Scratch evaluation on a high performance polymer

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Abstract

The scratching process is a well know concept and is usually defined as a kind of surface abrasion, where plastic deformation is promoted by relative friction between soft phase and a hard intender. It is necessary to reduce material loss to minimum or even to reach zero to have an efficient and effective functionality of the materials. Polymers being highly sensitive to wear and scratch damage, their various modes of deformation such as, tearing, cracking, delamination, abrasive and adhesive vary with a narrow range of contact variables like applied normal load, sliding velocity, interfacial lubrication and testing temperature. This is particularly important when these materials are used to improve the tribological performance by adding various types of fillers such as, carbon fibers, graphite, PTFE, TiO_2, and ZnS are added. The polymers with nanocomposites have the advantages over micro- composites from the viewpoint of wear and scratch damage, the underlying mechanism of damage in the single asperity mode is still unclear. The goal of this study is to experimentally evaluate the deformation modes and the friction processes involved during the scratching of polymer reinforced with nanocomposites. The scratches were produced on the semi-crystalline polyetheretherketone (PEEK) surface using a Rockwell C diamond indenter was pressed onto the flat surface of each sample, until a complete load-indentation depth-curve was achieved. These scratched surfaces were assessed with optical microscope and scanning electron microscope (SEM) for prevailing deformation mechanism and the geometry of damage.

Keywords

scratch, deformation, semicrystalline PEEK, tribological performance, nanocomposites.

1. Introduction

When two materials are sliding against each other under the influence of a normal force, usually, the surface of the sharper material loses mechanical cohesion and debris is formed that is dislodged from the contact zone resulting in wear or scratch damage. It is necessary to reduce material loss to minimum amount or even to reach zero to have an efficient and effective functionality of the materials. However, polymers are highly sensitive to wear and scratch damage, their exhibit various modes of deformation such as, tearing, cracking, delamination, abrasive and adhesive even with a narrow range of contact variables like applied normal load, slider velocity, interfacial lubrication and testing temperature. This is particularly important when these materials are used to improve the tribological performance of bearings, coatings, optical, and plastics engineering applications for consumer products.

The advantages to use scratch damage in polymers is because their usage can be expanded to other applications such as electronic, household and automotive, where long term esthetics is important of scratch. Another advantages is that they can obtain the deformation characteristics for a range of imposed conditions (load, speed, temperature, etc) by a simple test (Briscoe, Evans et al. 1996; Jardret, Zahouani et al. 1998) and still to understand the friction models such as plowing and sliding contributing to friction (Briscoe and Sinha 2003).

A scratch damage is a mark that forms visible grooves and/or surface damage; this is the typical damage mode for surfaces that withstand heavy moving loads by swivels or ball bearings. The complexity of the subject is underlined by the numerous others factors that influence the material response of polymers to scratches; these include scratch loads and speed, coefficients of friction, geometry, and number of scratch tips, amount and types of fillers or additives (Wong, Lim et al. 2004).

In this investigation, various types of nanocomposites are added in a high performance polymer to improve the tribological properties and the effects of nanocomposites in the scratch damage. These nanocomposites often have a deleterious effect on the surface appearance of the polymer due to the poor scratch resistance it imparts. The reason for such an effect is still poorly understood and it will be shown in this work the usefulness of the current method in investigating this effect (J. Jancar 1999). Previous researches (R.A. Vaia 2002; A. Sviridenok 2007; Z.Z Yu 2007) generally defined three major characteristics and form the basis of performance of polymers fillers such as: nanocospically confined matrix polymer chains; nanoscale inorganic constituents, and nanoscale arrangement of these constituents. The full use of these fundamental characteristics of nano-reinforcements in polymers facilitates the achievement of enhanced properties in polymer nanocomposites, which are not displayed by their macro- and microcomposites counterparts. Furthermore, interfaces between nanofillers and matrix in polymers nanocomposites constitute a very high-volume fraction of the bulk material, which is important for bonding of filler to matrix. From the tribological aspect, the benefit of polymer nanocomposites is that the material removal is expected to be less than the micro-sized particle composites since the nano-additives have similar sizes to the segments of the surrounding polymers chains. However, for polymers nanocomposites, there are diver’s parameters that may control the friction, wear and scratch damage and include size, aspect ratio, hardness, nature of polymer/particle interface and transfer films that may arise due to the interaction of the particles and counterface, leading to the complexity in understanding the tribological behaviour of these materials. In this work we are investigated one of the previous parameters mentioned to understand the scratch behaviour of a polyetheretherketone (PEEK) polymer filled with nanocomposites, such as, carbon fibers, graphite and PTFE and to determine if the effects of the those nanocomposites affect the scratch behaviour and the material surface, using simple scratch test with a progressive load and assessed with optical microscope and scanning microscope (SEM) for prevailing deformation and geometry damage.

2. Experimental thecniques

Scratch test device

Scratch experiments were performed in a scratch tester Millennium developed by Tribotechnic with the standard ISO/EN 1071-3, ASTM G171, this test method consists of scratching on the surface with a diamond tip on which is applied a constant or progressive load. The main criteria are that the scratching process produces a measurable scratch in the surface being tested without causing catastrophic fracture, or extensive delamination of surface material. It is applicable to a wide range of materials. These include metals, alloys, and some polymers. Because the degree and type of surface damage in a material may vary with applied load, the applicability of this test to certain classes of materials may be limited by the maximum load at which valid scratch width measurements can be made. When the scratch is concluded, the sample moves under the video system, to examine the different finds of damage done by the tip and correlate it with load applied. A Rockwell C diamond tip indenter with a radius of 0.2 mm was pressed onto the flat surface of each sample until a complete load-indentation was achieve as it can be observed in the figure 1. The scratcher moves across the samples with a scratching speed of 10mm/min while simultaneously applying a progressive load of 50N. After testing, an optical microscopy (OM) and scanning electron microscopy (SEM) was used to investigate the scratch surface characteristics.

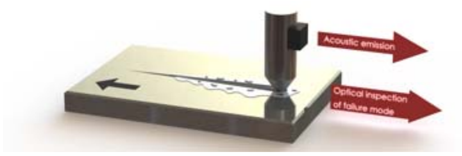

Figure 1. Scratch test device

Materials

The semi-crystalline PEEK has found a special interest, as it is characterized by a comparatively good processability as well as outstanding mechanical properties such as toughness and strength. Whereas PEEK has been identified as a good tribological polymer in general, have clearly highlighted the influence of morphological parameters such as crystallite size and degree of crystallinity as well as orientations on the resulting friction and wear performance. In addition, few fibers are incorporated in the PEEK matrix to increase the mechanical properties, reduced the friction and providing significant enhancement of stiffness and strength.

We have chosen this polymer in the present study to determine the effect of the fibers in the evaluation of scratch deformation, two different PEEK semi-crystalline polymers commercially supplied by INM- Leibniz Institute in Germany proprietary materials were selected based on their characteristics: (i) a wear grade, 10%vol short carbon fiber reinforced PEEK + 10%vol graphite as solid lubricants identified by the name of PEEK-S01 and; a self-lubricating grade PEEK with 10%vol PTFE+10%vol graphite + 10%vol short carbon fiber identified with the name of PEEK-E02. The mixture of the PEEK with various fillers was achieved by twin-screw-extruders by injection moulding with standard screw configurations, the polymers samples were presented in sizes of 70x70x4mm plates and it has been cut to the size of 40x40x4mm in a square shape. The average diameter of the particles was 300nm. In the Table 1 presents mechanical properties of the studied materials.

Table 1. Mechanical and thermal properties of the materials used in this work.

Materials properties	PEEK+CF+Gr (PEEK-S01)	PEEK+CF+Gr+PTFE (PEEK-E02)
Tensile strength (MPa)	126.8	145
E- modulus (MPa)	4696.8	11500
Impact strength (KJ/m^2)	7.5	6
Fracture toughness (MPa *m $^{1/2}$)	4.6	2
Density (g/cm^3)	1.36	1.45
Melting point (°C)	343	343

3. Results and discussion

Various properties were observed in this study such as penetration depth, tangential force, surface damage as a function of load, there are plotted in the figure 2 and 5 for the two PEEK filled with composites were shows the experimental results of scratching test. In both figure, it can be observed that the scratch force is increased as the load increased. From the Coulomb's law, it is evident that there is a linear relationship between scratch force and applied load. In previous investigations by Sujeet Sinha (Sinha and Lim 2006) founds that the scratch forces for all polymers that their investigated such as polypropylene, polycarbonate, polyvinylchloride, polyetheretherketone etc, were very close to each other for lower loads, but friction forces increases as the normal load (or scratch depth is increased) similar to our results. And they concluded that the interfacial friction effects are larger for softer polymer as the depth of scratch and hence the real contact will be greater for softer polymer than for harder polymer. Thus, the scratch force is adjusted by the indenter tip based on the interfacial friction and yield properties of the polymer. This is applicable to all polymers which deform in ductile manner.

The figure 2 is a typical testing curve for polymers under progressive load scratch test of the PEEK-E02 sample, where the tangential force curves show small magnitude of fluctuations at the beginning and at the middle of the test due to the inertial effects of instantaneously accelerating the scratch head to the designated scratch speed. But after this, the tangential force increase slightly and in a digressive manner to its final level, influences at the end by the movement of the indenter.

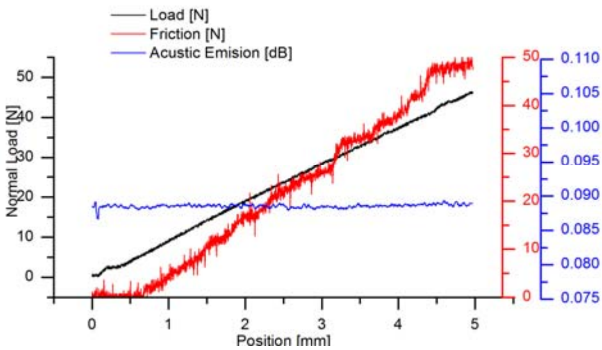

Figure 2. Typical course of the scratch loads versus scratch length measure in PEEK-E02 with a progressive load of 50N

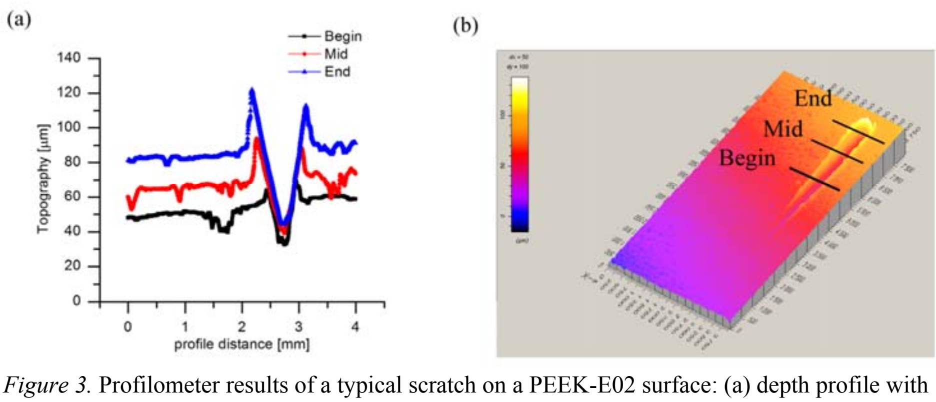

Figure 3. Profilometer results of a typical scratch on a PEEK-E02 surface: (a) depth profile with clear evidence of the scratch with different positions of the scratch; beginning, middle and end (b) 3D view of the scratch test

An evaluation of the scratch produced using profilometry leads to the profiles shown in figure 3 (a) the penetration depth with less material deformation at the beginning and a progressively increasing residual scratch towards the end of the scratch. At this position the scratch depth is also slightly deeper from the normal scratch course, due to the back-movement of the scratch head. In the figure 3(b) a corresponding a 3D view of the scratch following the original scratch direction, so that the deeper valley and the pushed-up hills at the rim of the scratch are more visible.

Additionally a few analysis of scanning electron microscopy images for the PEEK-E02 polymer figure 4 and figure 7 for PEEK-S01, were performed to understand how material deformation and removal processes take place during scratching. This has indications in the understanding of abrasive wear process for polymers because abrasive wear mechanism takes places by hard asperities or loose debris particles.

In figure 4, the sample of PEEK polymer filled with carbon fibers, graphite fibers and PTFE shows that the surface deforms with the formation of noticeable cracks, microcracks, materials removal and some detached debris particles within the scratch zone for PEEK-E02 material. In some cases depending upon the type of the materials and the severity of the contact, the material deformation can take place around the scratch tip with or without wear debris formation (Rajesh and Bijwe 2005).

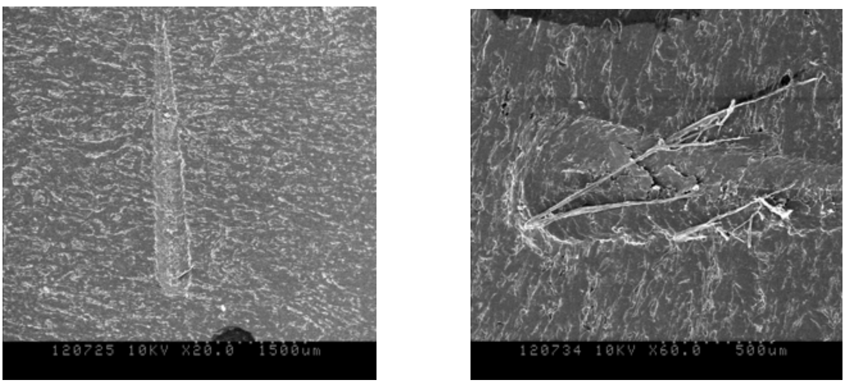

Figure 4. The scratch surface damage observed using scanning electron micrographs on PEEK-E02 under the scratching load of 50N and scratching velocity of 10mm/min

For the PEEK-S01 material filled with carbon fibers, graphite fibers shows smaller differences in the curve under the progressive load of the scratch test compared with the PEEK-E02 polymer as it can be seen in the testing curve under load scratch test figure 5, the appearance of the scratches on the both materials is rather similar but in this case with less fluctuations and smooth changes during the scratching test. The scratch frictional force, and contact zone size increased almost linearly with applied load.

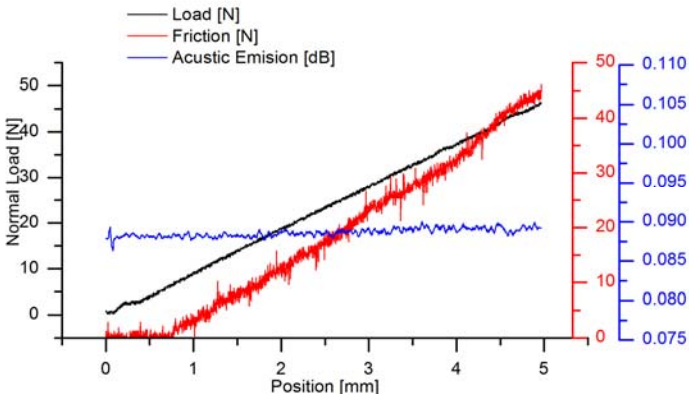

Figure 5. Typical course of the scratch loads versus scratch length measure in PEEK-S01 with a progressive load of 50N.

The depth profilometry allows obtaining more precise information on the remaining penetration depth during scratching test. In the figure 6 (a) we can see the variation of the penetration in the bulk of polymer for the PEEK-S01 filled with carbon fibers and graphite it can be seen how is slightly deeper and smoothly in the beginning until the end of the scratch test. And it can be notice in the figure 6 (b) the 3D images and the penetration depth and the grooves formation during the scratching test in the material tested.

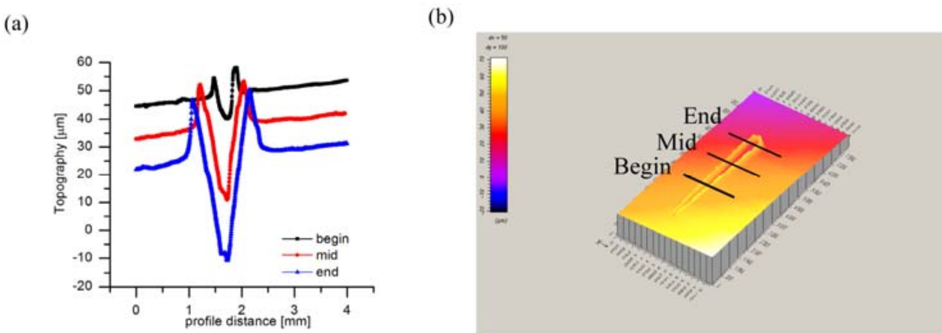

Figure 6. Profilometer results of a typical scratch on a PEEK-S01 surface: (a) depth profile with clear evidence of the scratch with different positions of the scratch; beginning, middle and end (b) 3D view of the scratch test.

Also, by scanning electron microscopy (SEM) of the PEEK-S01 polymer in the figure 7 we can see the contour of the scratched surface when the load was further increased. The PEEK-S01 surface deforms with the formation of noticeable cracks within the scratch. A regular crack formation was seen, which can be due to surface stresses that the microstructure in the semi-crystalline polymer were subjected. Comparing SEM images of the PEEK-S01 with the material PEEK-E02 it can be observed there is not clear formation of debris of the PEEK-S01 than in the SEM images of the PEEK-E02. A well-defined edge on the two sides of the scratch groove and a smooth scratch surface are formed after scratch pattern.

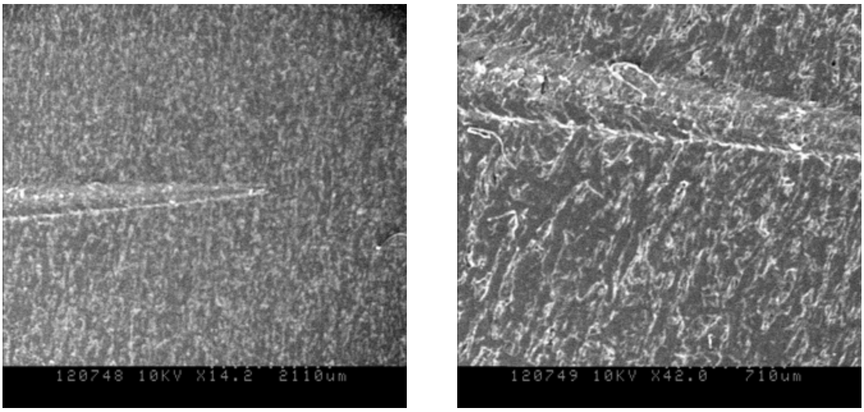

Figure 7. The scratch surface damage observed using scanning electron micrographs on PEEK-S01 under the scratching load of 50N and scratching velocity of 10mm/min

Conclusions

In this paper a scratch test method has been evaluated into a polymers filled with nanocomposites to determine the effect of the filler into the scratch resistance. The progressive load that was applied was 50N at 10mm/min of scratching speed. The scratch force was study the trends with increasing normal load and the penetration depth of scratch. Further scanning electron microscopic was carried out on the scratched polymer surface to investigate the deformation material and the removal characteristics. From this investigation we can draw the following conclusions:

Adding different fillers in the matrix of PEEK polymers it do not have significantly change in the scratching test and the penetration depth is slightly more deeper for the PEEK-S01 than PEEK-E02 this can be attributed that the PTFE the solid lubricant its accumulated around the carbon and the graphite fiber acting as a smoothing material.

Observing the SEM micrographs of the scratches on the material illustrates cracks, microcraks, material removal and debris in the contour of the scratch zone for both types of polymers.

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