

The mechanical and tribological properties of nitric acid-treated carbon fiber-reinforced polyoxymethylene composites

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ABSTRACT: The carbon fibers have been exposed to nitric acid oxidation treatments and introduced into polyoxymethylene composites (POM/CF). The nitric acid treatment increases the number of the flaws, roughness of the surface, and disorder of carbon atoms on fiber, as well as introduces reactive functional groups, which could lead to a better mechanical bonding between fiber and the matrix. It is shown that the impact strength and fiber-matrix adhesion in composites (POM/mCF) are superior to those for POM/CF composites. Simultaneously, the addition of mCF improves flexural strength and modulus relative to virgin POM significantly. Average friction coefficient values of POM/CF composites are lower than that of POM/mCF composites. As the percentage of fiber increases, the trend of wear ratio of the composites goes down initially and bumps up afterwards. The results indicate that the proper contents of CF and mCF in composites range from 5 wt % to 20 wt %. Scanning electron microscopy of worn surface morphology has revealed that the main wear mechanism of the composites were adhesive wear and ploughing wear. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2015**, *132*, 41812.

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INTRODUCTION

Carbon fibers (CF) present exceptional properties, such as high stiffness, high specific strength, and outstanding wear resistance, which have attracted considerable research interest in exploiting them as excellent reinforcements for composite materials. Polymer-matrix composites reinforced with carbon fibers have been widely used in tribological applications such as bearing, slides, gears and seals, due to their excellent mechanical properties, low friction against metal counterparts, and the ability of self-lubricating.^{1–4} Previous studies on carbon fiber composites were mainly focused on thermosetting resin.^{5,6} However, thermoplastics composites are designable, ease to be processed and more convenient for recycling.^{7,8} Many researchers have made efforts in understanding the modifications in the tribological behavior of polymers with the addition of fiber reinforcements.^{9,10}

Polyoxymethylene (POM) is an excellent engineering plastic, and also a substitute of metals in many occasions.^{11,12} It has low friction coefficient due to the flexibility of the linear molecular chains and good wear resistant properties because of its high crystallinity and high bond energy. Efforts are devoted in exploring the mechanical and tribological properties of POM to fully exploit its application. Both inorganic and organic additives have been selected to modify POM, such as PTFE, MoS₂, calcium carbonate,

talc, diatomite, clay, and glass fibers.^{13–15} Most of the fillers mentioned above cannot improve mechanical and tribological properties simultaneously, over which carbon fiber has advantages. It can significantly improve the strength of the composites, and the incorporation of CF in POM enhances the wear resistance greatly: as the CF content increased, the wear decreased.¹⁶

There is considerable interest in investigating the mechanical and tribological properties of carbon fiber-reinforced polymer composites.^{17,18} Nonetheless, the use of carbon fibers in POM composites is problematic because of their relatively unreactive surface, which leads to potential issue, like inadequate adhesion with the matrix.¹⁹ Several types of surface treatments have been reported to overcome these drawbacks, among which nitric acid oxidation treatment is an effective way to strengthen adhesion through enhancing physical bonds by roughening and involving more surface contact area, as well as introducing reactive functional groups.^{20,21} Some basic research^{22–24} studied the influence of nitric acid oxidation on the interfacial properties of carbon fiber-reinforced polymer composite and investigated the nature of the chemical functionalities created. This paper, which is on the basis of previous research, is hoped to do a more comprehensive study.

This study presents a clear comparison of nitric acid-treated carbon fiber and virgin carbon fiber-reinforced POM composites

with respect to the mechanical properties and tribological behaviors. The disorder degree and the functional change of carbon fiber, the fiber length distribution, dynamic mechanical analysis, and the main wear mechanisms were also investigated. It is helpful to a better understanding of the role of improved interfacial adhesion on the overall properties of polymer composites.

EXPERIMENTAL

Materials

The carbon fibers used in this study were T700SC PAN-based 12 K tow fibers purchased from Toray Industries, Inc. Granular POM (M90-44, MFR = 9.0 g/10 min, injection molding grade) was purchased from Polyplastics Nitric acid (AR grade, 65%) was supplied by Sinopharm Chemical Reagent.

Surface Treatments of Carbon Fiber

Acid treatment was accomplished by immersing fiber in nitric acid (concentration of 65%) under an optimum treatment condition (at 25°C for 24 h). After acid exposure, the fibers (mCF) were again washed repetitively with distilled water, until the pH of the wash water was 7.

Composites Preparation

POM was mixed with carbon fibers (10 mm in length) in a Haake batch mixer (Thermo Fisher Scientific, USA) at optimized temperature of 200°C for 10 min, 50 rpm. The fiber content was 5, 10, 20, and 30 wt % for comparison. After preheated at 210°C for 8 min, compounds from Haake mixer were compression molded into several 100 mm × 100 mm × 2 mm composite sheets with a molding temperature of 210°C and a molding pressure of 15 MPa. Water jet cutting machine was used to cut samples into tested size.

Characterization of the Composites

Scanning Electron Microscope. The surface of carbon fiber before and after nitric oxidation treatment, the impact section morphology and the fractured surface morphology analysis of the samples were performed with Nova NanoSEM 450 scanning electron microscope (FEI company), with acceleration voltage of 5 kV. The samples of composites were sputter-coated with gold before testing.

Raman Spectroscopy. Raman spectroscopy was carried out using a Senterra R200-L dispersive Raman microscope (Bruker Optics, GER). A He–Ne laser was used to give monochromatic red light of 532 nm wave length at a power of 5 mW at the specimen. The band intensity, band width, and band position were obtained using a Lorentzian curve-fitting procedure.

Fourier-Transform Infrared Spectroscopy. Fourier-transform infrared (FTIR) spectroscopy of the composites were measured in Paragon 1000 spectrometer (Perkin Elmer), using KBr compression method, scan range 450–4000 cm^{-1} , resolving power of 0.1 cm^{-1} .

Fiber Length Distribution Measurement. The fiber length distribution of composites was measured using polarizing microscope (Leica Microsystems GmbH, Germany) after a patch of composite heated in an electric box furnace at 600°C for 15 min to decompose resin. A drop of glycerol was added afterwards in the middle of the slide and a small amount of fiber was evenly dispersed in glycerin. The specimen covered by the

slide was put on the polarizing microscope to observe and take photographs. The WinROOF Image Analysis & Measurement Software (Mitani Corporation, Japan) was used to measure the length of carbon fiber and at least 500 data were calculated.

Mechanical Performance Test. Unnotched Izod impact test was done on Izod impact testing machine (RAY-RAN Test Equipment, UK) with a pendulum according to ISO180-2000. Flexural properties were evaluated at a strain speed of 2 mm/min using a universal material testing machine (MTS Company) according to ISO178–2011. Five samples of each group were tested to get the average strength and modulus of the composites. Dynamic mechanical analysis of the composites was performed using a DMA 242C (NETZSCH Germany) in the temperature range of –100°C to 150°C with a heating rate of 4°C/min, the experimental frequency range was 1 Hz and the loading regime was three point bending.

Tribological Test. Tribological test was conducted on an MFT-R4000 Reciprocating friction and wear tester (Lanzhou Institute of Chemical Physics, China) using a pin with several values of surface roughness as the reciprocating specimen. The frequency was 10 Hz and the applied load was 150 N. The testing time was 1 h.

RESULTS AND DISCUSSION

Characterization of Fiber

Commercial carbon fiber tow or yarn contains thousands of carbon fibers in a sing tow and the surface of fiber was smooth [Figure 1(a)]. After nitric acid treated, the sizing agent disappears and fibers become relatively soft and loose [Figure 1(b)]. The surface of virgin carbon fiber is smooth because of the existence of epoxy coating [Figure 1(c)]. HNO_3 treatment appears to have significant effect on the creation of surface flaws [Figure 1(d)]. Fiber surface roughness can affect both the fiber strength and composite fiber/matrix bonding, increased roughness weakens fibers through stress concentrations, but also allows for better mechanical bonding to the matrix.²⁵

Figure 1(e) shows the Raman spectroscopy of virgin carbon fiber and nitric acid-treated carbon fiber. According to the electronic characteristics of carbon fiber, the common features of carbon fiber in Raman spectroscopy are in the region of 800–4000 cm^{-1} in wavelength. We focus on two major peaks around 1360 cm^{-1} (attributed to a crystallite size effect) and 1580 cm^{-1} (arising from the infinite crystal of fiber), so-called *D* and *G* bands, respectively.^{26,27} Previous studies have shown that the integrated intensity of the *D* band increases as the crystallite size decreases in carbon fiber, and the ratio ($R = I_D/I_G$) of the integrated intensity of the *D* band (I_D) to that of the *G* band (I_G) has been used to give an indirect measure of the disorder degree of carbon fiber. The band integrated intensities were obtained using a Lorentzian curve-fitting procedure. The results show that after the treatment, the peak shape of *D* band becomes broader and the values of *R* improve from 3.71 to 4.03. It indicates that nitric acid oxidation treatment reduces the crystallite size of fiber surface and the percentage of disordered carbon on the fiber surface increases.

Fourier-transform infrared (FTIR) spectroscopy is used to observe the functional change of carbon fiber with nitric acid

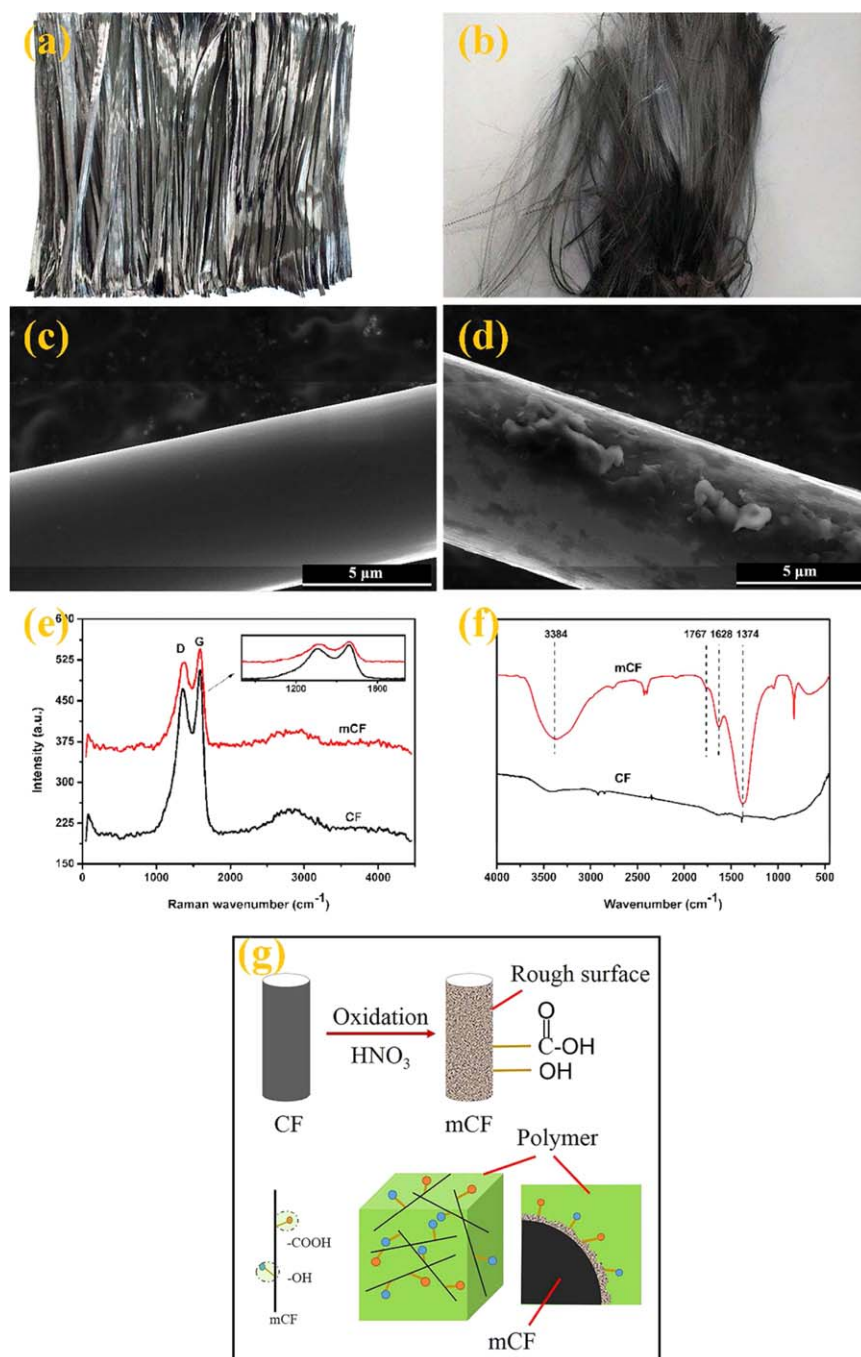


Figure 1. Macroscopic appearance of CF (a) and mCF (b); SEM images of the surface of CF (c) and mCF (d); Raman spectroscopy of CF and mCF (e); FTIR spectroscopy of CF and mCF (f); changes of fiber after acid treatment (g). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

treatment. The FTIR transmittance spectra of carbon fibers before and after modification are shown in Figure 1(f). As expected, after the oxidation treatment, strong absorption bands around 3384 cm^{-1} and 1374 cm^{-1} appear which represent the -OH group. The bands around 1628 cm^{-1} and 1767 cm^{-1} are assigned to the stretching vibrations of -C=O group. These observations confirm that nitric acid treatment can effectively enhance the fiber surface activity and introduce reactive functional groups.

In summary, the nitric acid treatment introduces reactive functional groups, increases the number of the flaws, roughness of the surface and disorder of carbon of fiber, which could lead to a better mechanical bonding between fiber and the matrix [Figure 1(g)].

Mechanical Properties

Static Mechanical Properties. The impact strength of POM/CF composites and POM/mCF composites show a downward trend

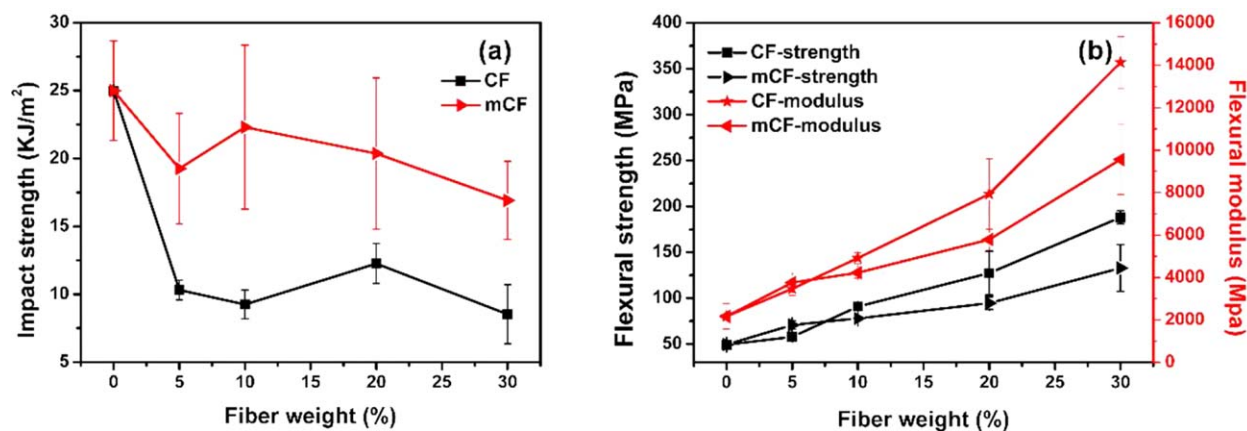


Figure 2. Impact strength (a) and flexural properties (b) of composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

overall with the increase of fiber content [Figure 2(a)]. Addition of carbon fibers decreases impact strength of POM-based material, which is caused by the second phase formed by inert carbon fiber. This second phase obstructs the continuity of the basic POM phase due to the lack of chemical bonding between fibers and the matrix. The impact strength of POM/mCF composites with the same percentage composition of fiber are higher than that of POM/CF composites. The nitric acid treatment alters fiber's surface roughness to increase bonding strength between fibers and matrix which is proved by the SEM images of POM/CF(80/20) and POM/mCF(80/20) composites (Figure 3). It is much harder for mCF to be pulled out from the matrix.

The flexural properties of POM/CF and POM/mCF composites are shown in Figure 2(b). It is found that the flexural strength and modulus of carbon fiber-reinforced composites increase with increasing fiber content, because matrix can transmit stress to carbon fiber through interface, and carbon fibers with high modulus can withstand stress significantly better than the POM matrix.²⁸ Flexural strength and modulus of composites are determined by fiber properties and the interface bonding between carbon fiber and resin. In normal condition, the strength and modulus of carbon fibers reduce after nitric acid oxidation treatment,²⁹ which lead to the decrease of strength and modulus of POM/mCF composites. In addition, the average fiber length in the composites plays an important role.

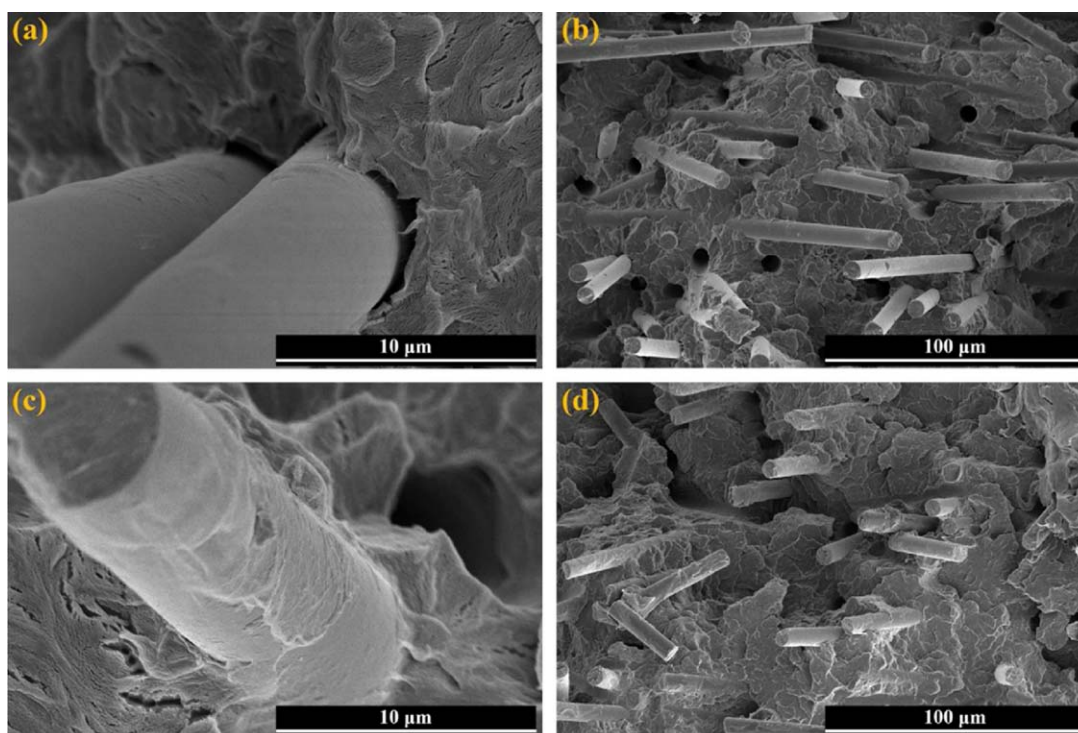


Figure 3. Impact rupture surface of POM/CF(80/20) (a, b) and POM/mCF(80/20) (c, d). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

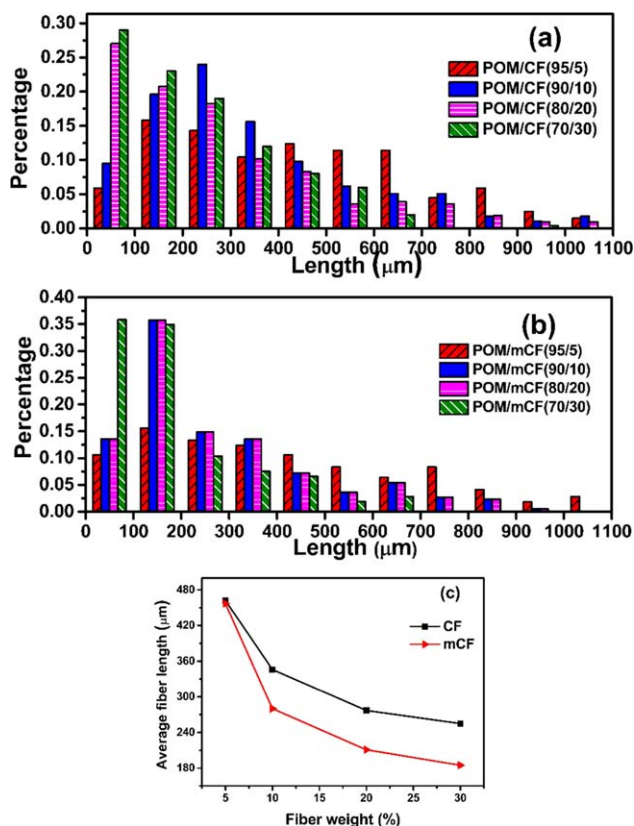


Figure 4. Fiber length distribution (a, b) and average fiber length (c) of composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table I. Tan δ Peak Height for Different Composites

Sample	Peak height	Sample	Peak height
Neat POM	0.0159		
POM/CF(95/5)	0.0158	POM/mCF(95/5)	0.0146
POM/CF(90/10)	0.0158	POM/mCF(90/10)	0.0141
POM/CF(80/20)	0.0127	POM/mCF(80/20)	0.0098
POM/CF(70/30)	0.0108	POM/mCF(70/30)	0.0107

Figure 4 illustrates that the average fiber length reduces with the increase of CF and mCF content. The latter is shorter, because after nitric acid treatment, the fiber's strength reduces which makes it easier to break. In the same case of fiber content, flexural strength enhances with increase in fiber length.^{30–34} The average length of mCF in composites is shorter than that of CF, the former contain more fiber ends and more micro-crack exist in the matrix.

Dynamic Mechanical Properties. Dynamic mechanical analysis (DMA) is one of the most powerful tools to study the behavior of plastic and polymer composite materials.³⁵ The storage modulus, often related to the Young's modulus, describes the stiffness of a material.³⁶ Figure 5(a,b) illustrates the DMA plots of storage modulus versus temperature of composites. The addition of fiber to the matrix increases the storage modulus value, because the movements of the polymer chains are restricted by interactions with fibers, which allows greater stress transfer at

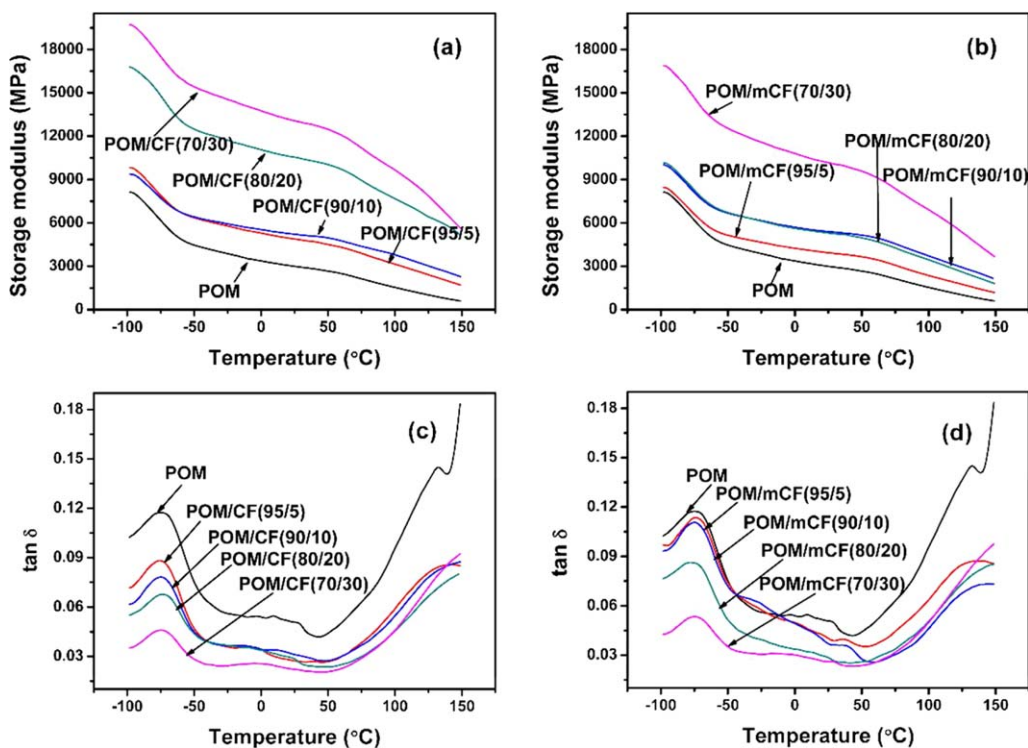


Figure 5. Storage modulus of POM/CF composites (a) and POM/mCF composites (b); the damping factor ($\tan \delta$) curve of POM/CF composites (c) and POM/mCF composites (d). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

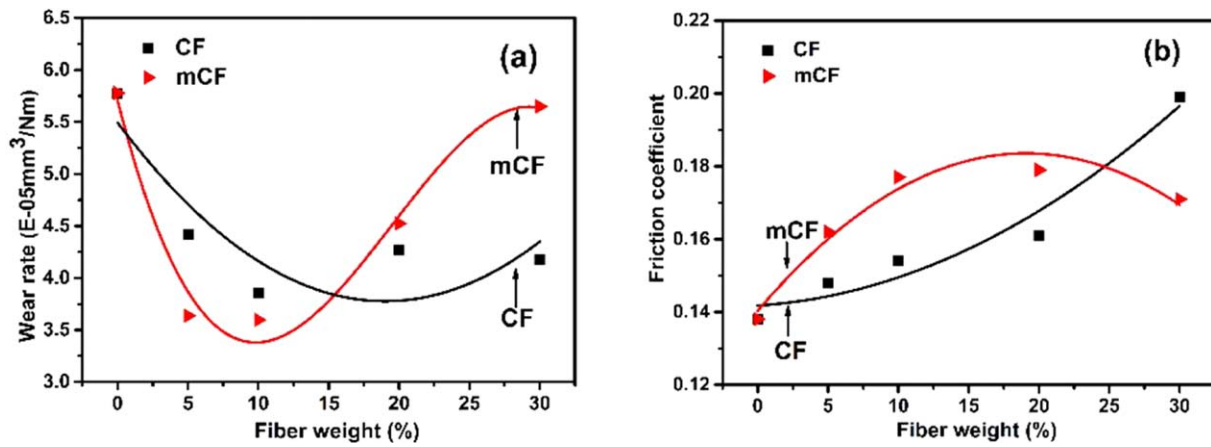


Figure 6. (a) wear rate (b) friction coefficient of composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the interface and hence the increase of the stiffness.^{37,38} The POM composites with mCF show a smaller storage modulus when compared with POM/CF composites which is consistent

with the results of flexural strength and modulus. The reason could be that the strength and modulus of carbon fiber reduce after the nitric acid treatment.

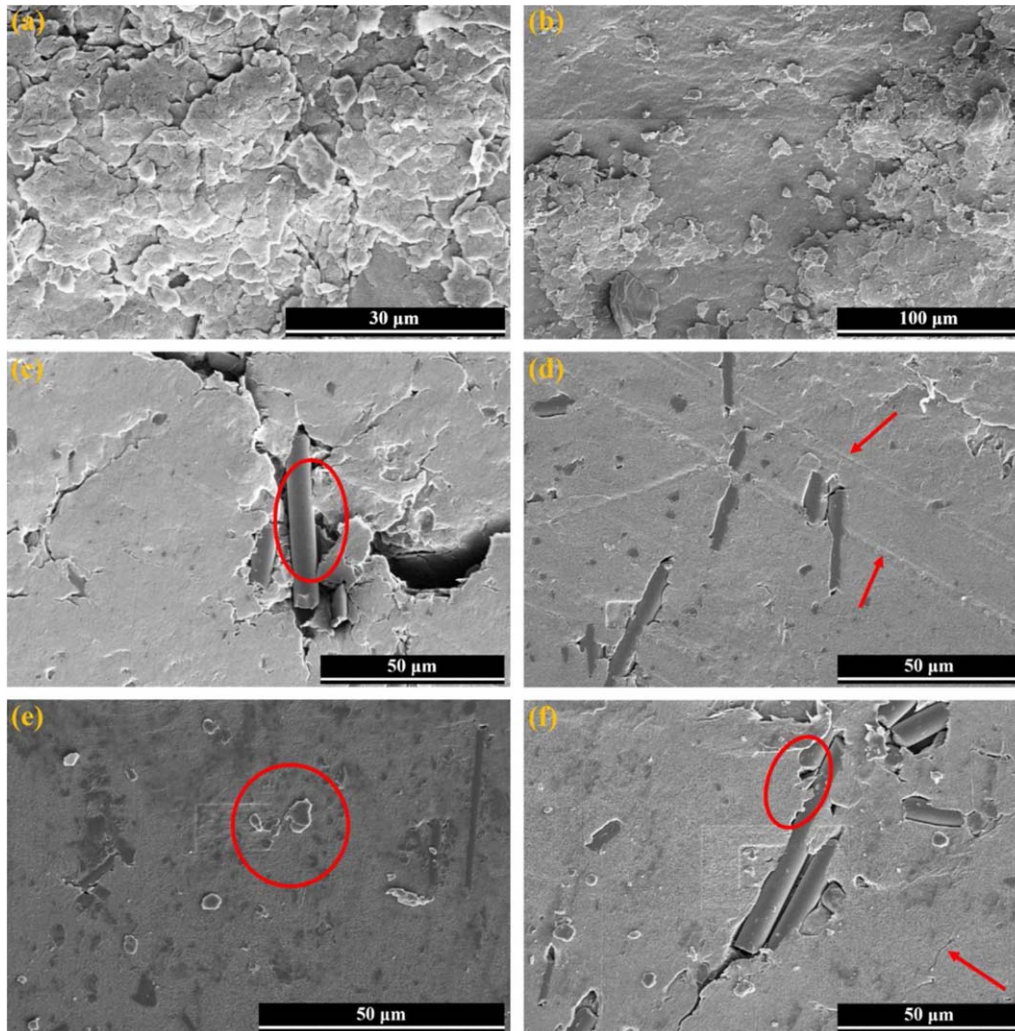


Figure 7. Worn surfaces of virgin POM and composites with 10 wt % fiber content: POM (a, b); POM/CF (c, d); POM/mCF (e, f). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The ratio of loss modulus to storage modulus ($\tan \delta$) is an important parameter in relation to the dynamic behavior of fiber-reinforced composites.³⁹ The variation in $\tan \delta$ for neat POM and composites as a function of temperature is represented in Figure 5(c,d). The damping factor values for the composites are lower than those for the neat POM. The incorporation of carbon fibers reduce the height of the $\tan \delta$ peaks (Table I). This is because the fibers carry a greater extent of stress and allow only a small part of it to strain the interface.^{40,41} When the fiber contents are the same, the peak height of POM/mCF composites are lower than those of POM/CF composites. Composites with poor interfacial bonding between the fibers and matrix will tend to dissipate more energy and a lower peak height may be associated to a good interface. Therefore, the results clearly show that nitric acid treatment contributes to a good fiber-matrix adhesion, in which case effective stress transfer between the matrix and fiber takes place.⁴²

In thermoplastics, the $\tan \delta$ value of a polymer reaches a maximum when the polymer is heated up to the glass transition temperature (T_g). It can be seen that T_g values of carbon fiber-reinforced composites nearly do not change with the increasing CF or mCF content.

Friction and Wear Performance

The tribological properties of composites were measured on a reciprocating friction and wear tester using a pin with several values of surface roughness as the reciprocating specimen. It is observed that the friction coefficients of composites increase with carbon fiber content while the POM/mCF(90/10) has the lowest wear ratio at all the conditions investigated (Figure 6).

It is generally accepted that the wear behavior of short fiber-reinforced composites is dominated by the key process of fiber peeling-off, which typically occurs through such sequential stages: fiber thinning, fiber cracking and fiber removal. Longer carbon fiber-reinforced composites exhibit better wear resistance in comparison to the shorter ones, because longer fibers are more difficult to be peeled-off from the matrix.⁴³ When fiber content is 5 wt %, 10 wt %, 20 wt %, and 30 wt %, the average fiber length of POM/CF composites is 462, 346, 277, and 255 μm , respectively. The length of fiber in POM/CF(95/5) is relatively longer, micro interspace exists between fiber and matrix, crack germinates and develops in the stress concentration place first, and makes the friction components fall off resulted in the rise of wear rate. As for POM/CF(80/20) and POM/CF(70/30), fibers are shorter and wreck continuity of the matrix, thus friction components are easier to fall off compared with POM/CF(90/10). The results indicate that the proper contents of CF and mCF in POM range from 5 wt % to 20 wt %.

The improved wear resistance may be also attributed to better adhesion of the fiber with the matrix, since higher fiber content in the bulk leads to an increase amount of fiber distributed on the friction surface and carbon fiber has good self-lubricating and antifriction characteristics. After nitric acid treatment, however, the self-lubricating property of fibers reduces, which results in the increase of friction coefficient of POM/mCF composites. The adhesive strength of fiber/matrix affects wear mass loss significantly when fiber content is low. With elevated fiber content,

the self-lubricating and antifriction characteristics of fiber play a more important role. Hence, the wear ratios of POM/CF composites are lower.

In terms of SEM observations of worn surfaces, the main wear mechanisms of POM and composites are abrasive wear [Figure 7(e)] and ploughing wear [Figure 7(d)]. There are more abrasive grains on the worn surface of POM [Figure 7(a,b)] than that on the composites. Furthermore, mCF is more difficult to be peeled-off from the matrix [Figure 7(f)]. This effect further causes the improved wear resistance and lower wear ratio.

CONCLUSIONS

In this study, the mechanical and tribological properties of POM/CF and POM/mCF composites are investigated. The roughness of the surface and the increased disordered carbon on the surface of nitric acid-treated fiber are proved, as well as the introduction of reactive functional groups. The nitric acid treatment alters fiber's surface roughness in a way that enhances the impact strength of the composites. Simultaneously, the addition of mCF improves flexural strength and modulus relative to virgin POM significantly. The $\tan \delta$ peak heights of POM/mCF composites are lower than that of POM/CF composites, which indicates a good adhesion between the fiber and the matrix. The POM/mCF(90/10) has the lowest wear ratio at all the conditions investigated, showing that the incorporation of mCF in POM improves the wear resistance and reduces the wear ratio. The researches in this work are promising to provide a simple way, which is suitable for industrial application, to manufacture nitric acid modified carbon fiber/POM composites. Such composites with good mechanical and tribological performances, could be better used in tribological applications.

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