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The role of surface topography in the evolving microstructure and functionality of tribofilms of an epoxy-based nanocomposite

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ABSTRACT

The topographic effect of steel counterface, finished by mechanical grinding with R_a ranging from 0.01 to 0.95 μm, on the structure and functionality of the tribofilm of a hybrid nanocomposite, i.e. epoxy matrix filled with monodisperse silica nanoparticles, carbon fibers and graphite, was systematically investigated using a pin-on-disc sliding contact geometry. The nanostructure of the tribofilm was comprehensively characterized by using combined focused ion beam and transmission electron microscope analyses. It was identified that oxidation of the steel surface, release, compaction and tribosintering of silica nanoparticles and deposition of an epoxy-like degradation product as well as fragmentation of carbon fibers are main mechanisms determining the structure and functionality of the tribofilm. The size of roughness grooves determines the type and size class of wear particles to be trapped at the surface. An optimum groove size leading to a maximum of surface coverage with a nanostructured tribofilm formed mainly from released silica nanoparticles was identified.

1. Introduction

Polymer composites have been increasingly used in last decades as tribo-materials subjected to dry sliding conditions where low friction and wear are desirable. In-situ formation of a tribofilm on the friction interface during friction process was identified to be one of the key factors dominating the tribological performance of polymer materials [1–3]. The third-body layer exerts pronounced effects on contact geometry, adhesion and tangential shear force occurring on the friction interface.

Counterface topography plays an important role as well by controlling mechanical compaction of wear particles and asperity-dominated interactions, e.g. plowing and adhesion. Menezes et al. [4] reported that the transfer layer formation of polypropylene was dependent on the surface texture of the steel mating surfaces. Franklin et al. [5] found that the composition and structure of the transfer layer of POM/PTFE blends depended on the counterface topography. Burris et al. [6] revealed that the topography of stainless steel counterface exerts an important influence on the thickness and morphology of the transfer film of PTFE/Al_2O_3 nanocomposites. The effects of three kinds of steel counterface finishing, i.e. mirror polishing, grinding and grit blasting, on the tribological behavior of PEEK-based hybrid nanocomposites were studied in our previous work [7]. It was demonstrated that variation of counterface topography leads to distinct differences not only of tribofilm coverage, but also of tribofilm composition.

Besides mechanical interactions occurring on the friction interface, we believe that the counter-face topography exerts a striking influence on the stress and flash temperature of contact asperities, which can play an important role on tribochemical reactions. Especially for composites filled with multifunctional fillers, topographic as well as structural investigations are needed for obtaining a better insight into the complex tribological mechanisms taking place at the sliding interface.

Numerous works manifested that the addition of nanoparticles into polymer matrices improved tribological properties due to the formation of a "high-quality tribofilm" [3,6,8–10]. Harris et al. [11] recently reported that the tribofilm formation of PTFE/nano-Al_2O_3 composites was a process that involves complex chemical interactions. The authors revealed that PTFE chains were broken during sliding and underwent a series of reactions to produce carboxylate chain ends, which chelated to both the metal surface and to the surface of the alumina filler particles. These tribochemical reactions form a robust tribofilm which leads to 4 orders of magnitude improvement in wear resistance over unfilled PTFE. In last years, it was identified that further addition of inorganic nanoparticles into fiber-reinforced polymer composites improved greatly the tribological

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Table 1
Designations and mean roughness R_a of the ground discs.

Designations	D0.01	D0.09	D0.16	D0.27	D0.49	D0.75	D0.96
R_a (μm)	0.01	0.09	0.16	0.27 ± 0.02	0.49 ± 0.04	0.75 ± 0.01	0.96 ± 0.01

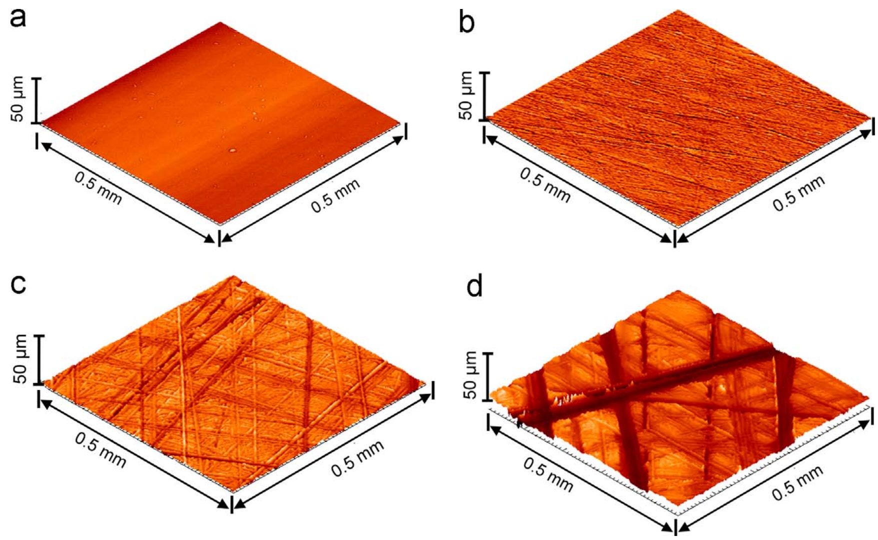

Fig. 1. 3-D surface topographies of (a) D0.09, (b) D0.16, (c) D0.49 and (d) D0.95.

performance [7,12–15]. The decisive role of the tribofilm formed on the surface of steel counterpart is evidenced since the removal of the tribofilm leads to an immediate increase of friction and wear [16]. In comparison to the nanocomposites filled only with nanoparticles, the tribofilm formation process of the hybrid nanocomposite is surmised to be more complex. Based on nanocharacterizations of the tribofilm's structure, it was assumed that mixing of wear products and tribo-sintering of nanoparticles were main mechanisms inducing the tribofilm formation [17].

In most applications of metal-polymer sliding pairs, the metallic counterface is finished by mechanical grinding or machining and roughness grooves are present on the initial counterface. It is therefore of both fundamental and practical interests to understand how the groove dimension influences the tribofilm's structure and functionality. In this work, a series of counterfaces were obtained by carefully grinding a standard bearing steel with different grades of abrasive papers. First, the counterbody topographies of wear scars produced by an EP-based hybrid nanocomposite were investigated with an FE-SEM (Field Emission Scanning Electron Microscope). Then selected sites were subjected to a comprehensive FIB-TEM investigation comprising of the preparation of cross-sectional thin lamellae by Focused Ion Beam milling and subsequent characterization in an analytical Transmission Electron Microscope. An attempt was made to reveal the relationship between the counterface topography, tribofilm structure and tribological behavior of the hybrid nanocomposite. The main objective of this work is to yield a deeper insight into the topographic effect on the formation and functionality of the tribofilm of the hybrid nanocomposite.

2. Experimental

2.1. Composite preparation

The hybrid nanocomposite studied in this work contains 10 vol% short carbon fibers, 8 vol% graphite and 5 vol% nano-silica. The composite was prepared with diglycidyl ether of bisphenol A epoxy resin (DER331, DOW). Cycloaliphatic amine hardener (HY 2954, Huntsman) was used as curing agent. The average diameter of the nanoparticles was 20 nm. The nanoparticles were in-situ synthesized and supplied as colloidal silica masterbatch (Nanopox F400, Evonik). Due to the in-situ synthesis procedure, the nano-SiO_2 particles were monodispersed in epoxy without obvious agglomeration, as proved by TEM inspections in previous works [18]. The silica masterbatch was diluted with EP resin and then required amounts of milled carbon fibers (A-385, Tenax) and graphite flakes (RGC39TS, Superior Graphite) were mixed in the resin using a vacuum dissolver (Dispermat, VMA-Getzmann). After being blended with the curing agent, the mixture was cured at 70 °C for 8 h, followed by 8 h at 120 °C. More details on material preparation are available in a previous publication [16].

2.2. Counterface preparation

Standard 100Cr6 discs (LS2542, supplied by INA) were used as sliding counterpart. In order to get different surface roughness, the discs were first polished (1 μm diamond polishing suspension) and then ground carefully in house with a series of sandpapers having different grits (P80, P180, P240, P400, P800, P1200), respectively. After the polishing and grinding process, the discs were cleaned with acetone in an ultrasonic bath. The surface roughness of each steel disc was carefully controlled using a white light profilometer (FRT).

For simplification purpose, the discs are designated in this work according to their roughness. The designations and mean roughness R_a of the ground discs are listed in Table 1. 3-D surface topographies of discs D0.01, D0.16, D0.49 and D0.96 are displayed in Fig. 1. Note that on the surfaces of D0.49, D0.75 and D0.96 (ground with sandpapers P240, P180 and P80), the roughness grooves show distinctly different dimensions. That is, large grooves and relatively small grooves coexist on the steel counterface (Fig. 1c and d). Such a surface feature is important for the final tribofilm structure, as described below.