

Effect of Polyethylene-graft-Maleic Anhydride as a Compatibilizer on the Mechanical and Tribological Behaviors of Ultrahigh-Molecular-Weight Polyethylene/Copper Composites

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ABSTRACT: Ultrahigh-molecular-weight polyethylene/copper (UHMWPE/Cu) composites compatibilized with polyethylene-graft-maleic anhydride (PE-g-MAH) were prepared by compression molding. The effects of the compatibilizer on the mechanical, thermal, and tribological properties of the UHMWPE/Cu composites were investigated. These properties of the composites were evaluated at various compositions, and worn steel surfaces and composite surfaces were examined with scanning electron microscopy and X-ray photoelectron spectroscopy. The incorporation of PE-g-MAH reduced the melting points of the composites and increased their crystallinity to some extent. Moreover, the inclusion of the PE-g-MAH compatibilizer greatly increased the tensile rupture strength and tensile modulus of

the composites, and this improved the wear resistance of the composites. These improvements in the mechanical and tribological behavior of the ultrahigh-molecular-weight-polyethylene-matrix composites with the PE-g-MAH compatibilizer could be closely related to the enhanced crosslinking function of the composites in the presence of the compatibilizer. Moreover, the compatibilizer had an effect on the transfer and oxidation behavior of the filler Cu particulates, which could be critical to the application of metallic-particulate-filled polymer composites in engineering. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 93: 948–955, 2004

Key words: compatibilization; composites; mechanical properties; polyethylene (PE)

INTRODUCTION

Ultrahigh-molecular-weight polyethylene (UHMWPE) exhibits good chemical stability, biocompatibility, and wear resistance. It has been widely used in bearing applications, particularly as a material in acetabular prosthesis components for hip and knee total joint replacement, for more than 30 years because of its excellent biocompatibility. However, the wear of UHMWPE and the resultant wear debris coming from the bearing surfaces may cause osteolysis and aseptic loosening, which has been recognized as one of the major causes of the premature failure of total joint replacement.^{1–3} Therefore, many attempts have been made to improve the wear resistance and mechanical properties of UHMWPE. It has been found that the wear resistance of UHMWPE can be significantly increased by the introduction of crosslinking to the chain structure of UHMWPE by means of peroxide

and plasma or γ -ray radiation.^{4–8,9} It is also feasible to greatly increase the wear resistance of UHMWPE by the incorporation of various inorganic or organic fillers such as metallic, oxide metallic, polymer, fiber, and ceramic particulates.^{10–19}

Nevertheless, the friction-reducing and wear-resistant mechanisms of various fillers in different polymer matrices are still not well understood, although it has been recognized that the wear reduction is largely attributable to the enhanced adhesion of the transfer film to the counterface by the fillers. Because the transfer film is held by a combination of chemical or physical bonding between the filler and the substrate and physical or mechanical bonding between the polymer and the filler,²⁰ the dispersion of the filled phase and the interfacial adhesion among various composite components play important roles in determining the wear resistance and mechanical properties of the composites.

It is feasible to improve the dispersion of fillers in a polymer matrix and increase the adhesive ability between the polymer and the fillers by the incorporation of compatibilizers in the composites. Some copolymers or graft polymers with reactive groups and similar chemical structures help to reduce the dispersed-

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phase size and enhance the interfacial adhesion between the polymer matrices and the fillers. For example, polyethylene-*graft*-maleic anhydride (PE-*g*-MAH) has been used as a compatibilizing agent in many polymer-polymer alloys²¹⁻²⁴ because it has high polarity, good adhesion to metal, glass, or ceramic, and compatibility with various polymers due to the polar groups in its molecular chain. Unfortunately, little has been reported on the effects of compatibilizers on the wear resistance of UHMWPE composites,²⁵ although some novel UHMWPE composites doped with proper compatibilizers may be much better potential alternatives to the conventional UHMWPE composites in current medical use. Accordingly, UHMWPE was filled with copper powders in the presence of PE-*g*-MAH as a compatibilizer to prepare novel ultrahigh-molecular-weight polyethylene/copper (UHMWPE/Cu) composites of much better interfacial bonding. This article deals with the effects of PE-*g*-MAH incorporation on the friction and wear behavior of the resulting UHMWPE/Cu composites.

EXPERIMENTAL

Materials

The UHMWPE powder used in this work was produced by the Second Factory of Auxiliary Agents of Beijing (Beijing, China). The molecular weight of UHMWPE was 3.5×10^6 g/mol. PE-*g*-MAH, with a grafting degree of 3.5 wt % and high-density polyethylene, was kindly supplied by the Petrochemical Research Institute of Lanzhou Petrochemical Corp. (Lanzhou, China). Maleic anhydride was produced by Xi'an Chemical Agent Factory (Xi'an, China). Copper powders (200-mesh grit size) were purchased from Beijing Chemical Agent Co. (Beijing, China).

Sample preparation

For a comparison of the effects of PE-*g*-MAH addition, the UHMWPE powders, copper powders, and mixed UHMWPE/Cu powders were modified with maleic anhydride. Maleic anhydride was dissolved in acetone, and then to the maleic anhydride/acetone solution were added the UHMWPE powders, copper powders, or mixed UHMWPE/copper powders under stirring for 10 min. The surface-modified UHMWPE powders, copper powders, and mixed UHMWPE/copper powders were obtained through the drying of the maleic anhydride/acetone solutions containing those powders in an oven at 60°C for 240 min. The modification of the powders by maleic anhydride was confirmed by IR spectrometry analysis on a Bio-Rad FTS165 Fourier transform infrared (FTIR) spectrometer (USA). The new FTIR peaks at 1786 and 1860 cm^{-1} , assigned to the carbonyl groups of maleic anhydride,

confirmed that maleic anhydride had uniformly dispersed on the surfaces of the various powders.

The modified UHMWPE, pure copper powders, mixtures of pure UHMWPE with the modified copper powders, mixtures of pure UHMWPE with pure copper powders, and mixtures of pure UHMWPE and PE-*g*-MAH with pure copper powders were mixed with PE-*g*-MAH in a torque rheometer mixer from a Haake Rheocord 90 system (Germany) at 180°C and 60 rpm for 10 min. The resulting mixtures were heated at 180°C for 90 min and then molded into plates (6 mm \times 7 mm \times 30 mm) under a pressure of 20 MPa. The composite plates to be tested were obtained after the cooling of the molded specimens in ambient air. In this study, the volume percentage of PE-*g*-MAH was divided by the total volume of the UHMWPE composites. That is, 10 vol % PE-*g*-MAH addition was the same as UHMWPE/PE-*g*-MAH/Cu with a volume ratio of 65/10/25.

Morphological and thermal analyses

The surfaces of the composite samples were coated with a thin layer of gold to allow morphology observations on a JEOL 5600LV scanning electron microscope (Japan).

The differential scanning calorimetry (DSC) analysis of the composites was carried out on a PerkinElmer DSC instrument at a heating rate of 10°C/min in a nitrogen atmosphere.

Evaluation of the mechanical and tribological properties

The mechanical properties of the composites were determined on an Instron 1121 tester at room temperature and at a crosshead speed of 100 mm/min according to ISO 178-1993. The Rockwell hardness of the composite samples was determined on a Rockwell hardness tester (model 156, Laiyang, China) at room temperature; according to ISO 2039/2-1981, the R scale was used for the Rockwell hardness measurements, and the measured Rockwell hardness should be HRR.

An M-2000 friction-and-wear tester (Xuanhua Tester Factory, Xuanhua, China) was used to examine the friction and wear behavior of the UHMWPE/Cu composites sliding against SAE52100 steel in a block-on-ring configuration. A schematic diagram of the friction-and-wear tester is shown in Figure 1. Block specimens (6 mm \times 7 mm \times 30 mm) were made of the UHMWPE composites, and counterpart rings 40 mm in diameter and 10 mm thick were made of SAE52100 steel. The friction-and-wear tests were performed at a normal load of 200 N, sliding velocities of 0.424 and 0.856 m/s, and an ambient temperature of about 25°C.

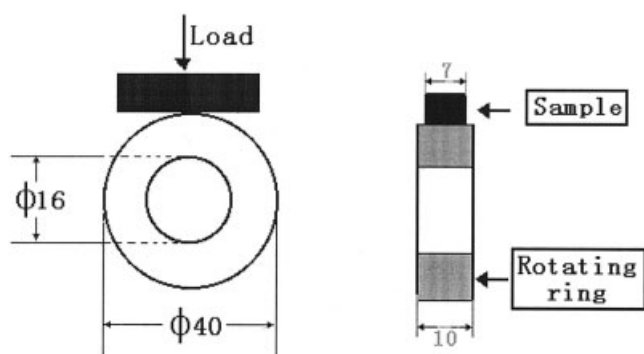


Figure 1 Schematic diagram of block-on-ring wear testing.

RESULTS AND DISCUSSION

Thermal analysis

The DSC curves of pure UHMWPE, UHMWPE/Cu, and UHMWPE/PE-g-MAH/Cu composites are shown in Figure 2, and the corresponding melting temperatures (T_m 's) and melting enthalpies (ΔH_m 's) are presented in Table I. As shown in Figure 2, the UHMWPE, UHMWPE/Cu composite, and UHMWPE/PE-g-MAH/Cu composite showed only one melting peak on the DSC curves, and this was attributed to the same backbone structure of the UHMWPE and PE-g-MAH. Thus, the ΔH_m values listed in Table I include those of both UHMWPE and PE-g-MAH. The crystallinity percentages of various PE-matrix composites could be calculated as the ratios of ΔH_m to ΔH_m^0 , where ΔH_m refers to the fusion heat of the composites to be tested and ΔH_m^0 refers to that of the total crystalline polyethylene (PE). Therefore, the crystallinity percentages of UHMWPE, UHMWPE/25% Cu, UHMWPE/5% PE-g-MAH/Cu, and UHMWPE/10% PE-g-MAH/Cu were calculated to be 56.79, 7.52, 9.94, and 11.6%, respectively, with ΔH_m values of 293 J/g for 100% crystalline PE, 166.4 J/g for UHMWPE, 22.04 J/g for PE of the UHMWPE/25% Cu composite, 29.12 J/g for PE of the UHMWPE/5% PE-g-MAH/Cu composite, and 34.06 J/g for PE of the UHMWPE/10% PE-g-MAH/Cu composite. The UHMWPE/PE-g-MAH/Cu composite with a higher PE-g-MAH content had a relatively higher crystallinity percentage, which might imply that PE-g-MAH as a compatibilizer was

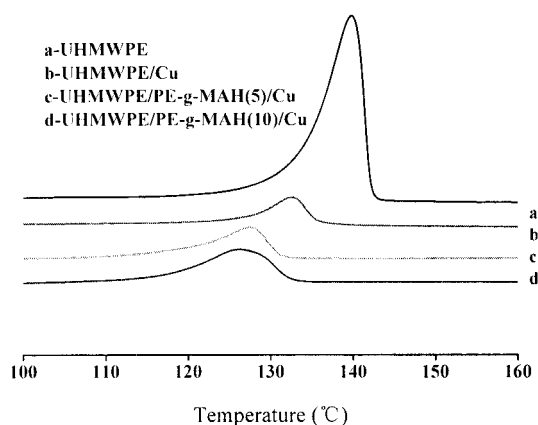


Figure 2 DSC curves of UHMWPE and UHMWPE/Cu composites: (a) UHMWPE, (b) UHMWPE/25 vol % Cu, (c) UHMWPE/5 vol % PE-g-MAH/Cu composite, and (d) UHMWPE/10 vol % PE-g-MAH/Cu composite.

able to induce and enhance the crystallization of UHMWPE. Moreover, as shown in Table I, the inclusion of the Cu filler led to a decrease in T_m of UHMWPE, whereas the doping of PE-g-MAH further reduced T_m to some extent. This was supposed to be attributable to the reduction of the crystallinity percentage of UHMWPE by the Cu filler incorporation.

Mechanical and tribological properties

Figure 3 shows the variation of the mechanical properties of UHMWPE/Cu composites with the volume fraction of the Cu filler. The tensile rupture strength of the composites decreased with increasing Cu content [see Fig. 3(a)]. This was because the inclusion of the Cu filler caused a discontinuity in the microstructures of the UHMWPE-matrix composites. At the same time, the UHMWPE/Cu composites compatibilized with various volume fractions of PE-g-MAH (5–20%) had much larger tensile rupture strengths than the uncompatibilized ones. For example, the UHMWPE/25% Cu composite had a tensile rupture strength of 14.2 MPa, whereas the UHMWPE/10% PE-g-MAH/Cu composite had a tensile rupture strength of 19.8 MPa. Similarly, the incorporation of the PE-g-MAH compatibilizer also led to a significant increase in the tensile modulus of the UHMWPE composite [see Fig. 3(b)]. This occurred because PE-g-MAH, containing polar functional groups, contributed to greatly increased interfacial bonding of the UHMWPE composites. In contrast, the introduction of the Cu filler reduced the tensile modulus of the UHMWPE composites and decreased with increasing Cu content. This was because the inorganic Cu particulates linked to UHMWPE by mechanical interlocking alone caused damage to the continuity of the polymer matrix and weakened the polymeric intermolecular interactions; this further re-

TABLE I
 T_m and ΔH_m of UHMWPE-Based Composites

UHMWPE/ PE-g-MAH/Cu (vol/vol/vol)	UHMWPE T_m (°C)	UHMWPE/ PE-g-MAH ΔH_m (J/g)
100/0/0	139.5	166.41
75/0/25	132.8	22.04
70/5/25	128.8	29.12
65/10/25	128.5	34.06

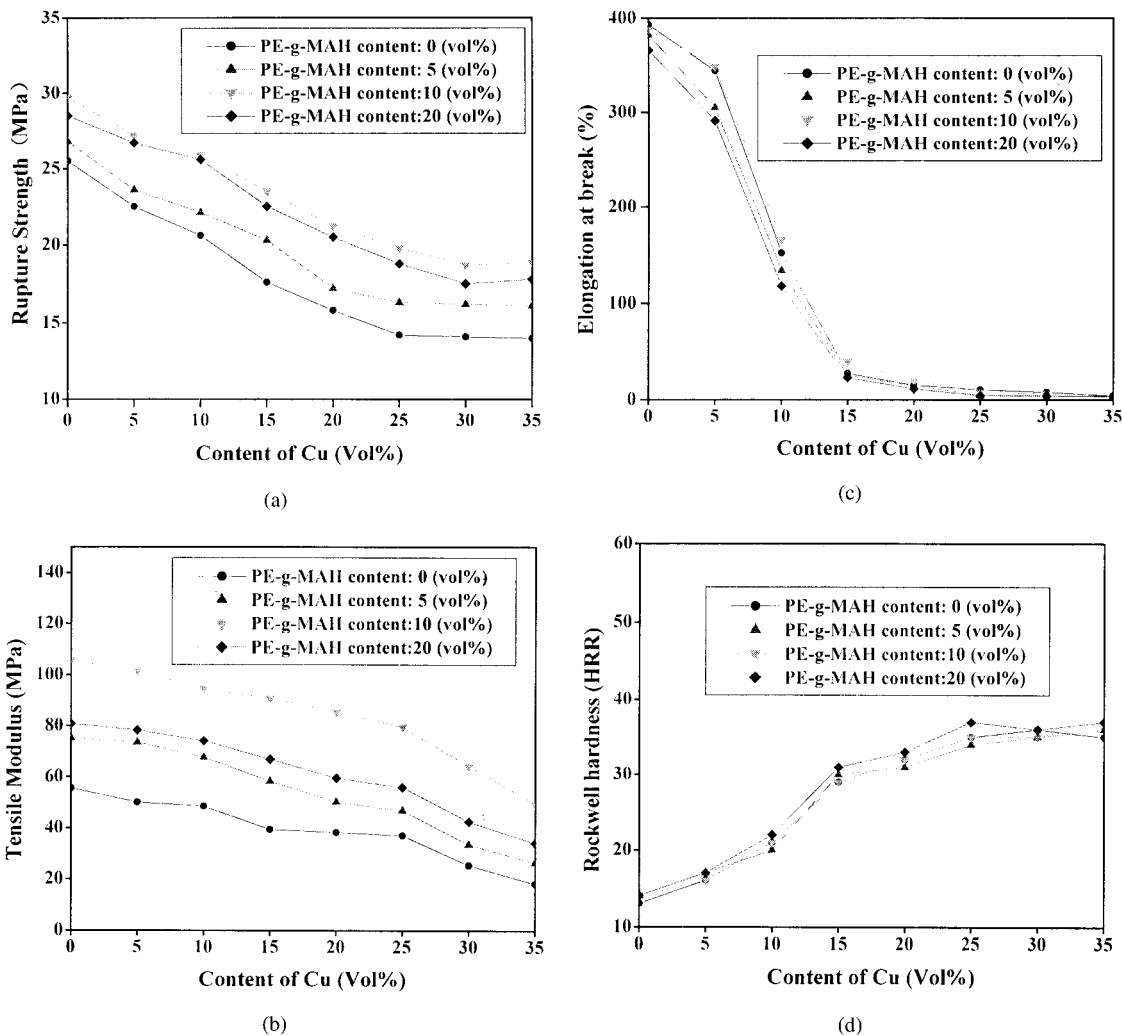


Figure 3 Mechanical properties of the UHMWPE/PE-g-MAH/Cu composites: (a) tensile rupture strength, (b) tensile modulus, (c) rupture elongation, and (d) Rockwell hardness (HRR)..

duced the bonding strength of the polymer-based composites. The rupture elongation values of the composites decreased with increasing Cu content when the PE-g-MAH compatibilizer was fixed at 10 vol % [see Fig. 3(c)]. The HRR of the composites increased with increasing Cu content, and the addition of the PE-g-MAH had little influence on the hardness [see Fig. 3(d)]. In summary, the incorporation of the PE-g-MAH compatibilizer increased the tensile rupture strength and tensile modulus of the UHMWPE/Cu composites; this was especially true at a PE-g-MAH volume fraction of 10%. Such a finding is similar to what has been observed for UHMWPE/polyurethane composites.²⁵ The 10% PE-g-MAH yielded higher mechanical properties than 20% PE-g-MAH, and this may be attributed to the effect of the compatibilizer on the interfacial bonding of the composites. That is, optimized interfacial bonding was realized when the volume fraction of PE-g-MAH was fixed at 10%; otherwise, the interfacial bonding was weaker. The

mechanism of the effect of the compatibilizer in polymer composites demands further investigation in future work.

Figure 4 shows the variations of the wear rate and friction coefficient of the UHMWPE/Cu composites sliding against SAE52100 steel with the volume fraction of the Cu filler. The wear rate and friction coefficient decreased with increasing Cu content until they assumed stabilized values at a Cu volume fraction of 25 vol %. This observation conforms well to what has been reported on poly(tetrafluoroethylene)/metallic composites²⁰ and can be attributed to the reinforcing and modifying effects of the particulate fillers. In other words, the Cu particles in the UHMWPE matrix functioned as solid lubricants, reducing friction, and as reinforcing agents, increasing the ability of the composites to resist shearing in the sliding process; this contributed to improves friction-reducing and wear-resistant behaviors of the UHMWPE-matrix composites.

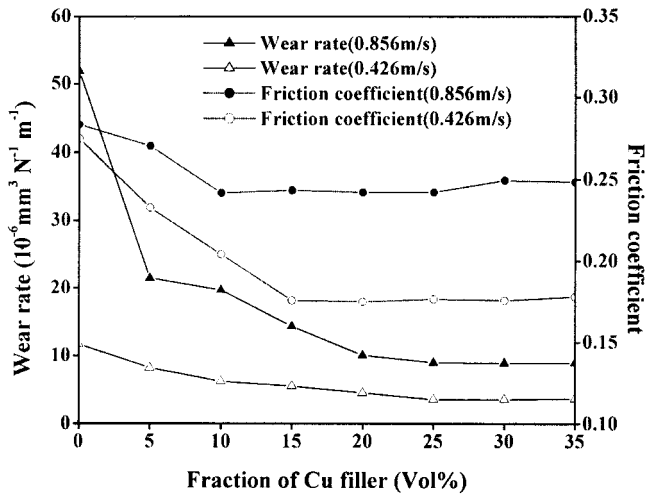
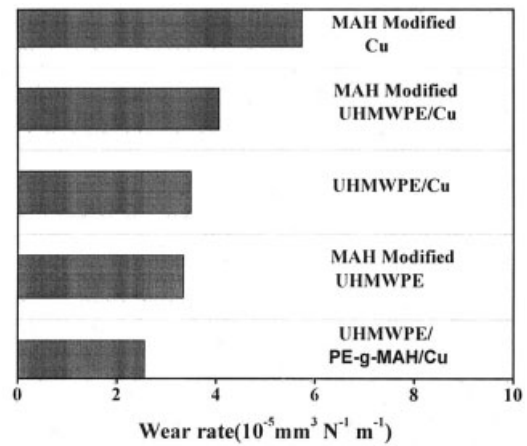


Figure 4 Variations of the wear rates and friction coefficients of UHMWPE-matrix composites with the Cu filler content.

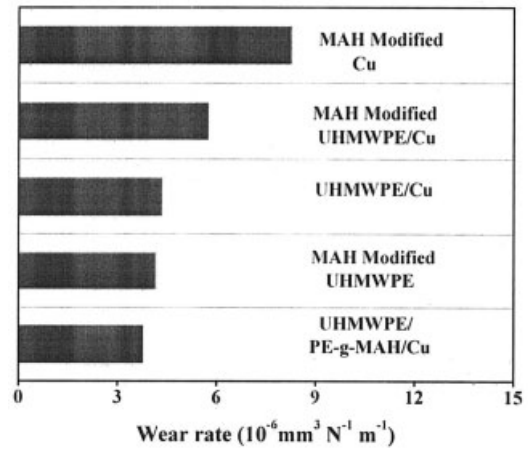
Figure 5 illustrates the effects of various modification patterns on the friction and wear behaviors of the UHMWPE-matrix composites. The UHMWPE/Cu/PE-g-MAH composites subjected to different modifying patterns had small wear rates, but the maleic anhydride surface modification of the Cu filler and the UHMWPE/Cu blends led to larger wear rates of the UHMWPE-matrix composites. Moreover, the wear rates of the UHMWPE-matrix composites modified in different manners showed little dependence on the sliding velocity.

Figure 6 shows the variations of the friction coefficients and wear rates of the UHMWPE/PE-g-MAH/Cu composites with the PE-g-MAH content. The ternary composites containing 5–10 vol % PE-g-MAH had almost the same wear rates as that of the binary composite UHMWPE/25% under a normal load of 200 N but recorded smaller friction coefficients than the uncompatibilized binary composite. Specifically, the ternary composite containing 10 vol % PE-g-MAH registered a friction coefficient as small as about half of that of the uncompatibilized binary composite. This is supposed to be attributable to the improved interfacial adhesion between the UHMWPE matrix and the Cu particulate filler in the presence of the PE-g-MAH compatibilizer, which was able to increase the interfacial bonding through its polar functional groups.

Figure 7 shows the variations of the wear rates and friction coefficients of UHMWPE/Cu and UHMWPE/PE-g-MAH/Cu composites with a normal load at a sliding velocity of 0.856 m/s. The wear rates of the UHMWPE/Cu and UHMWPE/PE-g-MAH/Cu composites increased with increasing applied load until they assumed nearly constant values at loads above 250 N. The friction coefficients for the two kinds of



(a)



(b)

Figure 5 Effects of various modifying patterns on the wear rates of UHMWPE-matrix composites at sliding velocities of (a) 0.424 and (b) 0.856 m/s.

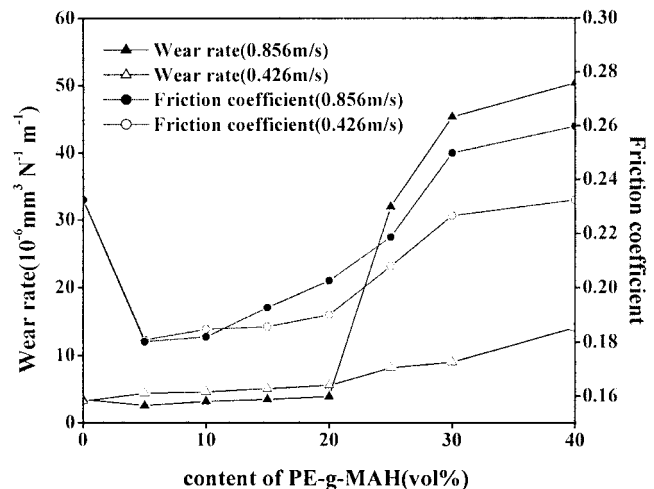


Figure 6 Variations of the friction coefficients and wear rates of UHMWPE-matrix composites with the PE-g-MAH content.

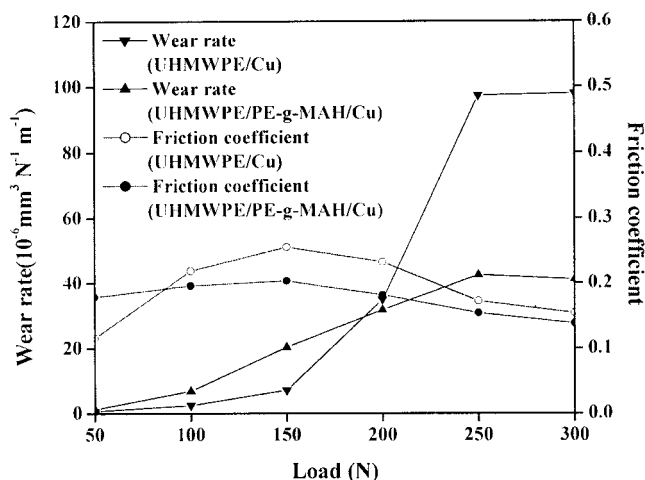


Figure 7 Variations of the wear rates and friction coefficients of UHMWPE/Cu and UHMWPE/PE-g-MAH/Cu composites with a normal load at a sliding velocity of 0.856 m/s.

UHMWPE-matrix composites also increased with increasing applied normal load and reached the maximum at a normal load of 150 N, and then the friction coefficients decreased to some extent with a further rise in the normal load up to 300 N. Like what is shown in Figures 5 and 6, the compatibilized ternary composite sliding against steel at 250 N had a small wear rate, about half of that of the uncompatibilized binary composite. In combination with the results shown in Figures 5–7, it is concluded that the compatibilized composites exhibited better friction-reducing behavior and wear resistance than the uncompatibilized one at relatively larger loads and sliding velocities.

Figure 8 shows typical scanning electron micrographs of the worn surfaces of UHMWPE/25% Cu and UHMWPE/10% PE-g-MAH/Cu composites at a

normal load of 300 N and a sliding velocity of 0.856 m/s. The UHMWPE/Cu composite was characterized by severe adhesion wear and plastic deformation as it slid against the steel counterpart [see Fig. 8(a)], whereas the UHMWPE/PE-g-MAH/Cu composite was dominated by mild adhesion wear and slight scuffing [see Fig. 8(b)]. This observation conformed well to the corresponding friction and wear behaviors of the UHMWPE/Cu and UHMWPE/PE-g-MAH/Cu composites.

X-ray photoelectron spectroscopy (XPS) analysis of worn surfaces of UHMWPE-matrix composites and steel counterparts

Figure 9 shows the Cu2p XPS spectra of the worn surfaces of UHMWPE/Cu and UHMWPE/PE-g-MAH/Cu composites and the corresponding worn surfaces of the steel counterparts. Only elemental Cu was detected by XPS on the Ar⁺-sputtered worn surfaces of the counterpart steel sliding against both the binary UHMWPE/Cu and ternary UHMWPE/PE-g-MAH/Cu composites [see Fig. 9(a,d)]. Elemental Cu was also detected by XPS analysis on the unsputtered worn steel surface sliding against the UHMWPE/Cu composite and on the worn UHMWPE/Cu surface [see Fig. 9(b,c)], but in this case, the intensities of the XPS Cu2p peaks were much smaller than that of the Ar⁺-sputtered worn steel surface. Moreover, as shown in Figure 9(b), XPS peaks assigned to both Cu²⁺ and Cu⁰ (934.5 and 932.4 eV, respectively) were detected on the worn steel surface sliding against UHMWPE/Cu by XPS. At the same time, the elemental Cu particulates incorporated into the binary UHMWPE/Cu composite remained chemically unchanged during the sliding process [see Fig. 9(c)], although the intensity of the XPS Cu2p peak of the worn surface of the UHMWPE/Cu composite was larger than that of

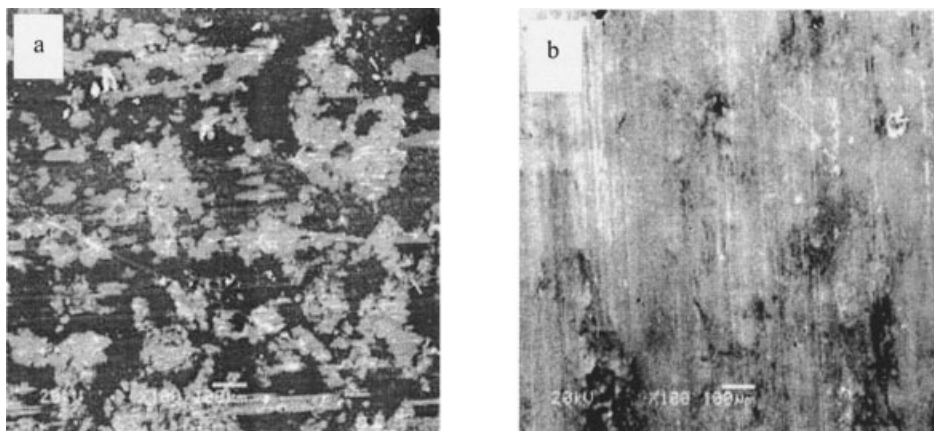


Figure 8 Scanning electron images of (a) UHMWPE/Cu composites and (b) UHMWPE/10% PE-g-MAH/Cu composites at a normal load of 300 N and a sliding velocity of 0.856 m/s.

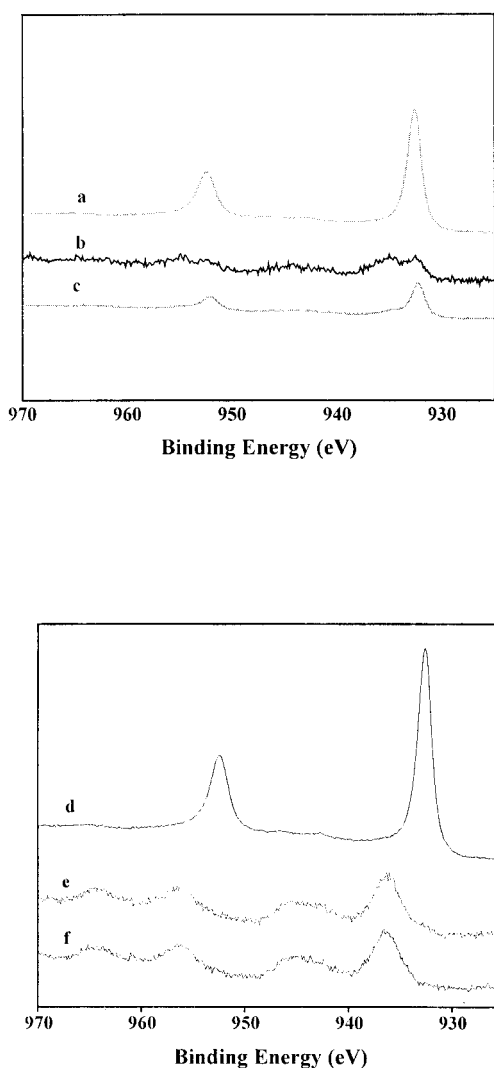


Figure 9 XPS spectra of Cu₂p of (a) a worn steel surface against a UHMWPE/Cu composite after Ar⁺ sputtering for 5 min, (b) a worn steel surface against a UHMWPE/Cu composite, (c) a worn surface of a UHMWPE/Cu composite, (d) a worn steel surface against a UHMWPE/PE-g-MAH/Cu composite after Ar⁺ sputtering for 5 min, (e) a worn steel surface against a UHMWPE/PE-g-MAH/Cu composite, and (f) a worn surface of a UHMWPE/PE-g-MAH/Cu composite

the worn steel surface [see Fig. 9(b,c)], whereas the intensity of the Cu₂p peak on the worn steel surface increased considerably after Ar⁺ sputtering. It is therefore concluded that the elemental Cu particulates in the UHMWPE/Cu composite were able to transfer onto the counterpart steel surface and experience oxidation to generate CuO as well. Such oxidation might be localized on the surface layer of the transfer film, and this was confirmed by the corresponding XPS analysis of the Ar⁺-sputtered worn steel surface, on which only elemental Cu existed [see Fig. 9(a)]. In contrast, only Cu²⁺ was detected on the worn steel surface sliding against the UHMWPE/PE-g-MAH/Cu

composite and on the worn composite surface itself [see Fig. 9(e,f)]; this is totally different from what is shown in Figure 9(c,d) and somewhat different from what is shown in Figure 9(b). In other words, both Cu²⁺ and Cu⁰ existed on the worn steel surface against the UHMWPE/Cu composite, only Cu⁰ existed on the worn UHMWPE/Cu surface, and only Cu²⁺ was detected by XPS on both the worn steel surface and the corresponding UHMWPE/PE-g-MAH/Cu surface. This implies that PE-g-MAH as a compatibilizer might have an effect on the transfer and oxidation behaviors of Cu particulate fillers and hence on the friction and wear behaviors of the UHMWPE-matrix composites. That is, the Cu particulates were weakly linked with the polymer matrix when the composite was not doped with the compatibilizer, and so the filled Cu particulates were more liable to be peeled off and to transfer onto the worn steel surface; they had less opportunity to be oxidized because of the easier successive peeling and transfer of the UHMWPE/Cu composite [see Fig. 9(b)]. This observation was also applicable to the worn surface of the UHMWPE/Cu composite; before the peeled-off Cu particulates were oxidized, freshly exposed and detached Cu particulates were involved in the successive sliding [see Fig. 9(c)].

In contrast, the Cu particulates incorporated into the ternary UHMWPE/PE-g-MAH/Cu composite were strongly linked to the UHMWPE matrix because of the enhanced crosslinking in the presence of the compatibilizer; this made it more difficult for the Cu particulates to be peeled off and transfer onto the counterpart steel surface. In other words, the exposed and peeled-off Cu particulates on the worn surface of the UHMWPE/PE-g-MAH/Cu composite or the transferred Cu particulates on the counterpart steel surface sliding against the UHMWPE/PE-g-MAH/Cu composite had more opportunities to experience oxidation because far fewer Cu particulates were peeled off and transferred in this case in comparison with the sliding of the UHMWPE/Cu composite against the steel because of the better mechanical strength of the UHMWPE/PE-g-MAH/Cu composites, which could be attributed to the crosslinking reinforcing action of the compatibilizer. The deterred transfer with respect to the UHMWPE/PE-g-MAH/Cu composite conformed to its wear resistance being better than that of the UHMWPE/Cu composite; this was especially true at larger normal loads.

CONCLUSIONS

The incorporation of PE-g-MAH as a compatibilizer significantly improved the mechanical strength and wear resistance of UHMWPE-matrix composites filled with Cu particulates, and it was closely related to the crosslinking reinforcing action of the compatibilizer.

The UHMWPE/PE-g-MAH/Cu ternary composite with 10 vol % compatibilizer and 25 vol % Cu filler had the best wear resistance among the tested composite specimens. Thus, we suggested setting the critical volume fraction of PE-g-MAH in the UHMWPE/PE-g-MAH/Cu composite at 10%. PE-g-MAH as a compatibilizer affected the transfer and oxidation behavior of the Cu particulates as well. The Cu particulates, relatively weakly linked to the UHMWPE matrix in UHMWPE/Cu, were more liable to be peeled off and had less chance of experiencing oxidation because of the easier successive peeling off and transfer, whereas the Cu particulates strongly connected to the UHMWPE matrix in the ternary UHMWPE/PE-g-MAH/Cu composite were harder to peel off because of the crosslinking effect of the compatibilizer. The peeled and transferred ones had more opportunities to experience oxidation because the successive peeling off and transfer of the Cu particulates in this case were significantly deterred on account of the better mechanical strength of the UHMWPE/PE-g-MAH/Cu composite.

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