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Microstructure, friction and wear analysis of thermoplastic based composites with solid lubricant

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Abstract – Thermoplastic based composites containing different weight fractions of molybdenum disulfide (MoS_2) solid lubricant were developed by injection molding. Polymethyl methacrylate (PMMA) and polycarbonate (PC) were chosen for the thermoplastic matrices. In order to characterize the interfacial adhesion between the matrices and the filler, we observed the fracture morphologies of selected composites. Micrographs of the fractured surfaces showed removal of MoS_2 particles by microcraking as well as the presence of voids in the case of PMMA/MoS_2 composites. These observations were confirmed by complementary images obtained using the X-ray tomography. The addition of an appropriate coupling agent may improve the adhesion between the MoS_2 particles and the polymer matrix. Tribological behavior of the composites was also investigated using a ball-on-flat microtribometer with a high chromium steel ball antagonist. It was found that the addition of MoS_2 particles didn't improve the tribological performance of the composite in the case of PMMA matrix unlike the case of PC matrix where the friction coefficient was considerably reduced.

Key words: Composite / thermoplastic / MoS_2 / solid lubricant / interface / adhesion / friction / wear

1 Introduction

In the last decade, research activities in the field of thermoplastic composites have moved towards the progress of "cost performance" engineering materials. Engineering thermoplastic composites are getting significant attention due to increased energy assessment. The advantages in terms of recyclability and lightweight together with the regulatory norms are covering increasing applications of engineering thermoplastic composites in the infrastructure applications, automotive, electrical, electronics... Indeed, it was claimed that the tribological behavior of engineering thermoplastic can be improved by filling them with inorganic particulate compounds or fibers [1–5]. In particular, polymers and coatings filled with solid lubricants have been extensively studied because of the increasing industrial and military applications [6–9]. In fact, solid lubricants such as polytetrafluoroethylene (PTFE), MoS_2 and graphite have excellent anti-friction and wear-resistance performances. They are commonly used to solve tribological problems in applications where fluid lubricants are ineffective and undesirable [10–13]. Solid lubricants have been originally developed for aerospace applications and are currently widely used in many fields [13–17].

In particular, MoS_2 is between the most promising and important solid lubricants. It offers a remarkable effect in delivering the tribo-active components to the contacting surfaces. In fact, its hexagonal lamellar structure facilitates easy glide between the S-Mo-S layers which results in a very low coefficient of friction [18, 19]. Nevertheless, a highly exothermic oxidation reaction may form molybdenum oxide (MoO_3) at temperatures exceeding 350 °C [20, 21]. Bahadur and Gong [18] reported that wear was reduced considerably by the addition of MoS_2 to PTFE, polyamide 66 (PA 66) and polyimide (PI). They also reported that the MoS_2 fillers proportion affects the wear resistance of composites. In fact, the wear rate of PA decreased initially with the increase in MoS_2 content.

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The lowest wear rate value was obtained with an optimum content about 15 to 20% in weight of MoS_2. Beyond this mass ratio, the wear rate increased again. Pettarin et al. [19] found that MoS_2 improved the sliding and abrasive wear performance of High Molecular Weight Polyethylene (HMWPE) and High Density Polyethylene (HDPE). The authors found again an optimum content of MoS_2 to be about 10% in weight. Bijwe et al. [22] studied the effect of incorporation solid lubricants and glass fibers on the adhesive and abrasive wear performance of a polyethersulphone matrix. They reported that the addition of 2% of PTFE or MoS_2 with fibers influenced the performance in a beneficial manner in the case of adhesive wear mode and in a detrimental manner in the case of abrasive wear mode. On the contrary, Bijwe et al. found that the addition of MoS_2 to PTFE matrix does not impart a good wear behavior [23]. This result is obtained because the MoS_2 filler didn't contribute in carrying the load, thus, it enhanced the wear values. Likewise, Liu et al. reported that the incorporation of MoS_2 fillers to PA6 matrix was not very effective in reducing friction and contributed to the increase in wear [24].

In the present work, first we aim to evaluate the adhesion and cohesion properties of MoS_2 fillers incorporated to two different thermoplastic matrices, the commercial PMMA and PC grades. The choice of these two amorphous thermoplastics was attributed to their high friction and large wear rate. In the second hand, our purpose consists in evaluating the actual capability of MoS_2 in improving the tribological properties of the two polymers. SEM investigations on cryofractured samples of the two thermoplastic polymers with different weight fractions of MoS_2 were done. Besides, sliding tests and microscopical techniques were combined to elucidate the role of the fillers in the tribological behavior of the two considered thermoplastic/MoS_2 composites.

2 Experimental set up

2.1 Materials

Commercial PMMA and PC thermoplastic polymers were used as the matrices of the composites. The PMMA pellets were provided from *RÖHM GmbH Chemische Fabrik* of Germany (1.19 g.cm^{-3} density, 1.8 g/10 min IF). The PC granules were purchased from *Macrolon*® of Bayer, (1.02 g.cm^{-3} density, 38 g/10 min IF). MoS_2 powder (6 μm average size, 5.06 g.cm^{-3} density) with 95% purity was provided by Aldrich Chemistry (Sigma-Aldrich) (Fig. 1). The MoS_2 fillers content in the composites was varied from 2 to 10% in weight (wt %).

2.2 Composites preparation

The apparatus used to disperse the solid lubricant fillers within the polymer matrices was a Brabender®-Plastograph EC W50EHT internal mixer coupled with a mixer head with controlled rotation speed and temperature. In-situ torque measurement allows the control of the fillers dispersion into the matrix and its uniformity. After complete homogenization, the rotors were stopped and the blends were removed and sheeted through a laboratory mill. Then the blends were injection molded using an ARBURG Allrounder 220 S injection molding machine. For each of the two considered composites, the mixing parameters are given in Table 1 while the injection parameters are given in Table 2.

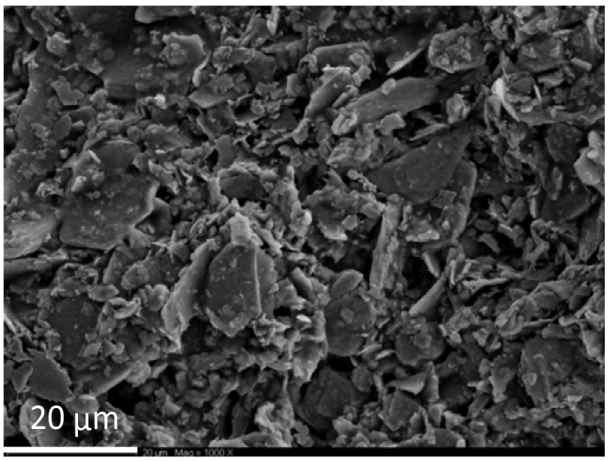


Fig. 1. SEM image of MoS_2 powder, as supplied.

Table 1. Mixing parameters of PMMA and PC composites [25].

Mixing parameters	Time (min)	Temperature (°C)	Rotation speed (rpm)
PMMA/MoS_2 composites	20	200	120
PC/MoS_2 composites	40	180	40

2.3 Microstructural characterization

2.3.1 Fractography analysis

Fractography analysis consists in the examination of fractured surfaces to deduce information upon the cohesion of the materials. This technique underpins material development and provides an insight into the physical processes by which composites are damaged and failed. Factors such as temperature, moisture, loading rate and, in the case of thermoplastic matrices, degree of crystallisation can have a considerable effect on the fracture morphology [26,27]. In addition to these factors, the composite fracture morphology is directly affected by the mechanical strength of the filler/matrix interface. Fractography has proved to be a powerful and reliable tool for the composite engineer, and is a vital technique for the overall development of composite structures [28]. In our study, the fractured samples were gold-coated and examined with a SEM (JEOL JSM 6400 F).

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Table 2. Injection molding parameters of PMMA and PC composites.

Injection molding parameters	Barrel temperature (°C)	Injection pressure (MPa)	Injection speed ($mm.s^{-1}$)
PMMA/MoS_2 composites	250	120	20
PC/MoS_2 composites	290	75	35

Table 3. Friction test conditions for PMMA and PC composites.

Test conditions	Ball diameter (mm)	Normal load (N)	Displacement magnitude (mm)	Hertz pressure (MPa)	Number of cycles
PMMA/MoS_2 composites	5	0.5	1	46	1000
PC/MoS_2 composites	19	5	2	41	10 000

2.3.2 X-ray tomography

X-ray tomography is a non-destructive testing for the study of a wide range of specimens. The internal structure of the sample can be investigated by taking a large number of projection images while rotating the specimen between projections. This set of projections can be recreated into a raw three dimensions volume. One of the most important challenges in X-ray tomography lies within the investigation of this reconstructed volume. A lot of image processing tools are accessible in two dimensions, but porting them to three dimensions volume requires a lot of additional effort [29]. For ease of illustration in this publication, we will only discuss two-dimensional images, however, volumetric information can also be obtained.

2.4 Friction and wear test

Friction and wear tests were conducted using a reciprocating ball on flat microtribometer. The composite flat specimens were rubbed against a high chromium steel ball 100Cr6 ($Ra = 0.12\ \mu m$) under a triangular alternated displacement at 1 Hz frequency. As illustrated in Table 3, the ball diameter depends on the nature of the composites. Even if the operating conditions are different, contact pressure is almost the same for the two kinds of composite. At least, three friction and wear tests were conducted for each composite. The used microtribometer can be divided into three functional sub-systems. The first one is the mechanical arrangement (measuring head) generating the required contact load (normal load) between the high chromium steel ball and the flat composite sample and allowing the tangential load to be monitored using a piezoelectric sensor. The measuring head is equilibrated by a counterbalancing arm allowing the magnitude of the dead load to be adjusted to very low values. The second sub-system is the micro-displacement arrangement based upon a translation table with a precise linear ball bearing. This table is coupled to a step-by-step motor using a ball screw. The third sub-system is a control and data acquisition unit used for driving the microtribometer and for data acquisition and processing. The treatment of the measurements and the display of the results are performed by PC based software.

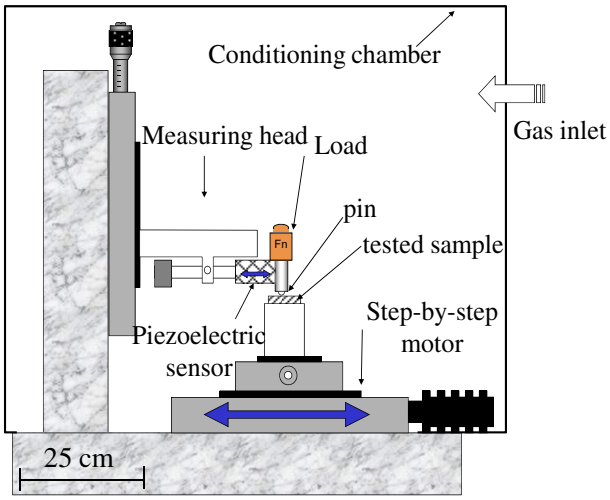


Fig. 2. Schematic representation of the ball on flat microtribometer.

The different parts of the microtribometer are shown in Figure 2. All the mechanical elements are fixed on an inertial marble structure, being ungrounded by damping blocks in order to avoid any vibrations or mechanical distortions of the system during the tests. The enclosure is made of PMMA and the regulation of the testing atmosphere is obtained by a gaseous flux. All tests were performed at ambient atmosphere and ambient relative humidity in the order of 55%. Table 3 illustrates friction test conditions for PMMA and PC composites.

3 Results and discussion

3.1 Mixing processing

The rheograms in Figure 3 show the torque against time results obtained during processing the PMMA

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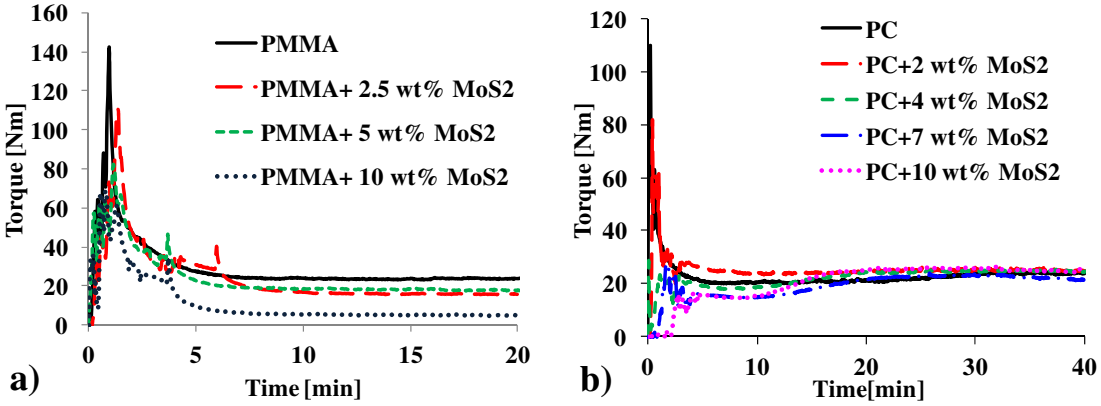


Fig. 3. Torque–time curves for; (a) PMMA/MoS_2 composites and (b) PC/MoS_2 composites.

composites and the PC composites in the totally filled chamber conditions. In all cases, an initial loading peak was registered just after the rotors were started and reflects the high viscosity of the unmelted thermoplastic polymer. The loading peak decreases as the fillers content increases due to the reduction in the charged weight of the polymer. Composites start to melt under high shear and mixing temperature, which is more than the set temperature, resulting in reduction in the viscosity. As a consequence, the torque undergoes a sudden decrease immediately after the loading peak followed by a subsequent gradual decrease. Few minutes after shearing (7 min for PMMA/MoS_2 composites and 20 min for PC/MoS_2 composites), the mixture shows stable torque indicating the completion of melting and almost constant viscosity at fixed mixing conditions [30,31].

3.2 Microstructure/fracture behavior

Figure 4 summarizes the SEM images of the fractured surfaces of PMMA/MoS_2 composite specimens. The composites display a two-phase morphology with PMMA continuous phase and a dispersed fillers phase of isolated long and narrow MoS_2 particles. PMMA matrix displays rough regions which increase in size with the weight fraction of the solid lubricant fillers. Figures 4a and b show the composite at 2.5 wt% MoS_2. A gap between the filler and the matrix is seen which corresponds to debonding at the PMMA/MoS_2 interface due to poor adhesion. Apparently, a more pronounced debonding phenomenon is observed for the other filler fractions (Figs. 4c–f). The PMMA/MoS_2 composites exhibit voids which correspond to the location of the extracted filler particles. The voids density increases with the filler's fraction. The filler is pulled from the matrix in order to dissipate the additional fracture energy. Chamis [32] found that the surface morphology of cracks is heavily dependent on the interfacial bonding conditions and results in either cohesive or adhesive failure.

For the PC/MoS_2 composites, the work was focused on the improvement of the interfacial properties between the MoS_2 flakes and the PC matrix. Thus, 8 wt% of Stearic Acid powder (SA) was added to the composite during the mixing process as a coupling agent which could probably increase the polymer-MoS_2 interaction [33]. In this case, 2 wt% of Paraffin Oil (PO) was also added to improve the viscosity of the mixture. Figure 5 compares the fractured surfaces of PC/MoS_2 composites at 10 wt% of MoS_2 with and without coupling agent. With no coupling agent, Figures 5a and b show the same mechanisms observed in the case of PMMA composites. We clearly see a phase separation between the PC matrix and the fillers as well as the presence of voids. This indicates that the polymer matrix and the MoS_2 particles didn't restrain together. Adjunction of coupling agent to the composite improved its adhesion properties (Figs. 5c and d). The interfacial interaction between the PC matrix and MoS_2 particles was stronger and this would be a reason for the improvement in the mechanical properties of the PC/MoS_2 composites [34]. Furthermore, it seems that composites with coupling agent achieve better fillers distribution.

3.3 X-ray tomography analysis

Figures 6a and b show the reconstructed 2D X-ray tomography images of PMMA/MoS_2 composite at 5 wt% of MoS_2. The black zones in Figure 6a give idea about the spatial distribution of the solid lubricant particles in the PMMA matrix. It seems that the distribution is homogeneous. The filler particles have different shapes and sizes. In fact, even if the average size of MoS_2 particles is around 6 μm, it can vary from 2 or 3 μm to more than 20 μm. The Figure 6b shows the existence of porosity in the composite. The biggest porosities seem to locate preferentially in the particles/matrix interface. This last result is in agreement with the fracture behavior analysis which indicated a poor adhesion between the MoS_2 flakes and the PMMA matrix.

3.4 Friction and wear behaviors of the composites

Figure 7 compares the friction behavior for PMMA/MoS_2 composites containing different wt% MoS_2. It can be seen that the adjunction of MoS_2

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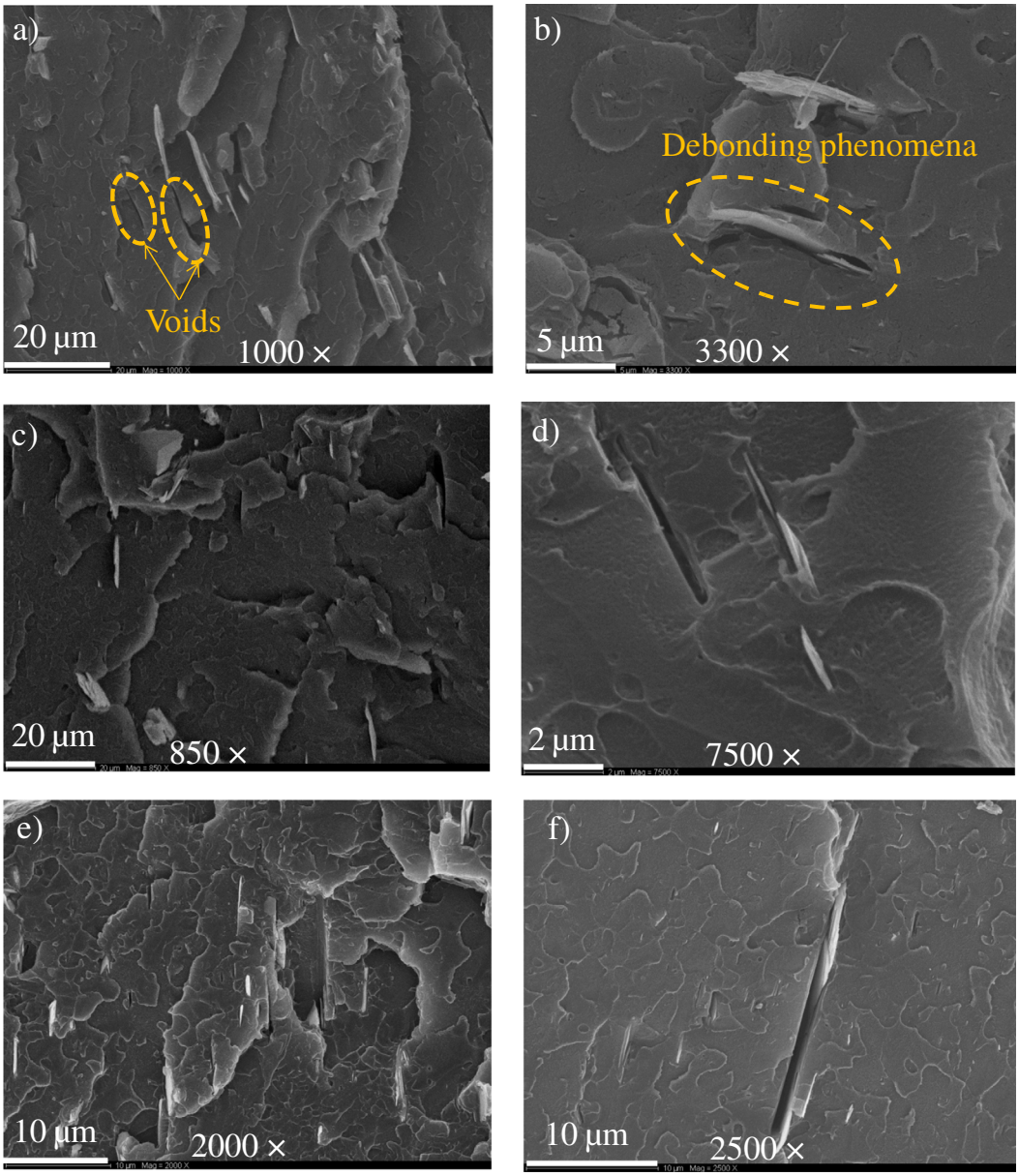


Fig. 4. SEM micrographs of the fractured surfaces of PMMA/MoS_2 composites containing: (a) and (b) 2.5 wt% MoS_2, (c) and (d) 5 wt% MoS_2, (e) and (f) 10 wt% MoS_2.

particles in the PMMA matrix does not contribute to the decrease in the friction coefficient during the test. This result can be linked to the poor adhesion between the PMMA matrix and the MoS_2 fillers. Nevertheless, it can be seen that the plot of the PMMA composite with 10 wt% of MoS_2 is constant, with low fluctuations. The particles trapped in the contact area contribute in giving a steady evolution of the friction coefficient.

OSM (Optical Scanning Microscopy) technique is used to qualitatively assess the worn surface texture of the unfilled PMMA specimen. The obtained three dimensions (3D) profile indicates that wear of the neat polymer against the high chromium steel ball was predominantly abrasive (Fig. 8a). Wear losses analysis was based on measurements made with the optical profilometer. Typical two dimensions (2D) profiles of the wear tracks are shown in Figures 8b and c for the unfilled PMMA and PMMA/MoS_2 composite containing 10 wt% MoS_2 respectively. There is no major difference between the 2D profiles of the two worn surfaces. We can conclude that addition of MoS_2 particles to the PMMA matrix does not improve the wear behavior. Due to the poor adhesion between the MoS_2 particles and the PMMA matrix, the repeated friction deformation causes continuous extraction of the fillers from the matrix during the test.

Typical evolutions of the friction coefficient with the number of sliding cycles for PC/MoS_2 composites (without coupling agent) containing different wt% MoS_2 are

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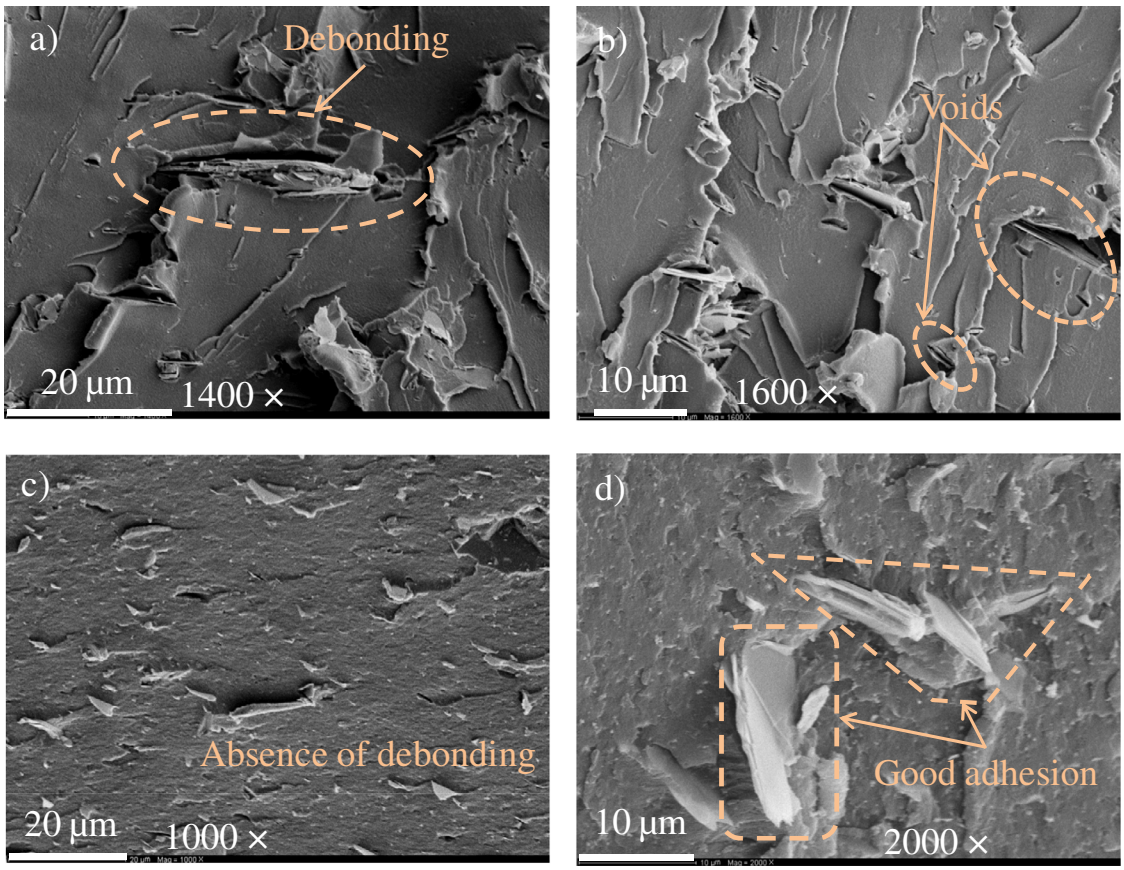


Fig. 5. SEM micrographs of the fractured surfaces of PC/MoS_2 composites containing 10 wt% MoS_2; (a) and (b) poor adhesion (without coupling agent), (c) and (d) good adhesion (with coupling agent).

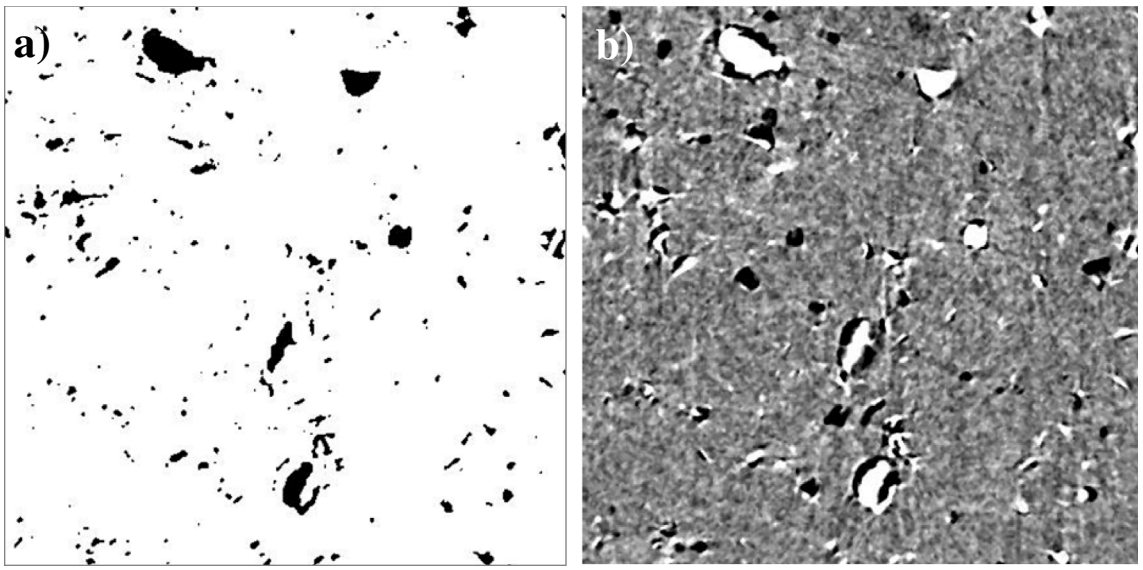


Fig. 6. Two dimensional section of PMMA/MoS_2 composite containing 5 wt% MoS_2 composite; (a) MoS_2 particles only (black), (b) MoS_2 particles (white) and porosities (black).

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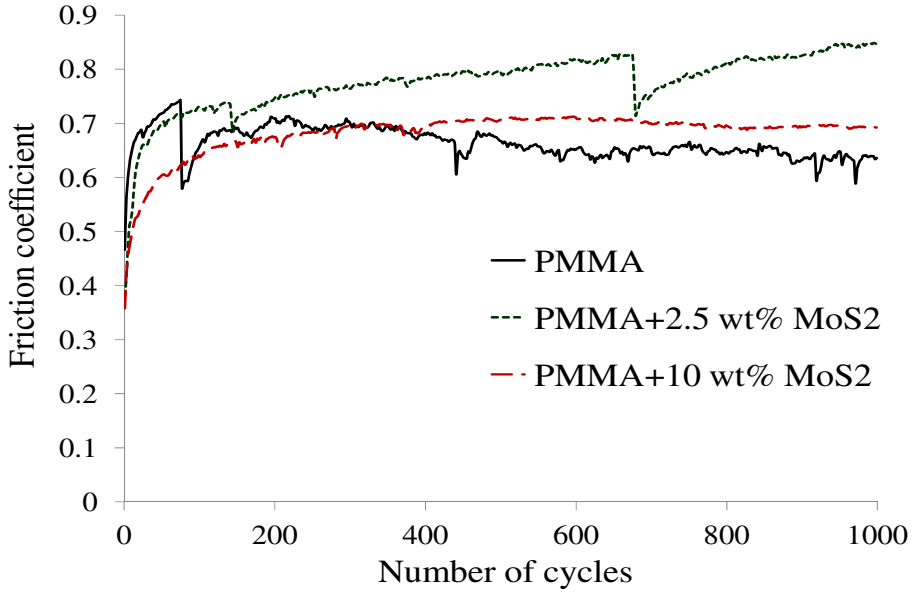


Fig. 7. Typical evolution of the friction coefficient versus the number of cycles for PMMA/MoS_2 composites containing different wt% MoS_2.

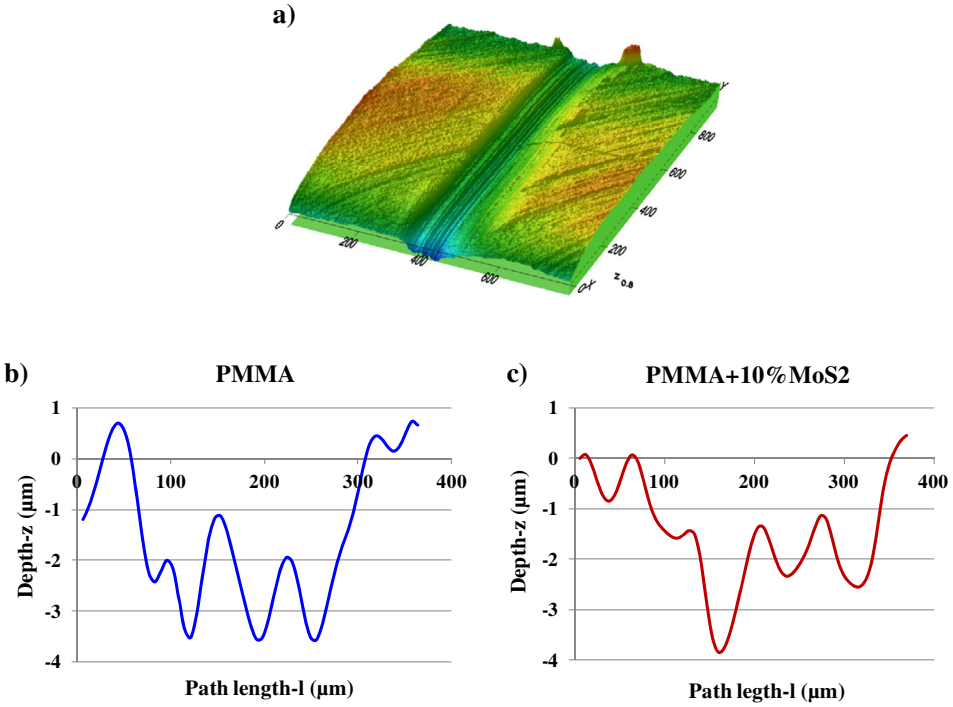


Fig. 8. (a) Three dimensions (3D) profile of the unfilled PMMA worn surface (OSM). Two dimensions (2D) profile of the wear track; (b) unfilled PMMA, (c) PMMA/MoS_2 composite containing 10 wt% MoS_2.

reported in Figure 9. Whatever the wt% MoS_2 is, the evolution of the friction coefficient with the number of sliding cycles shows two main stages. A running-in stage, for which the friction coefficient increases sharply to reach a maximum value after which it decreases gradually. A steady state stage, for which the friction coefficient remains constant as the number of sliding cycles increases. For this last stage, it appears that the stabilized value of the friction coefficient decreases as the wt% MoS_2 increases. Under sliding conditions, the solid lubricant debris is crushed between the polymer and the hard steel counterpart. A third body interface is formed and consequently the wear mechanism changed from solid bodies' friction to dry lubricated friction [35,36]. This mechanism is clearly seen in Figure 10. 3D profile of the unfilled PC wear track is obviously rough and not uniform showing

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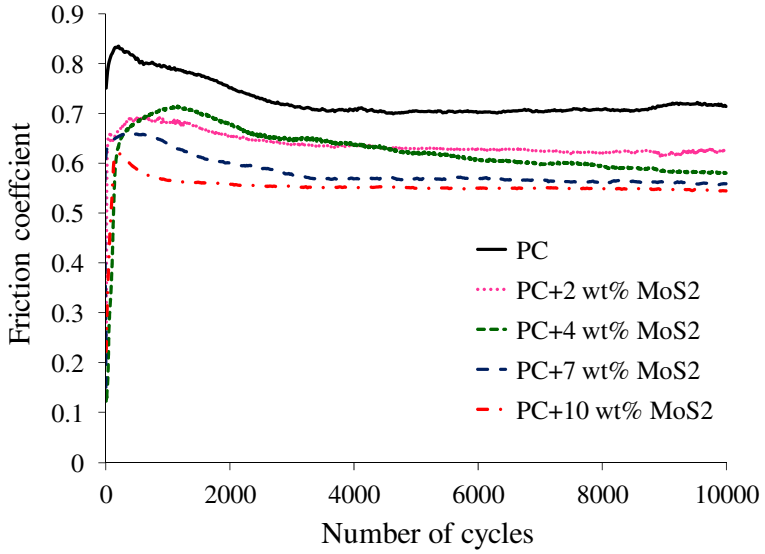


Fig. 9. Typical evolution of the friction coefficient versus the number of cycles for PC/MoS_2 composites containing different wt% MoS_2.

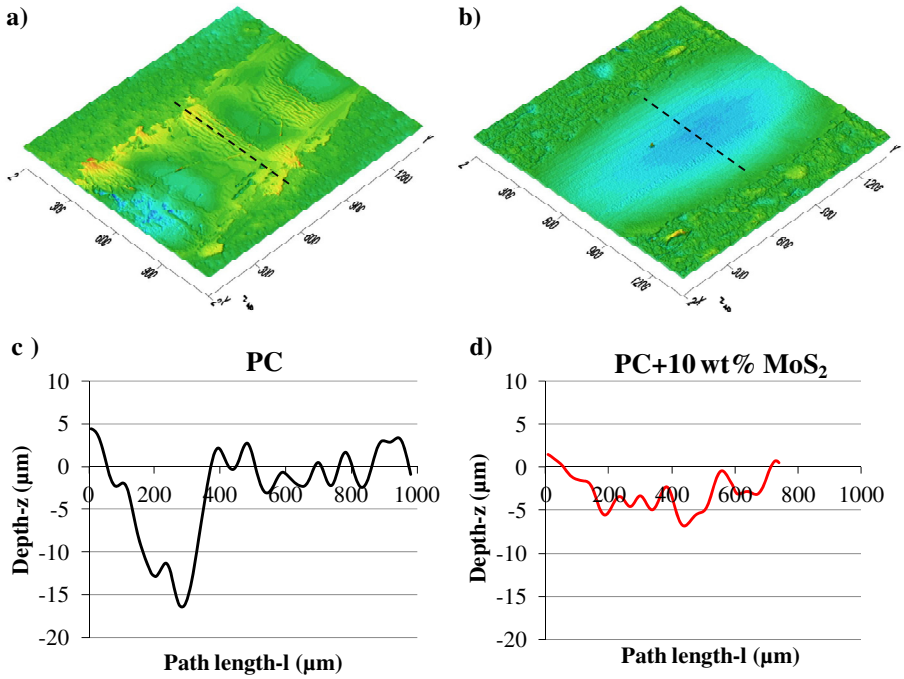


Fig. 10. 3D profiles of the worn surfaces (OSM); (a) unfilled PC, (b) PC/MoS_2 composite containing 10 wt% MoS_2. 2D profile of the wear track; (c) unfilled PC, (d) PC/MoS_2 composite containing 10 wt% MoS_2.

severe plastic deformation of the neat polymer (Fig. 10a). Nevertheless, Figure 10b indicates that the wear track of PC/MoS_2 composite containing 10 wt% MoS_2 seems to be more uniform and smoother with the presence of light scratches signs of mild abrasive wear mechanism. The typical 2D wear profiles of neat PC compared with that of the PC composite with 10 wt% of MoS_2 are also shown (Figs. 10c and d). Evident positive reliefs can be seen from the plot of the PC wear profile. The positive reliefs are the result of adherent wear debris on the polymer surface. With 10 wt% of MoS_2, the plot of the wear track exhibits almost negative relief. It can be seen also that the width and the depth of the wear track are interestingly smaller than that of neat PC.

4 Conclusion

In this study, the tribological behavior of two polymer composites filled with different weight fractions of MoS_2 was investigated. Firstly, the PMMA/MoS_2 and PC/MoS_2 composites were mixed using an internal mixer,

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then, the blends were injection molded. Laboratory tests and analysis were performed in order to characterize the composites. Micrographs of the fractured surfaces showed debonding and voids in the case of PMMA/MoS_2 composites. The same features were obtained with PC/MoS_2 composites without coupling agent. Complementary X-ray tomography images confirmed the presence of gaps between fillers and matrix. The lack of adhesion could be faced by adding an appropriate coupling agent which improves the interfacial interaction between the solid lubricant fillers and the polymer matrix such as the case of PC/MoS_2 composites. The tribological results of PMMA/MoS_2 composites showed a steady evolution of the friction coefficient at 10 wt% of MoS_2, even if the friction coefficient and the wear losses were not reduced relatively to the unfilled PMMA. The non improvement of the composites wear and friction behaviors was associated with the poor adhesion between the fillers and matrix. Unlike PMMA/MoS_2 composites, the MoS_2 powder improves the friction and wear behaviors of PC/MoS_2 composites. The composites filled with 10 wt% of MoS_2 correspond to the best friction coefficient.

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