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Wear and friction of epoxy based nanocomposites with silica nanoparticles and wax-containing microcapsules

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ABSTRACT

When subjected to wear by metal surfaces, epoxy normally exhibits rather high frictional coefficients (COF), causing high wear rate, exacerbated noise and frictional heat, thus limiting their service life. Liquid lubricants are known to reduce friction effectively, but they can be easily lost in operation. Therefore, encapsulating them is a proper solution to this issue. Herein, tribological properties of epoxy composites filled with wax-containing microcapsules (WMCs) and/or silica nanoparticles were investigated systematically. Results exhibited a tremendous decrease in the specific wear rates (W_s) and COF for the silica/WMC/epoxy ternary nanocomposites, specially three orders of magnitude reduction in W_s and a 10-fold reduction in COF were observed in specific test conditions. The wear mechanism was investigated based on worn surfaces and transfer films developed during wear tests. Furthermore, incorporating hybrid fillers negligibly deteriorates the mechanical properties of epoxy. Hence, the combination of rigid nanoparticles with WMCs is an appropriate choice when excellent tribological and mechanical properties are required.

1. Introduction

Polymer composites filled with solid or liquid lubricants have been frequently applied for wear and friction applications. The solid lubricants, such as graphite, graphene nanoplatelets and polytetrafluoroethylene (PTFE), can reduce the wear rate of polymer composites by the formation of thin and uniform transfer films, which consequently, reduces the adhesion between the polymer and counterpart [1–9]. Generally, the liquid lubricants result in lowered wear rates and frictional coefficients (COF) more significantly [10–13], compared to the solid ones.

In practice, the depletion of liquid lubricants is a serious problem [11–13], which reduces the service time and thus limits their applications. In view of these, some works [14–21] provide a solution to the above problem by incorporating of liquid-lubricant-containing microcapsules into the polymers. The liquid lubricants are sealed in the micron-sized polymer or ceramic shells (i.e. microcapsule). During wear, the shells are broken by frictional force, thereby makes the lubricants release gradually from microcapsules onto the surfaces of composite sample and counterface, and take effects.

However, a large number of microcapsules usually cause loss to the basic mechanical properties of polymers (e.g. hardness, modulus, and failure strength) [15–22]. A possible solution to this problem is to add some hard fillers to the microcapsule-containing polymers to recover these losses. So far, only a few works have been reported on the tribological properties of the ternary composites having both microcapsule and hard fillers. For example, Khun et al. [15,17] recently reported that short carbon fibers (SCFs) can increase the hardness of epoxy composites filled with wax-containing microcapsules (WMCs), and decrease the friction and wear of the samples simultaneously through lubricating effects of SCFs. In another work, they mentioned that the multiwall carbon nanotubes (MWCNTs) behaved in a similar way when improving hardness, modulus and wear resistance in the ternary composite samples [20].

In our previous work [23–27], we found that sol-gel silica nanoparticles, owing to their agglomerate-free dispersion and also a high surface area, can improve the basic mechanical properties (stiffness, strength, and fracture toughness) of epoxy materials as well as the wear

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resistance of acrylate-based polymers, as the nanoparticle content increases. It is, therefore, expected to tailor the wear resistance and other basic mechanical properties of the epoxy polymer by combining the WMCs with the silica nanoparticles together. Wear tests were carried out using a ball-on-disc mode at various testing conditions; the related wear mechanisms and the roles played by the different fillers were discerned by scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

2. Experimental

2.1. Materials

The nano-modified epoxy resin (Nanopox F400, Hanse Nanoresins, Evonik Industries, Germany) was used as the master batch, which consisted of 40 weight percent (wt.%) surface-modified nanosilica particles, ~25 nm in average size. The DGEBA neat epoxy resin (E51, equivalent weight = 185 g/eq) used was supplied by, Wuxi Resin Factory of Bluestar New Chemical Materials Co., Ltd., China. The curing agent (Albidur HE600) was a mixture of 4-methylhexahydrophtahlic anhydride (MHHPA, Puyang Huicheng Chemicals Co., Ltd., China) and N, N-benzyl dimethylamine (BDA, Sino-pharm Chemical Reagent Co., Ltd., China) at a weight ratio of 100:1. Their detailed information can be found in our previous work [28,29]. The WMCs were 200–300 μm in diameter; their information can be found in Refs. [15,17]. All materials were used without any further purification.

2.2. Sample preparation

For the preparation of silica/epoxy binary composites samples, the master batch "Nanopox F400" was diluted with a different mass of the neat epoxy resin E51; the mixtures were degassed in a vacuum oven at 60 °C for 30 min before a stoichiometric amount of the curing agent was added. The mixture was subsequently heated in an oil bath at 60 °C and mechanically stirred for 20 min prior to degassing in a vacuum oven for 15 min. Afterward, the degassed mixture was poured into a preheated steel mold and cured in a furnace. A four-step curing schedule was used: 30 min at 90 °C, 60 min at 120 °C, 30 min at 140 °C and finally 120 min at 160 °C, respectively. The cured samples were then allowed to cool down slowly to room temperature, then polished by a surface grinder on both sides to ensure sufficient surface flatness. The nomenclature of samples is as follows: Letter 'E' denotes the epoxy matrix; the first and second numbers stand for the weight fraction of silica nanoparticles, and/or WMCs, respectively. For example, 'E23_10' represents the ternary epoxy-based sample containing 23 wt% of nanosilica and 10 wt % WMCs.

TEM images of the silica/epoxy binary composites samples (Fig. 1) show the homogeneous dispersion levels of the nanoparticles in the epoxy matrix at low and high filler fractions, similar to the results in Refs. [24–27,30,31]. With the similar processes, WMC/epoxy and silica/WMC/epoxy ternary composite samples were also prepared.

2.3. Characterization

The Vickers hardness (H_v) measurements were performed using a Tukon 2500 automated Knoop/Vickers hardness tester. The hardness values were measured immediately after the indentation with a 0.02 kg load for 15 s. At least 3 specimens were used for each group of nanocomposites and 5 indentations were performed for each specimen.

Three-point bending tests were performed at room temperature (25 °C) on a Zwick universal testing machine at a constant crosshead speed of 0.5 mm/min according to the standard DIN EN ISO 178. The displacement of each specimen during the test was accurately measured by an extensometer. Moduli were calculated by considering the load in the 0.05–0.25% strain range. All presented data corresponds to the average of at least five measurements [25].

The glass transition temperature (T_g) was measured with a DSC (TA Instruments Q2000, USA) in temperature modulation mode under a nitrogen atmosphere (50 mL min^{-1}). Approximately 10 mg of the samples were sealed in aluminum pans. Modulated DSC measurements were carried out in the temperature range of −20 to 200 °C at a heating rate of 2.5 K min^{-1} with an amplitude of 0.5 K and the period of 60 s. Three samples of each composition were tested and their T_gs were obtained from the reversing heat capacity (C_p) curves [29].

Transmission electron microscopy (TEM, Tecnai G2 F20 U-TWIN, United States) and field emission scanning electron microscopy (SEM, Hitachi S-4800, Japan) were utilized to observe the homogeneity of nanosilica particles in the silica/epoxy nanocomposites and the transfer films formed on the steel ball counterparts.

2.4. Wear and friction test

Friction and wear behaviors of the samples were measured with universal tribometer (UMT-3, Bruker Corporation) under dry sliding condition through a ball-on-disc configuration, as illustrated in Fig. 2. The ball was made of 302 stainless steel with a diameter of 4.0 mm, a surface roughness, *R*a, of 90 nm and hardness of HRC 39. Before the wear tests, the specimens were polished with grit papers (No. 2000). The surface roughness of the polished surfaces was in the range of 90–120 nm. The diameter of the sliding track was 20 mm. The wear tests were performed under ambient conditions at different sliding velocities of 0.08–0.32 m/s, normal forces of 2–8 N, and test duration of

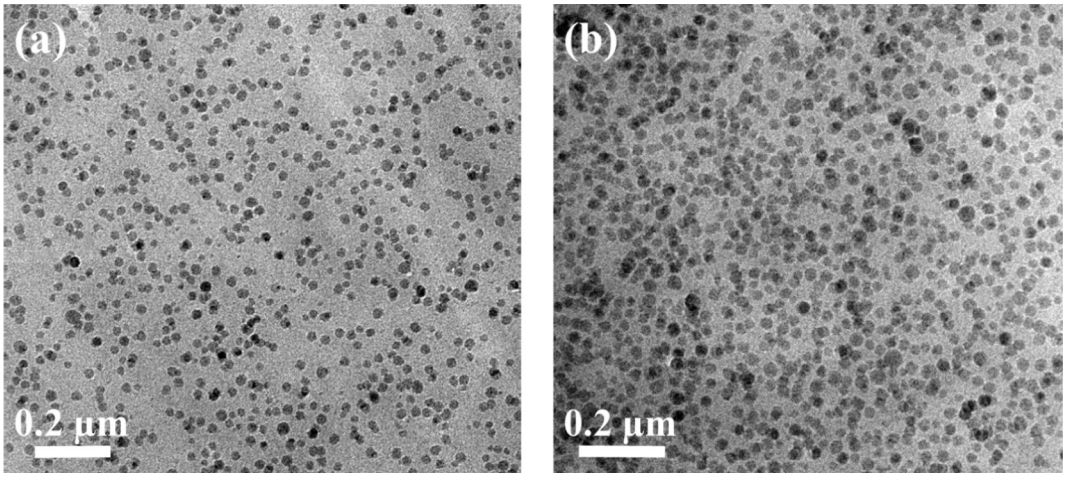

Fig. 1. TEM micrographs of ultra-thin sections for the silica/epoxy nanocomposite samples with (a) 5 wt% and (b) 23 wt% silica nanoparticles.