Possible Lubrication and Temperature Effects in the Micro-scratching of Polyethylene Terephthalate

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Possible effects of water lubrication and environmental temperature on micro-scratching of PET have been studied. Micro-scratching was conducted by a purpose-built micro-scratcher with silicon cubic corner tips. Two conditions (dry scratching and scratching in water) have been investigated. Micro-scratching in water was performed at three different water temperatures (9°C, 20°C, 73°C). Zum Gahr's formalism was used to quantify the micro-scratches. All scratches show evidence of ploughing by the tip. Micro-scratches obtained in water at 20°C tend to have a negative Zum Gahr parameter. This may be due to the polymer being more elastic under these conditions causing a significant upward relaxation of the groove walls after the tip action.

1. Introduction

Micro-scratching is a powerful materials characterisation technique which provides information that aids the understanding of wear properties of materials such as polymers. Previous work on single asperity of polymers includes that of Ducret *et al.* [1] and Sinha *et al.* [2]. Wear mechanisms at the micro- and nano-scale are expected to be considerably different from those at the macro-scale [3]. This is due to the increased role of van der Waals forces that control the formation and rupture of the junction between counterface and polymer. Some studies show that the tribological behaviour of polymers exhibits a strong dependence on the imposed friction conditions [4]. Friction-induced heat results in a temperature increase in the surface layer during sliding wear [5]. More specifically Briscoe *et al.* [6] have defined the surface layer of a polymer being worn in two zones: (i) the interface zone and (ii) the cohesive zone. This distinction depends on temperature. Temperature increases in the surface layer may influence the wear mode and the deformation of the polymer during sliding wear [4, 7]. Such temperature-dependent effects suggest that the environmental temperature may also affect the tribological behaviour of polymer surfaces [8].

Conceptually the effects of micro-scratching can be discussed using a parameterisation developed by Zum Gahr. Stround and Wilman [9] found out that only a proportion of the volume of a polymer scratch groove is due to the removal of wear debris. Predominantly the groove volume is due to plastically displaced material which forms pile-up ridges alongside the groove. Based on the formalism first described for ductile metals, a parameter f_{ab} may be defined that quantifies the degree of ploughing observed for a micro-scratch. The parameter relates the groove dimensions to the dimensions of the pile-up ridges with ideal micro-ploughing corresponding to $f_{ab} = 0$ and ideal micro-cutting corresponding to $f_{ab} = 1$ [10]. In a novel approach we have chosen silicon cubic corner tips to model single asperities in the micro-scratching of polymers. Such tips are readily available and may be used with well-defined geometries in either flat-on or edge-on mode. Polyethylene terephthalate (PET) provides an interesting test-case of lubrication and environmental temperature effects in micro-scratching.

2. Experimental details

2.1. Sample preparation

PET (Goodfellow Cambridge Limited) was used without any surface modification due to its low pristine surface roughness measured with AFM as 10 ± 4 nm. The amorphous 1 mm thick PET sheet was cut into 10 mm × 10 mm pieces. Samples were cleaned ultrasonically in an alcohol bath and dried in flowing nitrogen. Before and after micro-scratching samples were characterised using AFM and, following coating with a thin silver film to suppress charging, with SEM.

2.2. Scratching experiments

The micro-scratcher is illustrated in Fig. 1. The micro-scratcher consists of three parts: (i) a head with scratching tip, (ii) a micrometer screw, and (iii) a polymer sample holder. The normal load applied was 150 µN. The scratch length varied between 150–300 µm. The sliding velocity was 300 µm/s. The tip attacked in flat-on mode. Different environmental conditions were used. Firstly, a series of dry micro-scratches were performed. Secondly, three series of micro-scratches were performed with water lubrication at water temperatures of 9°C, 20°C and 73°C, respectively. In each series about 10 scratches were conducted.

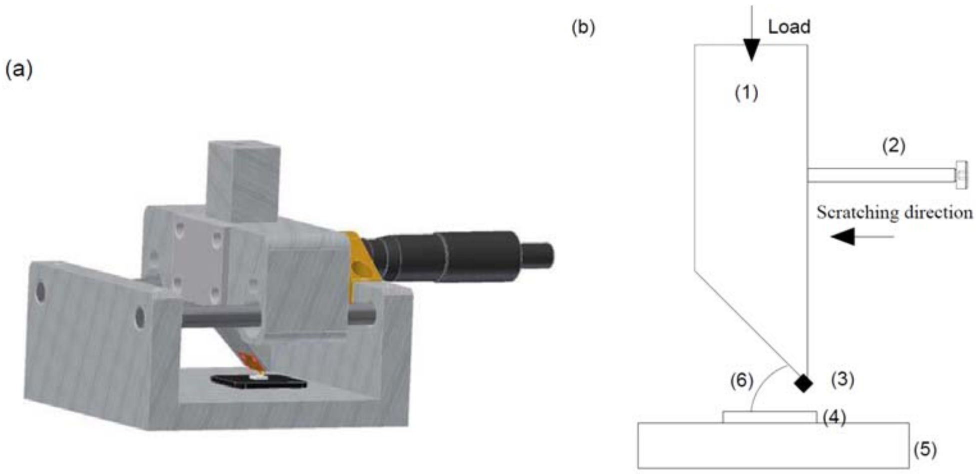

Fig. 1. (a) Illustration of the micro-scratcher; (b) Schematic of the micro-scratching beam: (1) load beam, (2) micro-meter screw, (3) silicon cubic corner tip, (4) PET sample, (5) sample holder, (6) 45° attack angle.

The scratching tips with 90° cubic corner geometry were cleaved from a commercial (100) silicon wafer along (110) crystal planes. The tip, after being cleaned ultrasonically, was imaged by SEM to verify correct geometry within the requirements of the experiment, see Fig. 2(a). Following micro-scratching the tip was imaged again to confirm that it was not modified by its action on the polymer. No polymer debris was observed on the tip.

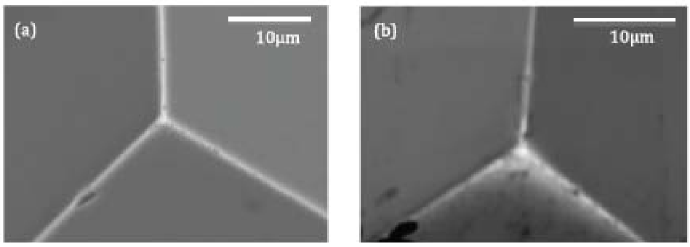

Fig. 2. (a) silicon cubic corner tip before scratch; (b) silicon cubic corner tip after scratch.

3. Results

Figure 3 displays SEM and AFM images of dry scratches. It is apparent that the micro-scratching gives reproducible results. All scratches show the characteristic symmetric ridges alongside the scratch groove, which are the result of ploughing action by the tip. The length of the scratch groove is 250 µm. No debris particles are evident in or near the groove.

Figure 4 shows SEM images of micro-scratches obtained under the four environmental conditions studied. The commencement of the scratching action results in slightly different morphological features. The following uniform motion of the tip, however, results in very similar grooves. Within the reproducibility of the scratching no significant differences of the scratching grooves are apparent.

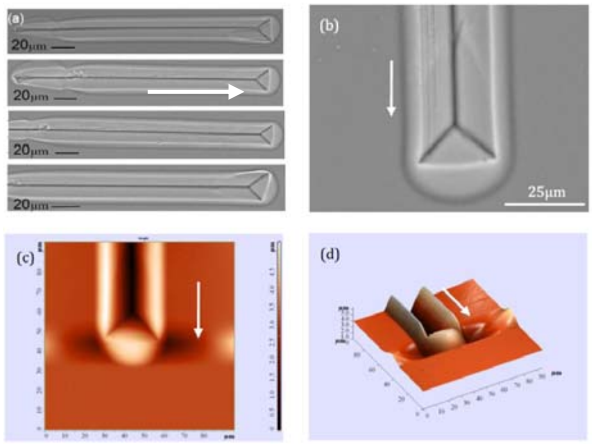

Fig. 3. (a) Reproducibility of microscratching of PET, (b,c,d) the end of a scratch groove, (b) SEM, (c) two-dimensional AFM image, (d) three-dimensional AFM image. The arrows indicate the scratching direction.

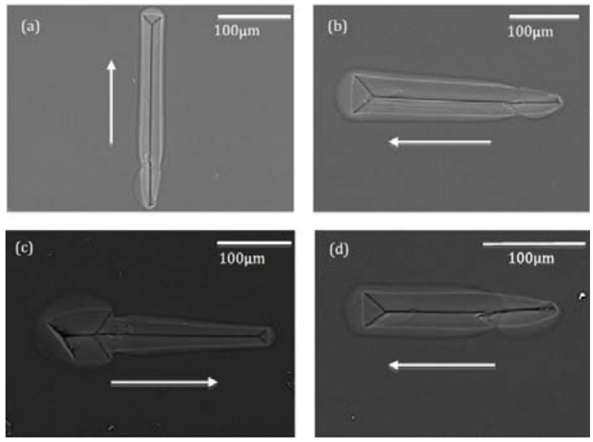

Fig. 4. (a) dry scratching, (b) scratching in 9°C water, (c) scratching in 20°C water, (d) scratching in 73°C water.

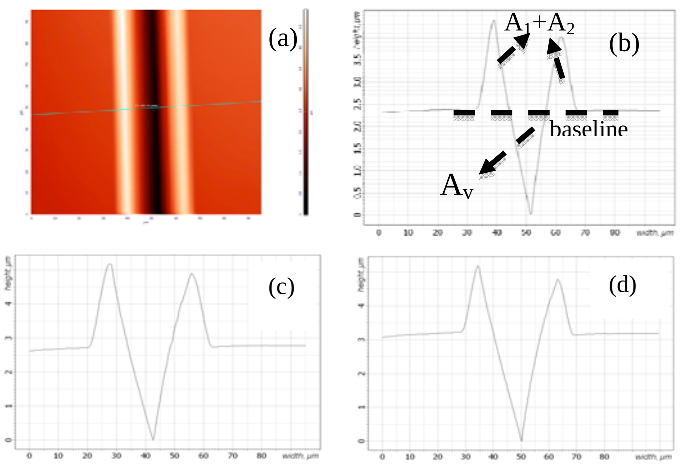

Fig. 5. Examples of measured Zum Gahr cross-sections. The groove after dry scratching in (c) is considerably wider than that after scratching in water at 20°C in (d).

Zum Gahr's cross-sectional profile analysis was used to quantify the scratch grooves. In this analysis the Zum Gahr parameter is defined as follows:

$$f_{ab} = \frac{A_v - (A_1 + A_2)}{A_v} \qquad (1)$$

Here A_v is the cross-sectional area of the scratch groove and (A_1+A_2) are the combined cross-sectional areas of the pile-up ridges. The results of the Zum Gahr analysis are shown in Figure.6. Dry scratching and scratching in water at 9°C and at 73°C give similar values of f_{ab}. Mean values are (0.21 ± 0.03), (0.16 ± 0.04), and (0.21 ± 0.04), respectively, and agree within uncertainties. In contrast the mean value of f_{ab} for microscratching in water at 20°C is negative with f_{ab} = – 0.25 ± 0.05.

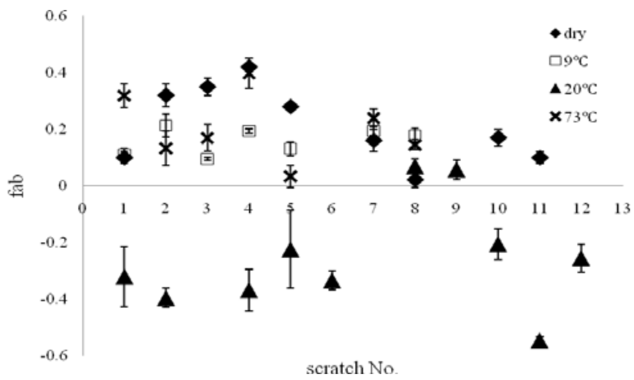

Fig. 6. Zum Gahr parameter f_{ab} values obtained for the different micro-scratching conditions.

4. Conclusions

The observation of ridges alongside the scratch groove indicates that micro-scratching of PET with cubic corner tips ploughs the material rather than cutting it. Scratching in water at 9°C and at 73°C does not significantly alter the properties of the micro-scratch when compared with dry scratching. All three conditions give positive values of f_{ab} which agree within uncertainties.

Interestingly, for a water temperature of 20°C the Zum Gahr parameter is negative. The negative value of the parameter may be explained as an upward relaxation of the compression experienced by the groove walls once the ploughing tip has passed. This reduces the cross-sectional area of the groove, adds material to the ridges and thus results in a negative Zum Gahr parameter. The observation suggests that the material is particularly elastic under this environmental condition and it warrants further investigation.

References

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