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IMPROVEMENT IN THE TRIBOLOGICAL PERFORMANCE OF POLYCARBONATE VIA THE INCORPORATION OF MOLYBDENUM DISULPHIDE PARTICLES

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**IMPROVEMENT IN THE TRIBOLOGICAL PERFORMANCE OF
POLYCARBONATE VIA THE INCORPORATION OF MOLYBDENUM
DISULPHIDE PARTICLES**

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ABSTRACT

The present study undertakes the assessment of the effect of the incorporation of molybdenum disulphide (MoS_2) flakes on the tribological behavior of polycarbonate (PC) matrix. Five weight ratios of the MoS_2 particles in the PC matrix were considered in the range of 0-10 wt%. The composites were developed by direct injection molding. The determination of various tribological parameters after wear testing (the friction coefficient, the wear volume loss and the profilometric traces) and microscopical observations by scanning electron microscopy (SEM) were combined in an attempt to understand the effect of the solid lubricant in the composites' tribological performance. It was found that the tribological properties were improved by the addition of MoS_2 particles. The maximum reductions in the friction coefficient and wear were obtained with MoS_2 mass content about 7 to 10 wt%. It seems that MoS_2 decreases the polymer surface melting by dissipating the generated heat. Under sliding conditions, the incorporation of MoS_2 particles enhances the abrasive component in the wear mechanism which is manifested by the formation of scratches in the two bodies in contact.

KEYWORDS: Polycarbonate; MoS_2 ; Composite; Solid lubricant; Friction; Wear

INTRODUCTION

There is a great interest in the development of new polymer composites due to the various advantages, such as cost effectiveness, ease in manufacturing and excellent performance. Polymer composites are recommended in tribo-applications owing to the possibility of producing required material performance with judicious choice of fillers in adequate proportions in the composite. A great deal of research work has been carried out on thermoplastic composites based on layered solid lubricants such as graphite, molybdenum disulphide and boron nitride (Ben Difallah, et al. (1); Zhang, et al. (2); Suresha, et al. (3); Wang, et al. (4); Bijwe, et al. (5)). These studies have not only shown an improvement in the tribological performance of the composites depending on the nature and the amount of the solid lubricant, but also been significant in introducing such fillers to several industrial applications (Mnif, et al. (6); Deng, et al. (7); Cho, et al. (8)). The lubricant ability of the inclusions may provide better functioning by reducing friction and wear values.

Particularly, Bahadur and Gong (9) classified molybdenum disulphide (MoS_2) as one of the most common solid lubricant used. This material has a crystalline lamellar structure with hexagonal sulphur planes sandwiched between two hexagonal molybdenum layers. The atoms lying on the same crystal layer are strongly covalently bonded to each other. The layers themselves are bonded by weak van der Waals forces. Winer (10) reported that the lubrication capacity of MoS_2 , i.e. low friction characteristics and easy cleavage, is intrinsic to its layered structure. When added to thermoplastic polymers, MoS_2 may enhance friction and wear properties (Bahadur and Gong (9); Pettarin, et al. (11); Zhu, et al. (12); Ben Difallah, et al. (13)). Bahadur and Gong (9) performed friction and wear tests on polyimide (PI), polyamide 6-6 (PA 66) and polytetrafluoroethylene (PTFE) with MoS_2 as a filler. On the one hand, they found that wear was considerably reduced, and on the other hand, they revealed that the wear resistance of composites was affected by the filler content. The wear rate of PA was minimum

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3 with an optimum content of MoS₂ about 15–20 wt%. Pettarin, et al. (11) found that the
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5 incorporation of MoS₂ to high molecular weight high density polyethylene (HMW-HDPE)
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7 improves its abrasive wear and sliding performance with a content of MoS₂ for minimum
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9 wear rate around 10 wt%. They also reported that, under sliding conditions, the main wear
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11 mechanism of HMW-HDPE was changed from severe melting wear to mild adhesive wear.
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13 As for Zhu, et al. (12), they reported that MoS₂ improved dry and water sliding wear rate of PI
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15 for an optimum content of MoS₂ around 10–15 wt%. In the same vein, we have published a
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17 previous work (13) dealing with the tribological behavior of two polymers composites filled
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19 with different weight fractions of MoS₂. Conclusive results were found in order to improve
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21 the knowledge about the impact of the development methodology on the interfacial adhesion
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23 between the filler and the matrix. We concluded that the lubricating ability of MoS₂ depends
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25 on the nature of the polymer, the development methodology and the interaction between the
26
27 filler and the matrix.
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31 Polycarbonate (PC) is an amorphous engineering thermoplastic polymer which combines
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33 transparency, good thermal stability, impact resistance and the easiness to be processed on
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35 conventional machinery. New PC composites are being developed in order to improve the
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37 tribological behavior of the base thermoplastic by using various fillers, such as zinc oxide
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39 (ZnO), silicon dioxide (SiO₂), short glass fibers, ... (Carrión, et al. (14); Carrión, et al. (15);
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41 Carrión, et al. (16); Wang, et al. (17); Ho and Jeng (18)). Carrión, et al. studied the wear
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43 response of PC when ZnO nanoparticles and organoclays were added to the polymer (14)-
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45 (16). By their research work, the authors confirmed the important reduction in the wear rate
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47 with respect to neat PC. As for Wang, et al. (17), they investigated the tribological
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49 performances of PC film and nano-SiO₂/PC composite coating, which exhibited lower
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51 frictional coefficient. Concerning Ho and Jeng (18), they analyzed the wear behavior of PC
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53 reinforced with 20 wt% short glass fibers. The authors reported that the wear volume loss was
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2 significantly influenced by the fiber orientation according to the sliding direction.
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4 Therefore, the fundamental apprehension of the role attributed to different fillers in
5 tribological performance is essential for successful applications. Through this paper, studies
6 are focused on the friction and wear properties of polycarbonate composites containing 2–
7 10% of MoS₂ powder. For this purpose, wear tests of the PC composites were performed
8 using a ball on flat microtribometer. The results obtained could provide evidence to explain
9 the changes in friction and wear mechanisms at various amounts of MoS₂ particles.
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20 21 **EXPERIMENTAL DETAILS**

22 23 24 **Materials and specimen preparation**

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26 A commercial PC thermoplastic polymer was used as the matrix material. The PC pellets
27 (with a diameter of approximately 2-3.5 mm) were provided from Altech, (1.20 g/cm³ density,
28 14 g/10 min IF). A flaky-shaped MoS₂ powder (6 μm average size, 5.06 g/cm³ density) with
29 95% purity was purchased from Aldrich Chemistry (Sigma-Aldrich) (Fig. 1). The considered
30 weight fractions (wt %) of MoS₂ were 2%, 4%, 7% and 10%. After being dried in a vacuum
31 oven at 110 °C for 4 h, PC was injection molded with MoS₂ using DEMAG ERGOTECH
32 125/475 injection molding machine. Table 1 gives the injection parameters.
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45 **Fig.1.** SEM image of the flake-shaped MoS₂ particles, as supplied.

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47 **Table 1.** Injection molding parameters of PC composites.

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52 Figure 2 gives a typical scanning electron microscopy (SEM) image of the fractured surface
53 of the 10 wt% MoS₂–PC composite. There is no sign of large agglomeration of the MoS₂
54 particles.
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5 **Fig 2.** SEM picture of the fracture surface of a PC matrix composite containing 10 wt% MoS₂
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7 flakes
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10 11 12 **Friction and wear testing**

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14 A specific home-made microtribometer was employed for friction and wear tests of the
15 PC/MoS₂ composites. The apparatus is in a reciprocating ball on flat configuration. The
16 composite flat specimens were rubbed against a 19-mm-diameter high chromium steel ball
17 under a constant applied normal load of 5 N. The tests were carried out for 10,000 cycles with
18 the ball oscillating at a frequency of 1 Hz over a displacement magnitude of 2 mm. At least,
19 three friction and wear tests were conducted for each composite. Figure 3 shows the used
20 microtribometer which can be divided into three functional sub-systems. Concerning the first
21 one, it is the mechanical arrangement (measuring head) generating the required contact load
22 (normal load) between the high chromium steel ball and the flat composite sample and
23 allowing the tangential load to be monitored using a piezoelectric sensor. The measuring head
24 is equilibrated by a counterbalancing arm allowing the magnitude of the dead load to be
25 adjusted to very low values. As for the second sub-system, it is the micro-displacement
26 arrangement based upon a translation table with a precise linear ball bearing. This table is
27 coupled to a step-by-step motor using a ball screw. With regard to the third sub-system, it is a
28 control and data acquisition unit used for driving the microtribometer and for data acquisition
29 and processing. The treatment of the measurements and the display of the results are
30 performed by PC-based software.
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51 The volume of each wear scar was measured using the optical 3D surface metrology software
52 program provided by an Infinitfocus Alicona® laboratory system. To eliminate the influence
53 of debris on wear volume measurements, the samples were ultrasonically cleaned. Optical
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3 Scanning Microscopy (OSM) technique was also used to explore the worn surface. PC/MoS₂
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5 composites wear mechanisms were analyzed using SEM, JEOL JSM 6400F. The surface of
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7 the sample was coated with gold before SEM observation.
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11 **Fig. 3.** Schematic representation of the ball on flat microtribometer.
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13 14 15 16 **RESULTS AND DISCUSSION**

17 18 19 **Friction testing results**

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21 Figure 4 displays typical evolutions of the friction coefficient with the number of sliding
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23 cycles for PC/MoS₂ composites. **The curves of friction coefficient are composed of two main**
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25 **stages; the first one is the running-in stage during which the friction coefficient sharply**
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27 **increases until it reaches a maximum value, and the second one is the steady-state stage where**
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29 **the friction coefficient is stabilized and remains constant as function of the number of sliding**
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31 **cycles.** In the second stage of sliding, it seems that the stabilized value of the friction
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33 coefficient decreases with the increase of the MoS₂ content.
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41 **Fig. 4.** Typical evolutions of the friction coefficient versus the number of cycles for PC/MoS₂
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43 composites containing different wt% MoS₂.
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47 The variations of the friction coefficient with MoS₂ content after 10,000 sliding cycles are
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49 better shown in Figure 5. The mean friction coefficient decreases with the increase in the filler
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51 content. In fact, it falls from about 0.70 to 0.61 when the MoS₂ mass content increases from 0
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53 wt% to only 2 wt%, whereas, it drops to 0.54 at 10 wt% of MoS₂. In this case, the reduction in
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55 the friction coefficient is about 0.16 over the range of 0-10 wt % of the MoS₂ inclusions. The
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3 solid lubricant filler can substantially affect the friction behavior of the PC matrix. The better
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5 response corresponds to 10 wt% of MoS₂.
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9 **Fig. 5.** Evolution of the mean friction coefficient versus the MoS₂ mass content after 10,000
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11 sliding cycles for PC/MoS₂ composites. Good test reproducibility was achieved. Error bars
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13 reveal a small amount of variability among the mean friction coefficients.
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16 17 18 **Wear test results**

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20 **We have measured the wear on different composites after 10,000 sliding cycles by using the**
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22 **3D metrology device Infinitefocus. Table 2 contains the 2D transversal profiles recorded at**
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24 **the middle of the wear track and the typical 3D datasets registered on the wear areas.** The 3D
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26 projections have been reconstructed by a precision optic allowing a complete detection of the
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28 surface with a vertical resolution of 500 nm. The goal is to trace microgeometry changes on
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30 composite surfaces after wear sliding tests.
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36 **Table 2.** Typical transversal profiles and three dimensional images recorded on the worn
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38 surfaces of the PC composites with different amounts of MoS₂ fillers after 10,000 sliding
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40 cycles.
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44 For the neat PC, positive reliefs were observed over the whole sliding track. Interestingly,
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46 adding 2 wt% of MoS₂ changed the surface morphology. Topographic scans of the composite
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48 show material removal inside the wear scar and the formation of plough areas. The same
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50 trends can be seen with the other filler fractions. Wear peaks seem to be much shallower and
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52 less abundant as the fillers content increases. Thus, a finer pattern is generated with the
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54 addition of MoS₂ flakes. This was further confirmed by the 2D transversal wear profile of PC
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56 and its composites. The plot of the PC 2D profile shows evident positive reliefs along the
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3 wear track. The irregular surface is occupied by molten wear debris which were extracted and
4 re-deposited onto the polycarbonate surface. The wear profile becomes totally different when
5 2 wt% of MoS₂ is added. In fact, it exhibits almost negative reliefs. Besides, the width and
6 depth of wear tracks are gradually smaller with the increase of MoS₂ amount.
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11 Further wear parameters can be specified from the surface topography data. For each
12 PC/MoS₂ composite specimen, the wear volume loss is estimated as the difference between
13 the grooved volume (V^-) and the pile-up volume (V^+) which are defined in the schematic
14 representation in Figure 6 (Cayer-Barrioz, et al. (19)).
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23 **Fig. 6.** Schematic representation of wear profile groove on a composite surface after the
24 sliding test.
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30 Figure 7 illustrates the evolutions of the measured grooved volume (V^-), the measured pile-up
31 volume (V^+) (Fig. 7-a) and the difference between them representing the wear volume loss
32 (Fig. 7-b) as function of the filler content. It can be seen that both of the grooved and the pile-
33 up volumes were reduced when MoS₂ wt% is increased. However, the pile-up volume was
34 immediately affected by the incorporation of 2 wt% of MoS₂. It falls from 0.0072mm³ to
35 0.0056mm³ when the content of MoS₂ is 2 wt%. Molybdenum disulphide inclusions proved
36 beneficial in reducing the severe adhesive component of the PC wear. On the other hand, the
37 grooved volume (V^-) of the different composites shows lower values than the pure PC. It falls
38 from 0.0112 mm³ with pure PC to 0.0068mm³ with PC composite at 10 wt% of MoS₂.
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41 Therefore, with the rise of MoS₂ content, the wear volume loss of the composites increased
42 from about 0.0040 mm³ for pure PC to 0.0051 mm³ for 2 wt% MoS₂ flakes-filled PC
43 composites. From this point, the wear volume of the composites rapidly falls with the increase
44 of the filler content. A minimum value of 0.0022 mm³ is reached with 10 wt% of MoS₂. It is
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3 clear that the wear resistance of PC is greatly enhanced by the addition of MoS₂ fillers. In
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5 addition, it can be established that pure PC has a relatively low initial wear associated with
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7 large particles adhering to the specimen surface and thus increasing the pile-up volume (V⁺).
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11 **Fig. 7.** Evolution of the wear characteristics of PC/MoS₂ composites versus the MoS₂ mass
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13 content after 10,000 sliding cycles: (a) grooved volume (V⁻) and pile-up volume (V⁺), (b)
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15 resulting wear volume losses. Good agreement is achieved in the experimental values of the
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17 measured volumes. In all cases, the average error percentages did not exceed 14% when
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19 measuring the mean volume values.
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25 In order to enhance the analysis of the composites worn surfaces, three-dimensional wear
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27 track profiles were developed under an Optical Scanning Microscope (OSM). Figure 8 gives
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29 typical profiles obtained on specimens tested for 10,000 sliding cycles for the neat PC and the
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31 PC with 10 wt% MoS₂. The wear track of the unfilled PC is obviously not uniform, showing
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33 severe plastic deformation of the neat polymer (Fig. 8-a). Nevertheless, with filling 10 wt% of
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35 MoS₂ to the matrix polymer (Fig. 8-b), the wear track of the composite seems to be smoother
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37 and more uniform with the presence of light scratches. These results satisfy, with a larger set
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39 of observations, the information obtained in Table 2.
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45 **Fig. 8.** Three dimensional profiles recorded on the worn surfaces after 10,000 sliding cycles
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47 (OSM): (a) unfilled PC, (b) PC+ 10 wt% MoS₂.
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51 **Analysis of wear mechanisms**

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54 Figure 9 presents different optical micrographs showing the overall view of typical well-
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56 developed wear scars of PC composites with various amounts of MoS₂ filler. The neat PC
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3 wear scar (Fig. 9-a) is characterized by material movements and a large amount of plastic
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5 deformations. The figures in the middle and in the right side correspond to the PC composite
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7 wear scars with 4 and 10 wt% MoS₂, respectively (Fig. 9-b and c). The unworn surface of the
8
9 unfilled PC is different from that of PC composites. Actually, the PC surface is uniform, and
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11 the scratches left after the injection molding operation are barely seen. On the contrary, the
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13 observations of PC composites indicate the presence of irregular topographic features on the
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15 unworn parts. Furthermore, abrasive grooves are clearly seen in the two worn surfaces.
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17 Nevertheless, the wear scar width seems to be much narrower with 10 wt% of MoS₂. The
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19 incorporation of MoS₂ inclusions to the PC matrix resulted in further improvement of the
20
21 tribological properties owing to mild abrasion of MoS₂ fillers. This is probably due to the
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23 formation of an interphase layer of wear debris between the two bodies in contact (Zhang, et
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25 al. (20)).
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32 **Fig. 9.** Optical observations of the different PC composites after wear tests.
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36 For a further understanding of the wear mechanisms, SEM micrographs were also recorded in
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38 order to manifest the final worn conditions. Figure 10 shows SEM micrographs of the PC
39
40 worn surface and its counterpart. As expected, PC surface is rough (Fig. 10-a) and covered by
41
42 wear particles, thus forming adherent agglomerate (Scott (21)). At higher magnifications (Fig.
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44 10-b), the SEM micrograph clearly shows accumulated wear particles under the repeated
45
46 sliding cycles. As per Arribas et al. (22), the generated wear debris gets entrapped in the
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48 contact region and forms an adherent layer with the production of severely plastically
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50 deformed material at the tip of the wear scar. Figures 10-c and d show representative
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52 samplings of the high chromium steel ball tested against neat PC at two different
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54 magnifications. The worn surface is almost not damaged and is surrounded by large plate-like
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3 wear debris. Their size is in the order of several tens of micrometers in the in-plane
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5 dimensions (Fig. 10-d). Consistent with the results reported elsewhere (Carrión, et al. (16)),
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7 the unfilled PC is shown to have poor tribological performance characterized by severe
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9 damage originally caused by the production of a large amount of wear particles in an
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11 adhesive-abrasive wear mechanism.
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16 **Fig. 10.** SEM micrographs of the worn surface in the unfilled PC (a) and (b); and its
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18 counterpart (c) and (d), 10,000 cycles.
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23 The micrograph presented in Figure 11 shows the worn surface of PC composite with 10 wt%
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25 of MoS₂ and its counterpart. The wear scar of the composite specimen appears smooth and
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27 relatively uniform with the presence of fine scratches (Fig. 11-a). At higher magnifications,
28
29 the micrograph reveals the existence of large debris patches attached at the extremity of the
30
31 wear track. The formed layer of plastically deformed material seems to be thinner and less
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33 adherent compared with that of PC sample (Fig. 11-b). The SEM micrographs of the
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35 corresponding pin counterpart are shown in Figure 11-c and 11-d, respectively. The worn
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37 surface of the steel counterpart shows a large amount of transferred wear debris (Fig. 11-c). It
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39 can also be seen that a thick and not uniform film was clearly formed showing slight abrasive
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41 scratches (Fig. 11-d). This non-coherent film is controlled by softening and solid lubricant
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43 supply. Softening mechanisms result in discontinuous transfer particles (Samyn, et al. (23)).
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45 According to Lancaster (24), this mechanism is attributed to reducing interaction between
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47 counterface roughness and polymer sample (Lancaster (24)). It can be established that the
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49 incorporation of MoS₂ flakes promotes the change of the wear mechanism from adhesion to
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51 abrasion since it has been run with other fillers (Carrión, et al. (14); Arribas, et al. (22)). In
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53 this case, several previous studies have focused on the great influence of wear particles during
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3 friction process (Zum Gahr (25); Bourithis, et al. (26)). Depending on their physicochemical
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5 properties, their size and shape, third body wear particles may lead to abrasive wear
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7 mechanism. In the case of the unfilled polycarbonate, severe surface damages characterized
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9 by wear debris detachment in a massive quantity were developed (Carrión; et al. (16); Lee, et
10
11 al. (27)). Under the repeated sliding cycles, serious plastic deformations and subsequent
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13 energy dissipation lead to the adhesion of wear particles and thus to a severe wear
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15 mechanism. On the contrary, the incorporation of MoS₂ flakes to the PC matrix seems to
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17 change the wear mode into a predominant abrasive one. In fact, it was clearly seen that fine
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19 rays were developed in the composite worn surface, furthermore, the number of adherent wear
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21 particles decreased significantly. Pettarin, et al. (11) reported that the addition of commercial
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23 MoS₂ to HMW-HDPE matrix contribute to enhance the generated heat dissipation, thus it
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25 inhibits the surface melting of the polymer (Pettarin, et al. (11)). Consequently, the wear
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27 resistance performance of HMW-HDPE was improved by the incorporation of MoS₂.
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34 **Fig. 11.** SEM micrographs of the worn surface in the PC composite with 10 wt% MoS₂ (a)
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36 and (b); SEM micrographs of the transfer film on the counterpart (c) and (d), 10,000 cycles.
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40 CONCLUSIONS

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42 In this study, the tribological characteristics of PC/MoS₂ composites were investigated using a
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44 ball on flat microtribometer. The experimental results indicated that the unfilled PC has a poor
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46 tribological performance characterized by the formation of layered particle debris in some
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48 parts of the polymer wear track. In this case, the sheared bodies in contact are put down again
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50 while the strength of wear particles is much poorer. Opportunities provided by visual display
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52 revealed that severe adhesive/abrasive wear mechanism coupled with serious plastic
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54 deformations occurred in the unfilled polymer. The incorporation of MoS₂ flakes improved
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3 the polymer tribological performances by reduction of friction and wear. The abrasive
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5 component of wear took a dominant place by the addition of MoS₂ particles. It was reported
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7 in a previous research work that the weak bonds between the filler and the matrix cause the
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9 polymer fracture and the detachment of MoS₂ particles in the sliding trace (Ben Difallah et al.,
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11 (13)). By this way, polymer heating under sliding cycles could be restrained. This result has
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13 confirmed the hypothesis that the MoS₂ filler was responsible of decreasing thermal
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15 conductivity in various thermoplastic polymers by the increase in heat dissipation during the
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17 sliding tests.
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32
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34
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Table captions

Table 1. Injection molding parameters of PC composites.

Table 2. Typical transversal profiles and three dimensional images recorded on the worn surfaces of the PC composites with different amounts of MoS₂ filler after 10,000 sliding cycles.

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Figure captions

Fig. 1. SEM image of MoS₂ powder, as supplied.

Fig 2. SEM picture of the fracture surface of a PC matrix composite containing 10 wt% MoS₂ flakes.

Fig. 3. Schematic representation of the ball on disk microtribometer.

Fig. 4. Typical evolutions of the friction coefficient versus the number of cycles for PC/MoS₂ composites containing different wt% MoS₂.

Fig. 5. Evolution of the mean friction coefficient versus the MoS₂ mass content after 10,000 sliding cycles for PC/MoS₂ composites. Good test reproducibility was achieved. Error bars reveal a small amount of variability among the mean friction coefficients.

Fig. 6. Schematic representation of wear profile groove on a composite surface after the sliding test.

Fig. 7. Evolution of the wear characteristics of PC/MoS₂ composites versus the MoS₂ mass content after 10,000 sliding cycles: (a) grooved volume (V^-) and pile-up volume (V^+), (b) resulting wear volume losses. Good agreement is achieved in the experimental values of the measured volumes. In all cases, the average error percentages did not exceed 14% when measuring the mean volume values.

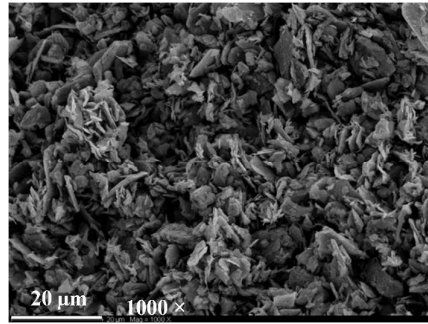
Fig. 8. Three dimensional profiles recorded on the worn surfaces after 10,000 sliding cycles (OSM): (a) unfilled PC, (b) PC+ 10 wt% MoS₂.

Fig. 9. Optical observations of the different PC composites after wear tests.

Fig. 10. SEM micrographs of the worn surface in the unfilled PC (a) and (b); and its counterpart (c) and (d), 10,000 cycles.

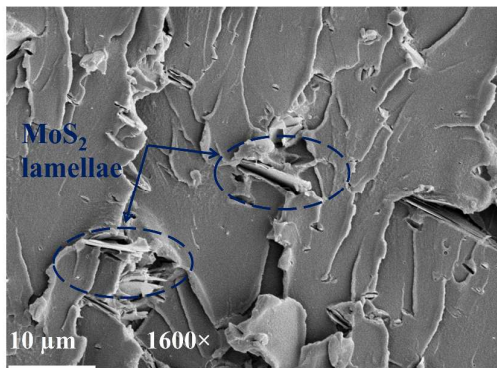
Fig. 11. SEM micrographs of the worn surface in the PC composite with 10 wt% MoS₂ (a) and (b); SEM micrographs of the transfer film on the counterpart (c) and (d), 10,000 cycles.

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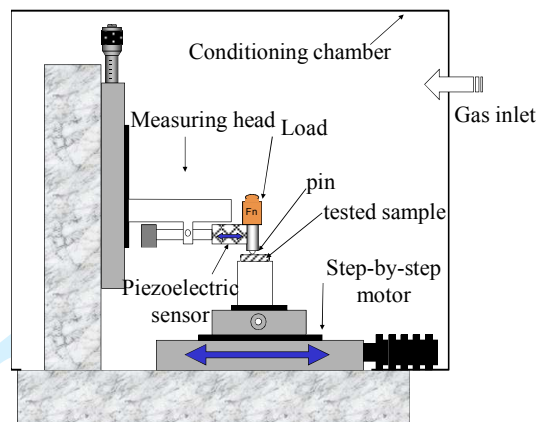
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Fig. 2



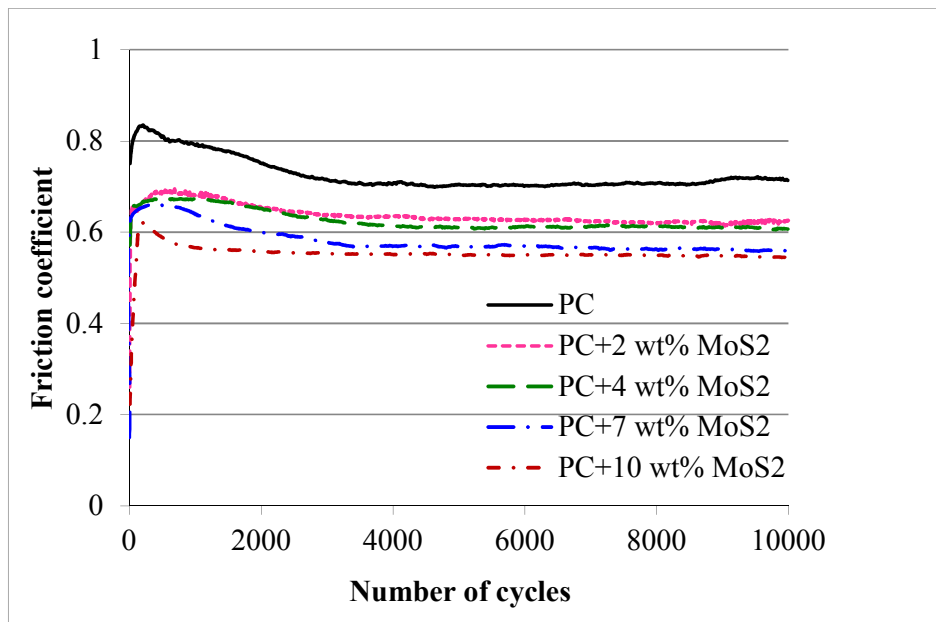
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Fig. 3



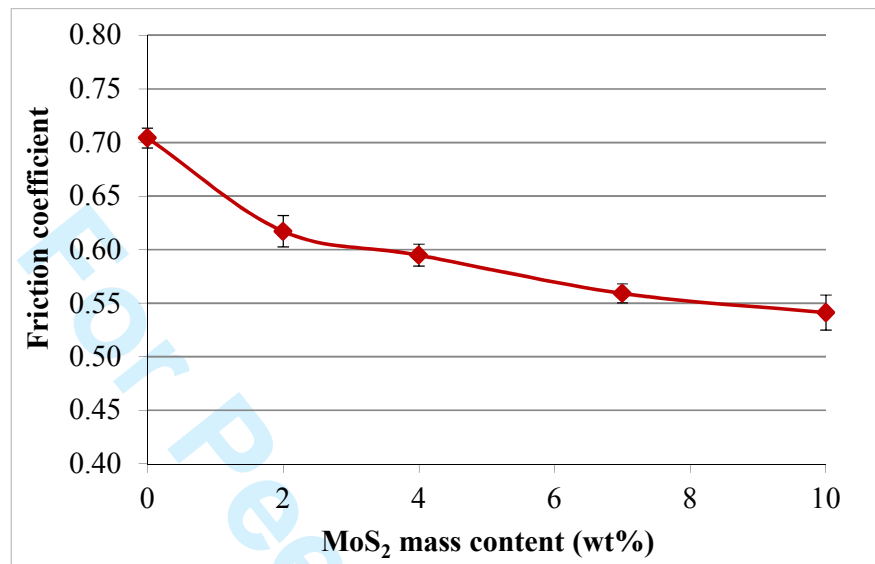
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Fig. 4



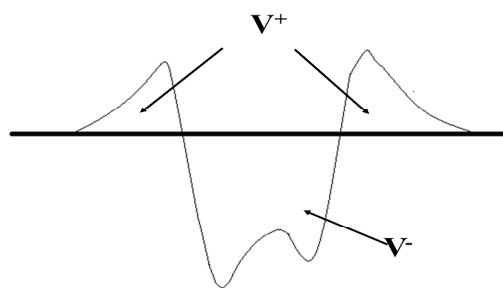
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Fig. 5



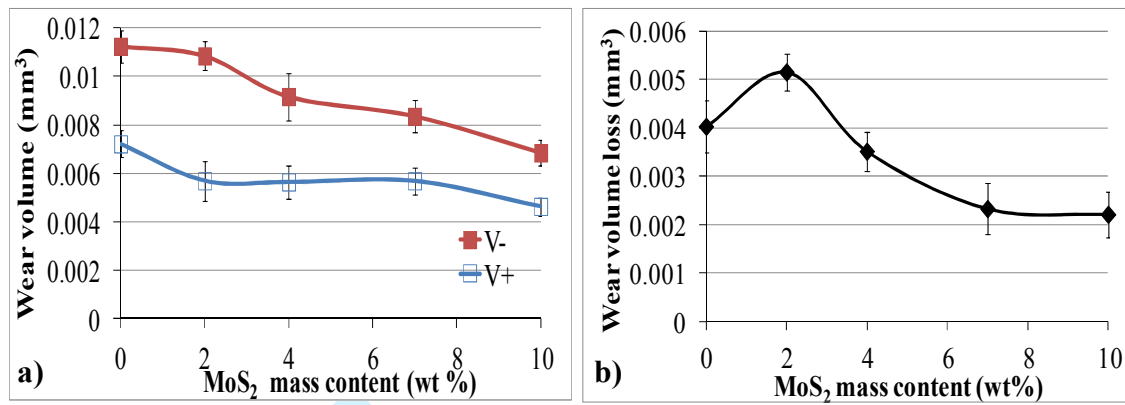
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Fig. 6



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Fig. 7



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Fig. 8

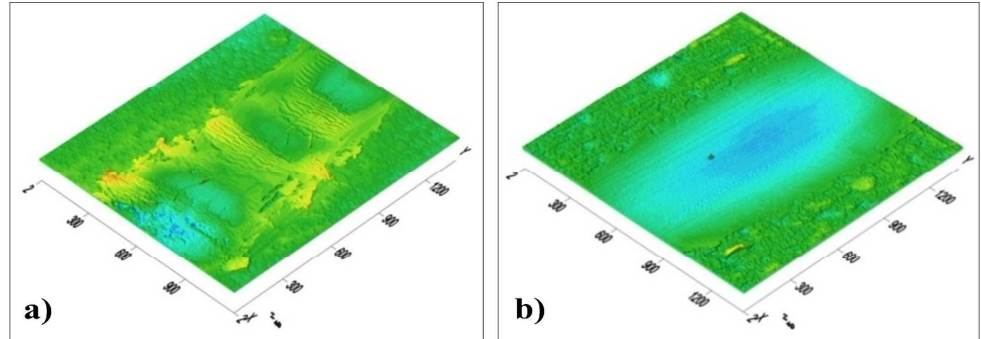
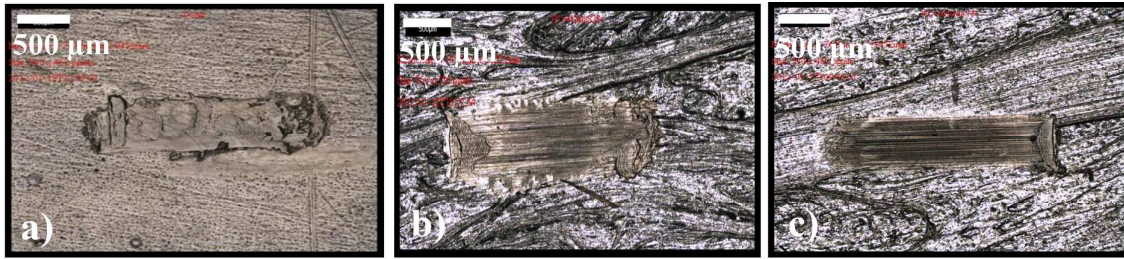
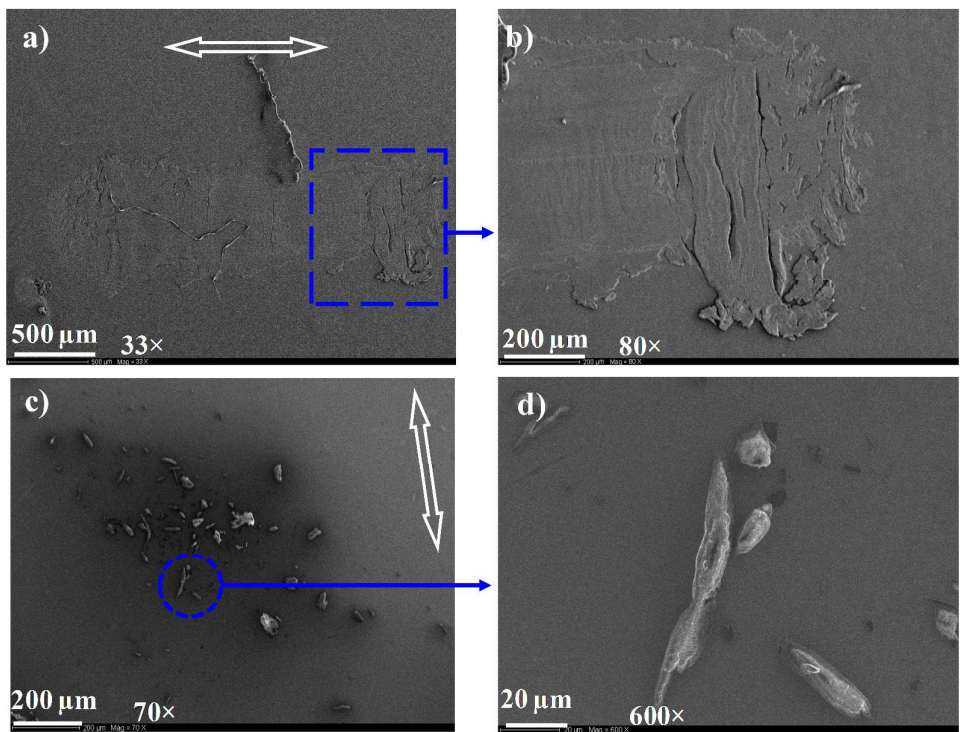


Fig. 9



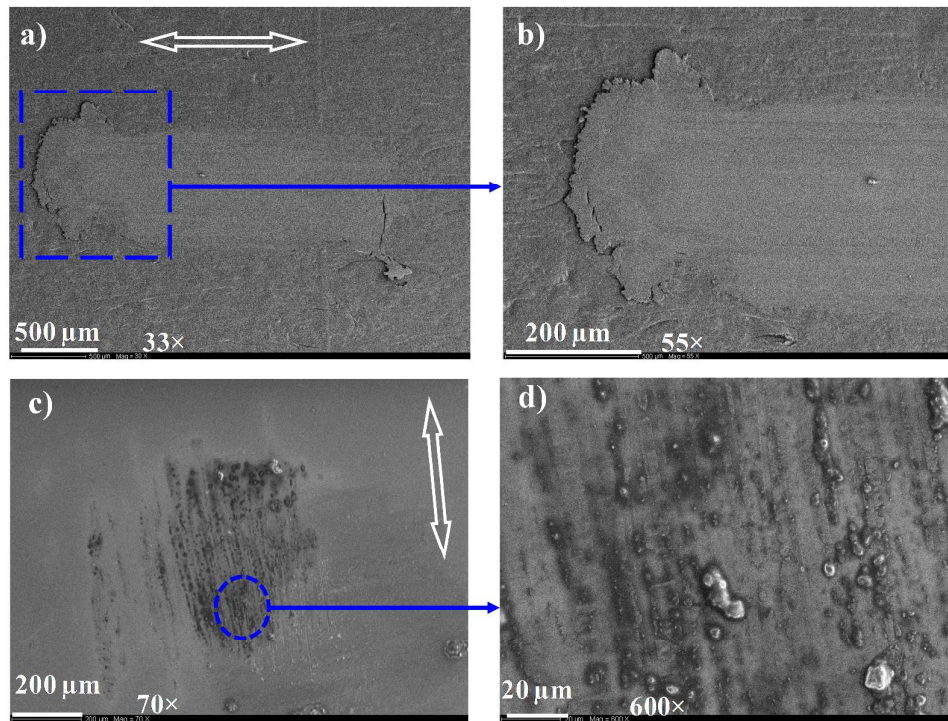
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Fig. 10



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Fig. 11

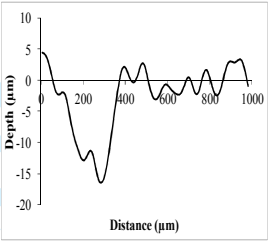
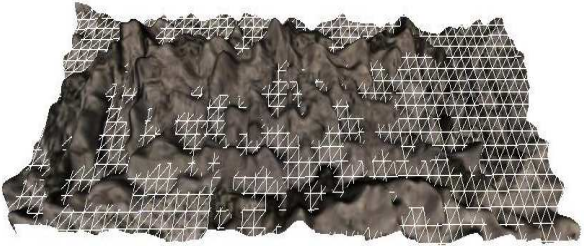
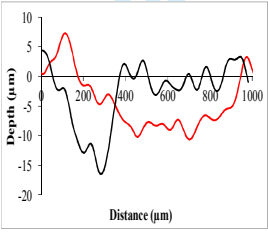

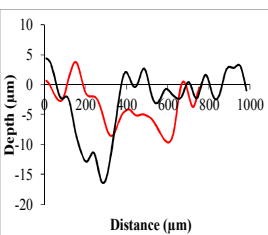
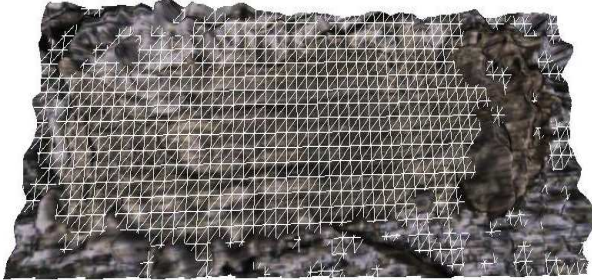
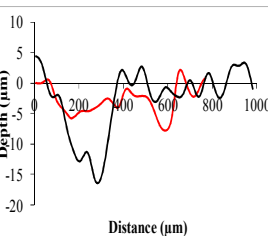



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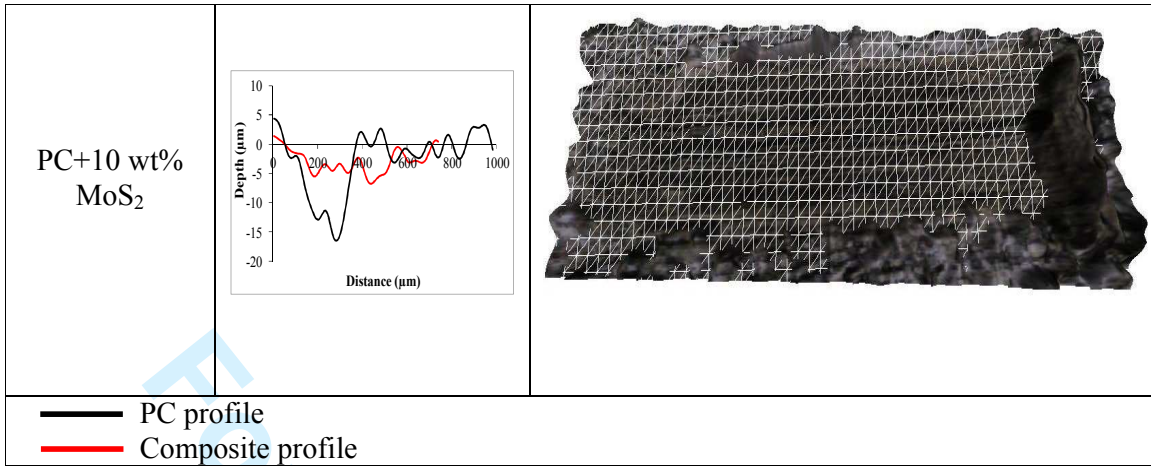
Table 1

Injection pressure (bar)	724
Injection temperature (°C)	290
Cycle time (s)	31
Holding pressure (bar)	310
Screw rotation speed (mm/s)	220

Table 2

Composite specimens	2D wear profile	3D micrograph
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PC+2 wt% MoS ₂		
PC+4 wt% MoS ₂		
PC+7 wt% MoS ₂		

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