



Significantly modified tribological performance of epoxy nanocomposites at very low graphene oxide content

Xiao-Jun Shen^{a,b,c}, Xian-Qiang Pei^c, Shao-Yun Fu^{a,c,*}, Klaus Friedrich^{c,d}

^a Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Beijing 100190, China

^b Materials and Textile Engineering College, Jiaxing University, Jiaxing 314001, Zhejiang Province, China

^c Institute for Composite Materials, Technical University of Kaiserslautern, D-67663 Kaiserslautern, Germany

^d CEREM, King Saud University, Riyadh, Saudi Arabia

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ABSTRACT

Epoxy resins are often used for the anti-wear purpose but their wear resistance is quite low and it is thus necessary to enhance their wear resistance. In this paper, the tribological performance of graphene oxide (GO)/epoxy nanocomposites is investigated for the first time at a very low GO weight fraction of 0.05–0.5%. The friction and wear tests conducted on the GO/epoxy composites against smooth steel show that the wear resistance is significantly enhanced by the addition of GO to epoxy and the specific wear rate is reduced by 90.0–94.1% relative to the neat epoxy when the GO content is equal to 0.5 wt %. Comparisons with other nanofillers including SiO₂, TiO₂, Al₂O₃, Si₃N₄ and carbon nanotubes indicate that GO shows an obvious superiority in enhancing the wear resistance of epoxy resins at very low contents. The superiority of the GO nanosheets is related to their high specific surface area, good GO-epoxy adhesion arising from their wrinkled rough surface, oxygenous functional groups, two-dimensional planar geometry and the enhanced glass transition temperature due to the introduction of GO. Finally, the friction and wear mechanisms were tentatively proposed for the GO/epoxy composites.

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1. Introduction

Due to the unique mechanical, electrical and thermal properties of graphene, graphene-based polymer nanocomposites have recently received much attention [1–5]. Graphene-based polymer nanocomposites often exhibit improved mechanical properties at low contents. For example, the addition of the graphene platelets up to 1.5 wt% induced an increase of stiffness of the cured epoxy network with an important increase of 40 °C on glass transition temperature and an evidently enhanced storage modulus for higher temperature [1]. The graphene/epoxy composite containing chemically modified graphene nanosheets at 4 wt% showed a significantly enhanced fracture energy release rate by 200% in comparison with the neat epoxy [3]. Compared to the pristine epoxy, the Young's modulus, tensile strength and mode I fracture toughness of the graphene/epoxy composite containing only 0.1 wt% graphene were increased by 31%, 40% and 53%, respectively [4]. The introduction of 4-aminobenzoic acid functionalized graphene

at 4 wt% into an epoxy matrix enhanced the tensile strength and modulus by 29% and 20%, respectively [5].

Since graphene oxide (GO) contains many oxygenous functional groups, many graphene based polymer nanocomposites especially based on epoxy resins have been developed by using GO as the nanofiller [6–10]. The addition of GO at 1.0 wt% to an epoxy led to the significant enhancements in the flexural strength (15%), flexural modulus (11%) and impact strength (45%) [6]. At 1 wt% GO, the flexural stiffness and strength were 12 and 23% higher than for the unmodified epoxy [7]. The addition of hexachlorocyclotriphosphazene and glycidol modified GO at 4 wt% to an epoxy resin led to significant enhancements in hardness (38%). By addition of 2 wt% GO to an epoxy resin, the storage modulus was increased by 113% [8]. The addition of chemically converted graphene oxide at about 0.04 wt% to an epoxy resin led to an enhancement in compressive strength (19.9%) and toughness (92%) [9]. Qiu et al. reported that the elastic modulus and tensile strength were increased by 25% and 10% at the 0.54 vol% GO content, respectively [10].

Epoxy resins are often used for the anti-wear purpose but their wear resistance is quite low and thus it is necessary to enhance their tribological performance. Lots of efforts have been made to improve the tribological performance of epoxy resins using various fillers including

* Corresponding author. Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Beijing 100190, China. Tel./fax: +86 10 8254 3752.

E-mail address: syfu@mail.ipc.ac.cn (S.-Y. Fu).

SiO₂, TiO₂, Al₂O₃ and Si₃N₄ particles, *etc.* [11]. Nano-fillers due to their smaller size, higher surface area and better interfacial bonding *etc.* are expected to be superior in enhancing the tribological performance of epoxy resins in comparison to traditional micro- or macro-particles [12–15]. Zhang et al. reported that an addition of 2.2 vol% polyacrylamide grafted SiO₂ (SiO₂-g-PAAM) nanoparticles to an epoxy could significantly reduce the wear loss [12]. Wetzel et al. acknowledged an improved wear resistance of the epoxy composites containing nano-sized TiO₂ up to 4 vol% [13]. Wetzel et al. also reported that the addition of 2 vol% Al₂O₃ nanoparticles to an epoxy could effectively improve the wear resistance [14]. Moreover, a similar tendency was observed by Shi et al. for the epoxy composites containing nano-sized Si₃N₄ fillers up to 0.8 vol% [15]. Recently, multiwalled carbon nanotubes (MWCNTs) have been successfully used to enhance the tribological performance of epoxy resins [16–19]. The addition of MWCNTs to the epoxy resin obviously lowered the specific wear rate [16–19] and the friction coefficient [16,19]. GO is regarded as the ideal nanofiller for epoxy resins because they have some similar groups such as epoxy groups and hydrogen groups. It is expected that the introduction of GO at a very low content would significantly modify the tribological performance of epoxy resins. However, the tribological behaviors of GO/epoxy composites have not been reported yet.

In the present work, the effective roles of GO nanosheets at a very low weight fraction of 0.05–0.5% on enhancing the tribological performance of epoxy were examined. In the applications of brakes *etc.*, both a high friction coefficient and a high wear resistance are desired [20]. For this case, the friction increase and wear reduction of the epoxy resins are of great practical importance. It was observed in this study that the introduction of GO at very low contents has significantly enhanced the wear resistance and meanwhile led to a significant increase in the friction coefficient, which is very useful for high frictional applications of brakes *etc.* In addition, comparison was given between the effects of GO and other nanofillers on the tribological performance of epoxy composites [12–15]. For common nanofillers, their introduction at a much higher content into epoxy resins enhanced the wear resistance with a lower effect compared to GO. Finally, detailed discussion was presented on the enhanced tribological performance by the introduction of GO nanosheets.

2. Experimental

2.1. Materials

Graphite powders were obtained from Qingdao AoKe ShiMo Co. Ltd, China. An epoxy resin (WSR615) based on diglycidyl ether of bisphenol-A (DGEBA), with an epoxy equivalent weight of 178–200 g/eq, was purchased from Wu Xi Resin Factory, China. The curing agent diethyltoluenediamine (DETDA) (a mixture of 2,4- and 2,6-isomers) with an amine equivalent weight of 44.3 was provided by KunShan Chemical Co. Ltd, China. Ethanol, concentrated sulfuric acid, potassium permanganate and hydrochloric acid were purchased from Beijing Chemical Works and used as-received. Sodium nitrate was purchased from Tianjin JinKe Fine Chemical Industry Research Institute.

2.2. Preparation of GO and epoxy nanocomposites

Graphite oxide was prepared by oxidizing graphite powders using an approach similar to the Hummer's method [21]. The resultant graphite oxide was subsequently dispersed in ethanol to form a suspension of 0.5 mg/ml. The resulted suspension was treated by ultrasonic technique (1000 W) for 1 h to exfoliate the graphite oxide to layered graphene oxide (GO). Regarding the preparation of the GO/epoxy composites, the epoxy precursor (DGEBA) was firstly mixed with the GO suspension, and the mixture was mechanically stirred for

2 h, then the mixture was treated by the ultrasonic process for another hour. Consequently, the resultant mixture was put in an oven at 80 °C for 24 h to remove the ethanol. Afterwards, the curing agent (DETDA) was added to the above mixture in the ratio of 23.6 (DETDA) to 100 (DGEBA). The resultant suspension was degassed with a vacuum pump to eliminate air bubbles and residual ethanol. Then, the as-prepared suspension was transferred to an open mould. The suspended blends containing different GO contents were cured at 80 °C for 8 h, then at 130 °C for 12 h. After curing, the samples were cooled naturally to room temperature. The epoxy composites containing the GO content of 0.05, 0.1, 0.2 and 0.5 wt% were finally prepared. The pure epoxy sample was also prepared under the same curing condition.

2.3. Characterization

Scanning electron microscope (SEM) images for graphite and GO were obtained using a Hitachi S-4300 microscope (Japan). An atomic force microscope (AFM) (Digital Instruments Co., Nanoscope IIIa) was used to investigate the morphology of GO. The AFM images were taken with 5 × 5 μm² scan area from the prepared samples. The SEM and AFM images of GO were taken using the ethanol solution containing 0.5 mg/ml GO dropped onto a silicon chip after the ethanol was evaporated. Additional transmission electron microscopy (TEM) measurement was carried out on a JEOL JEM-2010 instrument in bright field. The phase purity of the graphite and GO was characterized by X-ray diffraction (XRD) on an X-ray diffractometer with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$). Fourier transform infrared (FTIR) spectrum for GO was obtained using an Excalibur 3100 FTIR (Varian, USA) between 500 and 4000 cm⁻¹. Dried GO was mixed with KBr powders and pressed into tablets for this characterization. The specific surface area for GO was measured via Brunauer-Emmett-Teller (BET) technique by nitrogen adsorption isotherms at 77 K using a Quadrasorb SI-MP analyzer.

Dynamic mechanical thermal analysis (DMTA) was conducted for epoxy and composites by a Gabo Qualimeter Explexor, using the tension configuration. The size of the specimens for DMTA testing was 55 × 10 × 2 mm³. The complex modulus and loss factor of each sample were determined at a constant frequency of 10 Hz, raising the temperature from –120–250 °C at a heating rate of 2 °C/min. All wear tests were carried out on a Wazau pin-on-disc machine (Germany) under dry sliding conditions: normal pressure = 1 MPa or 0.5 MPa, and sliding speed = 1 m/s at room temperature for 10 h. The normal pressure of 1 MPa is a typical sliding condition and the sliding speed of 1 m/s is the same as in our previous work [13,14]. The path length and the number of cycles are 36,031 m and 347,400 cycles, respectively. The size of the wear pins was 4 × 4 × 12 mm³. Prior to wear testing, all the specimens were pre-worn with two different grinding papers (firstly P 800 and then P 1200) placed between the pin and the disc to average surface conditions and to reduce the running-in period. The counterpart, a disc of 100Cr6 steel, was cleaned by isopropanol and acetone before use. SEM images for the worn surfaces and wear debris of the epoxy composites were obtained by a JEOL JSM-6300 microscope (Japan). The samples for the SEM observation were prepared by directly spraying a thin gold layer on the worn surfaces of the samples and the wear debris after wear testing. Sliced thin sections of GO/epoxy composites with a thickness of about 60–80 nm, prepared by ultra-microtomy, were used to take the TEM images of the composites.

3. Results and discussion

3.1. Characterization of GO

X-ray diffraction (XRD) patterns of graphite powder and GO are displayed in Fig. 1. A sharp peak at $2\theta = 26.5^\circ$ can be distinguished

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