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11 In the case of the composite with the largest GNPs content (SiC20GNP), the situation
12 was completely different and the analysis of their Raman maps revealed three main
13 results (Fig. 5d): first, many red-coloured carbon-rich features turned to purple colour
14 after the tribotests, which would be associated to a mixture of SiC- (in blue) and
15 graphene (in red)-based debris; secondly, the area coloured in red/purple considerably
16 increased at 180 N as compared to the untested surface; and, finally, I_D/I_G significantly
17 augmented from 0.23 (unttested) to 1.20 (tested at 180 N), confirming the high
18 amorphization level of the GNPs into carbon-based debris.
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31 Micro-Raman results jointly with the SEM analysis suggest as the main wear
32 mechanism the successful tribofilm formation on the SiC20GNP surface during the
33 sliding tests with a large contribution of the carbon-based debris as a result of the
34 pulling-out, cracking and smashing of the GNPs. The sequence of the tribolayer
35 formation with the load can be followed in the SEM images of Fig. 6. In this way, at 50
36 N (Fig. 6a), many GNPs were revealed (dark features in Fig. 6a) and debonding was
37 observed at some SiC/GNP interfaces (Fig. 6b). Moreover, the reciprocating test
38 promoted the surface damage of the GNPs (Fig. 6b), in agreement with the higher I_D/I_G
39 ratio observed in the Raman maps, also showing numerous fine rounded debris on their
40 surfaces that resembles the incipient formation of a tribolayer. As the load increased
41 (Figs. 6c,d), the microfracture process became more noticeable, which led to both an
42 augment in the material removal (Fig. 3) and the formation of a more compacted but
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discontinuous tribolayer that appeared extensively cracked at the maximum load (180 N, Fig. 6d). The tribofilm promoted the continuous solid lubrication of the tribosystem, significantly reducing the friction coefficient (Fig. 2d) but it suffered a cyclic process of formation and destabilization that contributed to the observed increment in the wear volume (Fig. 3a).

Figure 6. SEM micrographs of the SiC20GNP specimens tested at: a) 50 N, b) 50 N (high magnification image of GNP), c) 100 N and d) 180 N.

It is well known that the mechanical properties of the counterparts have a great influence in the wear response, especially when fatigue and microfracture processes are involved. In fact, some authors have proposed different models to correlate the wear behaviour of ceramics materials with their mechanical properties [15-17]. In this way, Hsu et al. [17] developed a model for ceramics operating under a mild wear regime, which takes place in the present case, considering the wear process as a simple asperity-scale abrasion. These authors stated, using the Hertzian contact stress theory, that the

W_R is directly proportional to the mean Hertzian pressure (P_m) and an undetermined function associated to the surface roughness (f), and inversely proportional to the product of the hardness (H) and the effective elastic modulus (E^*):

$$W_R \propto \frac{P_m}{H \cdot E^*} f \quad (3)$$

where P_m , E^* can be assessed according to the following equations:

$$P_m = \frac{F_N}{\pi \cdot a^2} \quad (4)$$

$$a = \sqrt[3]{\frac{3F_N \cdot R_{ball}}{4E^*}} \quad (5)$$

$$\frac{1}{E^*} = \frac{1-\nu_{plate}^2}{E_{plate}} + \frac{1-\nu_{ball}^2}{E_{ball}} \quad (6)$$

being a the radius of the contact area, R_{ball} the radius of the ball, and E and ν the elastic modulus and the Poisson's ratio of each counterpart, respectively.

Taking into account the equation (3), W_R data of the current work were plotted versus $P_m/H \cdot E^*$ (Figure 7). As it can be seen, a good correlation of the experimental values to the fittings was achieved at 100 and 180 N (coefficient of determination, R^2 , of ~ 0.98), confirming the strong relationship of the wear response with H and E . It should be mentioned that despite the function dependent on the roughness was not considered, the fitting was good, which means that the contribution of the roughness to the wear behaviour in the present case is very limited. It is interesting to remark that the fracture toughness does not seem to have any impact on the wear resistance under the current isooctane lubricated conditions; whereas it has been proved to be decisive under dry testing ones [9] where the tensile stresses will presumably be much higher than in lubricated systems. In the case of data for 50 N, experimental values did not fit to the

model, exhibiting almost no dependence of W_R with $P_m/H \cdot E^*$ that could be due to the very low wear attained at this load. In fact, it seems that there is a competitive process between the lubricating effect of GNPs and the H and E^* . In this way, only for SiC20GNP composites that lubricating effect of GNPs is able to counterbalance the strong decrease in those mechanical parameters.

Figure 7. Wear rate (W_R) versus $P_m/H \cdot E^*$ for all tested materials according to Hsu et al.'s model [17]. GNPs content for each data appears between brackets.

4. Conclusions

Large graphene fillers contents (20 vol.%) are required under isooctane lubrication to promote a significant decrease in the friction coefficient of SiC, reducing this parameter up to 30% independently of the applied load. This response is explained by the formation and further destabilization of a solid lubricant carbon-based tribofilm on the worn surface of the composite due to the pulling-out, cracking and smashing of the GNPs during the sliding motion. The incorporation of 20 vol.% GNPs to the SiC matrix

also enhances the wear resistance up to 35% at low load, but it considerably decreases at high loads due to the tribofilm destabilization. As a consequence the wear behaviour of SiC/GNPs composites exhibits a strong dependence with the hardness and elastic modulus. The tribological results encourage the addition of graphene fillers to SiC ceramics to diminish the friction phenomenon occurred in GDI engines.

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