

## Research on the Tribological Performance of Cr<sub>2</sub>O<sub>3</sub> Filled with Bronze-Based PTFE Composites

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**ABSTRACT:** Enhancement of the wear resistance of bronze-filled polytetrafluoroethylene (PTFE) composites has been achieved using various fillers, for example, chromic oxide (Cr<sub>2</sub>O<sub>3</sub>), molybdenum disulfide (MoS<sub>2</sub>), graphite, and nanometer aluminum oxide (n-Al<sub>2</sub>O<sub>3</sub>), in the present study. The wear resistance was evaluated by a block-on-ring wear tester, and the effects of fillers on the wear resistance as well as the mechanism were investigated. The wear rate for the composite where the recipe containing 59% PTFE + 40% bronze + 1% Cr<sub>2</sub>O<sub>3</sub> was  $0.5 \times 10^{-5} \text{ mm}^{-3}/\text{N m}$  and for the composite in the recipe containing 60% PTFE + 40% bronze was  $4.2 \times 10^{-5} \text{ mm}^{-3}/\text{N m}$ , which meant that that Cr<sub>2</sub>O<sub>3</sub> increased the wear resistance by approximately 10 times. The differential scanning calorimetry measure analysis showed that Cr<sub>2</sub>O<sub>3</sub> had a positive effect on the crystallization of PTFE; the crystallinity of PTFE composites increased from 45% to 52%, which exhibited improved wear resistance. Wear testing and scanning electron microscope analysis had shown that Cr<sub>2</sub>O<sub>3</sub> had a positive effect on the formation of transfer film and keeping it stable to exhibit improved wear resistance. X-ray photoelectron spectroscopy results also showed that Cr<sub>2</sub>O<sub>3</sub> was effective in tribochemical reactions during sliding against stainless ring; these maybe responsible for forming transfer film and lowering wear rate of composite. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 41117.

**KEYWORDS:** composites; crystallization; friction; resins; surfaces and interfaces; wear and lubrication

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### INTRODUCTION

Polytetrafluoroethylene (PTFE) is one of the high-performance engineering plastics, which is widely used in industry due to its properties of low friction coefficient, self-lubrication, high temperature stability, and chemical resistance. PTFE and its derivatives are probably the most widely used plastic class in sealing technology. The material has virtually universal chemical resistance, a very low friction coefficient, and excellent dry running properties.<sup>1,2</sup> However, PTFE exhibits poor wear and abrasion resistances, leading to early failure and leakage problem in machine parts. To solve this problem, various suitable fillers were added to PTFE. Generally, reinforcements such as glass fibers, carbon fibers, bronze, and solid lubricants were added internally or incorporated into the PTFE. In these fields, many investigations<sup>3–7</sup> have been reported that the friction coefficient can, generally, be reduced and the wear resistance can be improved when the PTFE were reinforced with bronze, glass, carbon, and fibers. Lancaster<sup>8</sup> had studied that addition of high aspect ratio filler materials (carbon fibers, glass fibers) to PTFE can improve its wear resistance because of the preferential load-

supporting action by these fibers. There have also been some reports on the use of particulate filler materials like MoS<sub>2</sub> and graphite to modify the tribological properties of PTFE.<sup>9–13</sup> The above researches suggested that incorporation of filler materials resulted in the enhancement of tribological performance.

Bronze-filled PTFE composites were widely used as reciprocating seals; large quantities (40–60 wt %) of bronze reduce deformation under load and high wear resistance. The addition of bronze to PTFE will increase the friction coefficient, while the introduction of MoS<sub>2</sub> and graphite will modify the friction coefficient and wear resistance.

Cr<sub>2</sub>O<sub>3</sub>, commonly used as a paint pigment, we proposed that it is more effective to the bronze-based PTFE composite. In this research, Cr<sub>2</sub>O<sub>3</sub> was filled with bronze-based PTFE composite, which enhanced wear resistance by approximately 10 times. There is very limited work on the tribological performance of Cr<sub>2</sub>O<sub>3</sub>-incorporated PTFE composites. We compared Cr<sub>2</sub>O<sub>3</sub> with other solid lubricants such as MoS<sub>2</sub>, graphite, and n-Al<sub>2</sub>O<sub>3</sub>. As of now, n-Al<sub>2</sub>O<sub>3</sub> is popular to improve the wear resistance of PTFE.

**Table I.** Recipe (wt %) of PTFE Composites

Designation	Materials
A	PTFE
B	60% PTFE + 40% bronze
C	99% PTFE + 1% Cr <sub>2</sub> O <sub>3</sub>
D	59% PTFE + 40% bronze + 1% Cr <sub>2</sub> O <sub>3</sub>
E	55% PTFE + 40% bronze + 5% Cr <sub>2</sub> O <sub>3</sub>
F	59% PTFE + 40% bronze + 1% MoS <sub>2</sub>
G	59% PTFE + 40% bronze + 1% graphite
H	59% PTFE + 40% bronze + 1% n-Al <sub>2</sub> O <sub>3</sub>

The purpose of this research is to study the tribological performance and mechanism of incorporation of bronze and Cr<sub>2</sub>O<sub>3</sub> during sliding into PTFE composite and to choose materials as seal materials for longer life. The ultralow wear system of filled bronze-based PTFE composite has been discussed in this article, which is used to understand tribochemical mechanism.

## EXPERIMENTAL

### Materials

The PTFE (type: M18F) powder with particle size of 25–40 μm was supplied by Dakin Co., Japan. The average particle size of bronze, Cr<sub>2</sub>O<sub>3</sub>, MoS<sub>2</sub>, graphite was 40 μm and they were supplied by Nan Jin Zhining New Style Materials Co., Ltd. The particle size of n-Al<sub>2</sub>O<sub>3</sub> was 30 nm and was supplied by Shenzhen JingCai Chem Co., Ltd.

Pure PTFE and seven different PTFE-based composites, listed in Table I, are studied in this research.

### Specimen Preparation

The fillers and PTFE were kept in an oven at 105°C for 3 h before the mixing process. Fillers and PTFE were prepared and blended in a high-speed mixer. After mixing, materials were preformed by compressing in a mold at ambient temperature into compact form. The pressure for preform is 40 MPa. The preform was then removed from the mold and sintered. Samples of the composites were produced by free sintering in air at 380°C for 1 h and using 40°C/h heating and cooling rates to ambient temperature. Finally, the samples were machined to produce specimens for tests.

### Test Details

The tribological behaviors of composites in sliding against a stainless steel ring were evaluated on an M-2000 (Zhangjiakou Xuanhua Kehua Mechanical Machine Co., Ltd.) friction and wear tester. The contact schematic diagram of frictional pairs is shown in Figure 1. A stainless steel ring (304, with roughness,  $R_a = 0.2 \mu\text{m}$ ) of diameter 40 mm was used as the counterpart. The blocks and the rings were cleaned ultrasonically in acetone and allowed to dry for 1 h in laboratory air. Sliding was performed under ambient conditions: temperature:  $23 \pm 2^\circ\text{C}$ , humidity:  $50 \pm 10\%$  at a sliding velocity of 0.42 m/s. The applied load was 200 N. Each friction and wear test was performed for 60 min. To study more about wear resistance, experiments by varying sliding distance were performed. The friction coefficient  $\mu$  of the specimen were calculated values of

friction torque, the friction coefficient of each specimen was continuously recorded automatically throughout the test. At the end of each test, the size of wear scars was measured, then the wear volume loss of the composites specimen was calculated.

$$V = B \left[ R^2 \sin^{-1} \frac{S}{2R} - \frac{S}{2} \sqrt{\left( R^2 - \frac{S^2}{4} \right)} \right]$$

where  $B$  is the width of blocks (mm),  $S$  is the wear scar width (mm), and  $R$  is the radius of the steel ring (mm).

The wear rate,  $W$ , of each specimen was calculated from the relationship:  $W = V/(S_d P)$ , where  $V$  is the wear volume (mm<sup>3</sup>),  $S_d$  is the sliding distance (m), and  $P$  is the applied load (N). The average of the wear rates is cited in the Results section.

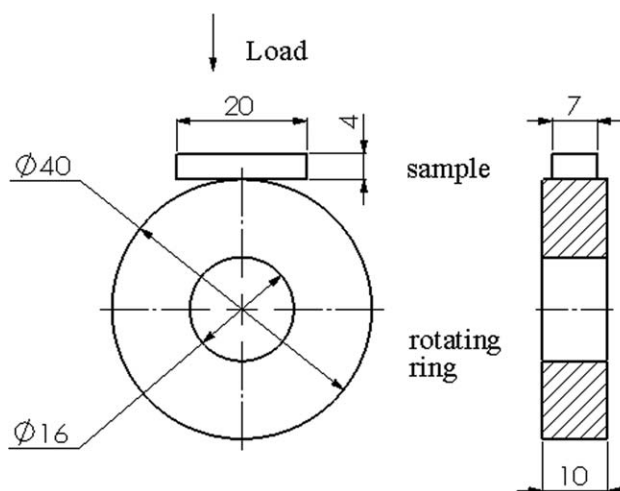
A differential scanning calorimetry (DSC) study was conducted on pure PTFE and the composites. DSC studied on the PTFE and PTFE composites was performed using a NETZSCH DSC 200F3 coupled with a computer station. The DSC test was conducted to study possible reactions taking place between the fillers and the matrix and to investigate the crystallinity of the composites. All experiments were conducted in nitrogen atmosphere and at a heating rate of 10°C/min up to 400°C.

$$X_c(\%) = \frac{\Delta H_m}{\Delta H_o} \times \left( \frac{100}{x} \right)$$

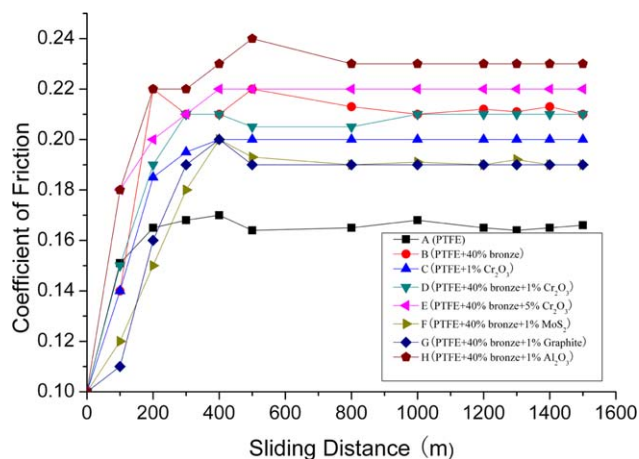
where  $X_c$  is crystallinity,  $\Delta H_m$  is the heat melting of composites,  $\Delta H_o$  is the heat melting of 100% crystalline PTFE, and  $x$  is content of PTFE matrix in the composites.

The morphologies of the composite worn surfaces were examined with scanning electron microscopy (SEM, JSM-6330F JEOL). Samples were coated with thin layer gold to increase the electrical conductivity before testing.

X-ray photoelectron spectroscopy (XPS, ESCALAB 250, Thermo Fisher Scientific) was used to detect element chemical states. Samples intended for XPS should be handled carefully because minute contamination can mask the surface structure of the samples. XPS was used to detect element chemical states of the worn and unworn surfaces; this was intended to find potential



**Figure 1.** Contact schematic diagram for the frictional couple (unit: mm).



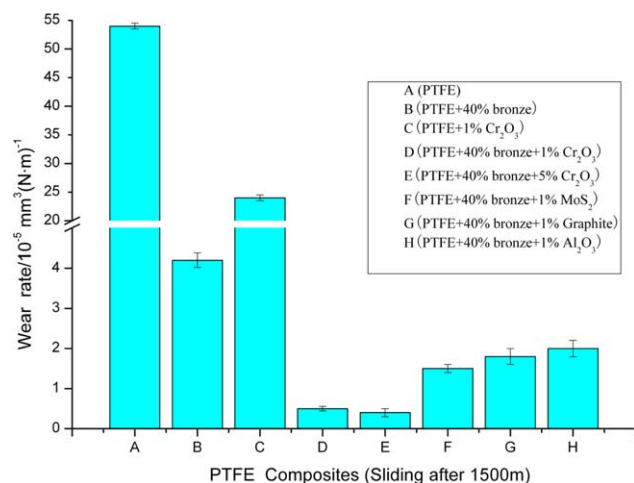
**Figure 2.** The friction coefficient of PTFE and its composites. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

tribochemical reaction, such as defluorination, conjugation of the carbon backbone, chain scission, or the formation of new side chains and end groups.

## RESULTS AND DISCUSSION

### Tribological Properties

**The Friction Coefficient.** Figure 2 shows the friction coefficients of each specimen obtained from the M-2000 wear tester. Results showed that the friction coefficients varied with sliding distance. All friction coefficients increased at the initial stage and then reached a stable stage. For pure PTFE, the friction coefficient was only 0.16. On adding fillers to PTFE, the friction coefficient was increased irrespective of the kinds and amount of filler. Fillers also effected the reach to the static stage of friction coefficients. The static friction coefficient of bronze-filled PTFE composite was 0.22; while adding  $\text{Cr}_2\text{O}_3$ ,  $\text{MoS}_2$ , and graphite to bronze-based PTFE composites, the friction coefficients were lowered. Results also showed that 1%  $\text{Cr}_2\text{O}_3$  filled in the 40% bronze-based PTFE composite helped to reach static



**Figure 3.** Wear rate of PTFE and its composites. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

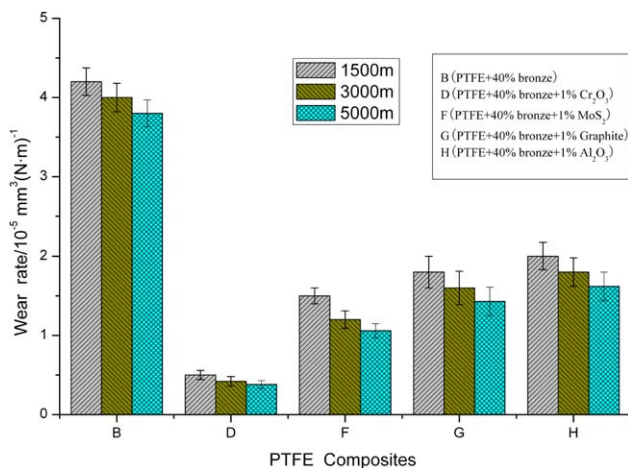
stage quickly and lower the friction coefficient.  $\text{Cr}_2\text{O}_3$ ,  $\text{MoS}_2$ , and graphite were solid lubricants that helped to form transfer films on sliding counterparts.

**The Wear Rate.** Wear rates of PTFE and its composites are shown in Figure 3. The wear rate of pure PTFE was  $54 \times 10^{-5} \text{ mm}^{-3}/\text{N m}$ . Adding fillers to PTFE could improve the wear resistance, even though less, for instance, 1%  $\text{Cr}_2\text{O}_3$  filled in to PTFE altered the wear rate of the composite by 50% as that of the pure PTFE. Bronze (40–60%) -filled PTFE composites were always used as reciprocating standard materials. However, the wear rate was not so effective as required;  $\text{MoS}_2$  or graphite was always added to composites to lower friction coefficient and enhance wear resistance for longer life of seals. It is interesting to find that for 1%  $\text{Cr}_2\text{O}_3$  filled to 40% bronze-based PTFE composite, the wear rate was  $0.5 \times 10^{-5} \text{ mm}^{-3}/\text{N m}$  and for composite B, it was  $4.2 \times 10^{-5} \text{ mm}^{-3}/\text{N m}$ . It enhanced approximately 10 times of the wear resistance of the bronze-based PTFE material.  $\text{MoS}_2$ ,  $\text{n-Al}_2\text{O}_3$ , and graphite also had some effects on the enhancement of wear resistance, but on a lower percentage. Therefore, it could be inferred that the addition of  $\text{Cr}_2\text{O}_3$  to PTFE was much more effective than  $\text{MoS}_2$ ,  $\text{n-Al}_2\text{O}_3$ , and graphite.

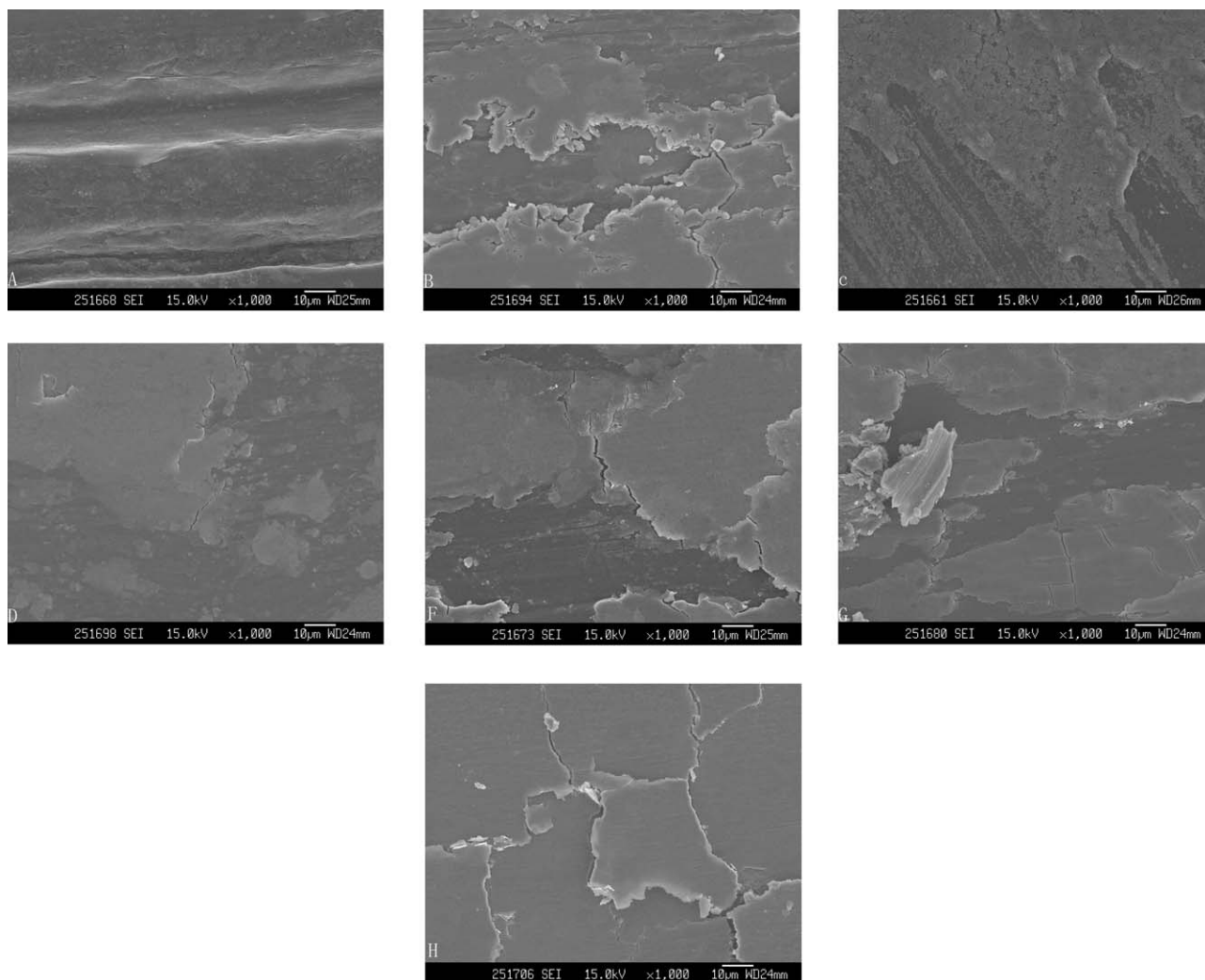
Results also showed that the addition of  $\text{Cr}_2\text{O}_3$  alone did not impart a good wear resistance to PTFE during sliding. This was because the particulates were ineffective in carrying the load.

When the filled  $\text{Cr}_2\text{O}_3$  content increased from 1% to 5% in the 40% bronze-based PTFE composite, the wear rate was lowered, but only less. Therefore, it could be inferred that increasing the percentage of  $\text{Cr}_2\text{O}_3$  was not more effective.

For the composites that exhibited the highest wear resistance, we tested different and long-duration sliding distances to repeat the wear resistance. Figure 4 shows the wear rate of PTFE and its composites after different sliding distances. Results showed that the wear rates of composites were lowered when sliding distance was increased. This was because in the first run-in period, the composites worn to the metal surface; after the run-in period, the transfer film was formed, then the composite was



**Figure 4.** Wear rate of PTFE and its composites after different sliding distances. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 5.** SEM images of the worn surfaces (A) PTFE, (B) 60% PTFE + 40% bronze, (C) 99% PTFE + 1%  $\text{Cr}_2\text{O}_3$ , (D) 59% PTFE + 40% bronze + 1%  $\text{Cr}_2\text{O}_3$ , (F) 59% PTFE + 40% bronze + 1%  $\text{MoS}_2$ , (G) 59% PTFE + 40% bronze + 1% graphite, (H) 59% PTFE + 40% bronze + 1%  $\text{n-Al}_2\text{O}_3$ .

not in direct contact with metal surface but with transfer-film of composites.

In the long sliding distance, results also showed that  $\text{Cr}_2\text{O}_3$  had better effect on the enhancement of wear resistance than  $\text{MoS}_2$ ,  $\text{n-Al}_2\text{O}_3$ , and graphite when filled to the bronze-based PTFE composites.

#### SEM of the Worn Surfaces

SEM micrographs of worn surfaces of PTFE and its composites after 60 min of wear test at 200 N and 0.42 m/s are shown in Figure 5. Results showed that different PTFE composites undergo different wear mechanisms, which were governed by the different actions of various fillers after sliding against steel counterparts.

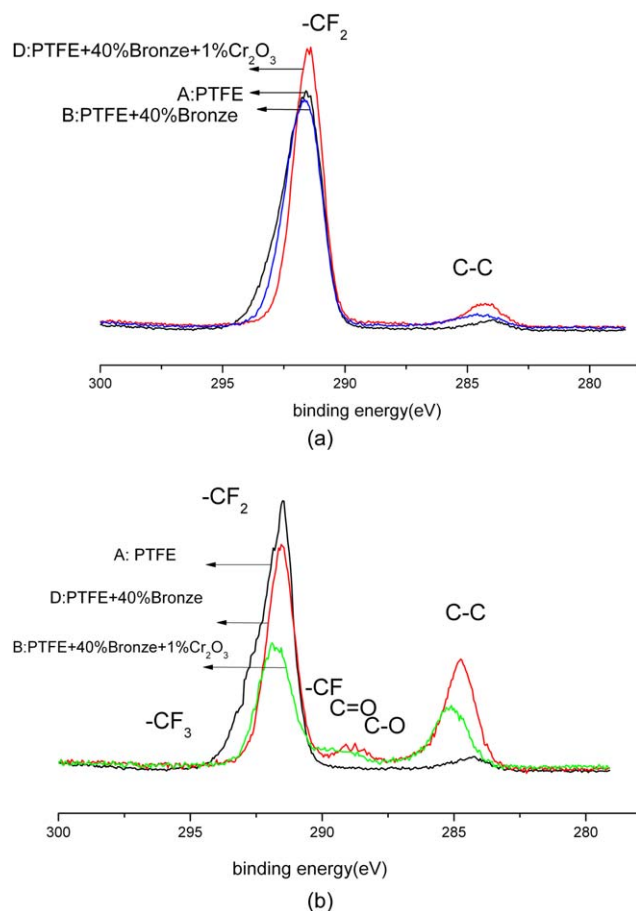
For pure PTFE [Figure 5(A)], it could be seen that plastic deformation and plough predominantly constitute the worn surface; there were also some debris on the worn surface. These contained all fatigue wear, adhesive wear, and abrasive wear mechanisms. There were no plastic deformation and ploughed marks on the worn surfaces of PTFE composites. This meant that the adhesive wear was relieved. There were more debris on

the worn surfaces of PTFE composites, which means that abrasive wear was the dominant wear mechanism.

Figure 5(D) shows that the wear debris were much smaller than others and this helped to form a uniform transfer film on the worn surface and counterpart steel surface easily, which finally led to a significant improvement of the wear resistance. The surfaces in Figure 5(F–H) were much smoother than Figure 5(A), as  $\text{MoS}_2$ , graphite, and  $\text{n-Al}_2\text{O}_3$  reduced the adhesion between the materials and counterparts. In addition, the interface between the debris and the substrate of the PTFE composites were ambiguous while PTFE is obvious, indicating that the transfer film was formed on the surface. Those resulted in the low wear resistance of the composites.

#### XPS Analysis of the Worn Surface

Figure 6(A) shows that for the unworn surfaces of PTFE and composites (B and D), there were two peaks, one at 292 eV for  $-\text{CF}_2-$  and the other at 284.8 eV for  $\text{C}-\text{C}$ . This implies that there was no reaction between fillers and PTFE. XPS spectrum [Figure 6(B)] of the pure PTFE worn surface showed that there



**Figure 6.** XPS spectra of C1s PTFE and composites surface: (A) unworn and (B) worn. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

was no new peak of C1s appeared which meant no tribochemical reaction occurred. There were new peaks for worn surfaces both B and D which meant that there were tribochemical reactions during sliding. The presence of new peaks around 290 eV was observed in the carbon spectra for worn surface of PTFE composites. For composite D, three new peaks<sup>14–16</sup> for  $-\text{CF}-$ ,  $\text{C}-\text{O}$ , and  $\text{C}=\text{O}$  were observed, which indicates reactions such as defluorination, conjugation of the carbon backbone, chain scission, or the formation of new side chains and end groups. As filling  $\text{Cr}_2\text{O}_3$  in bronze-based PTFE may advance the tribochemical reactions, advancement of the molecular chain of PTFE was broken. The species observed at 290 eV was observed in a much smaller quantity in composite B and was not observed on the wear surface of unfilled PTFE.

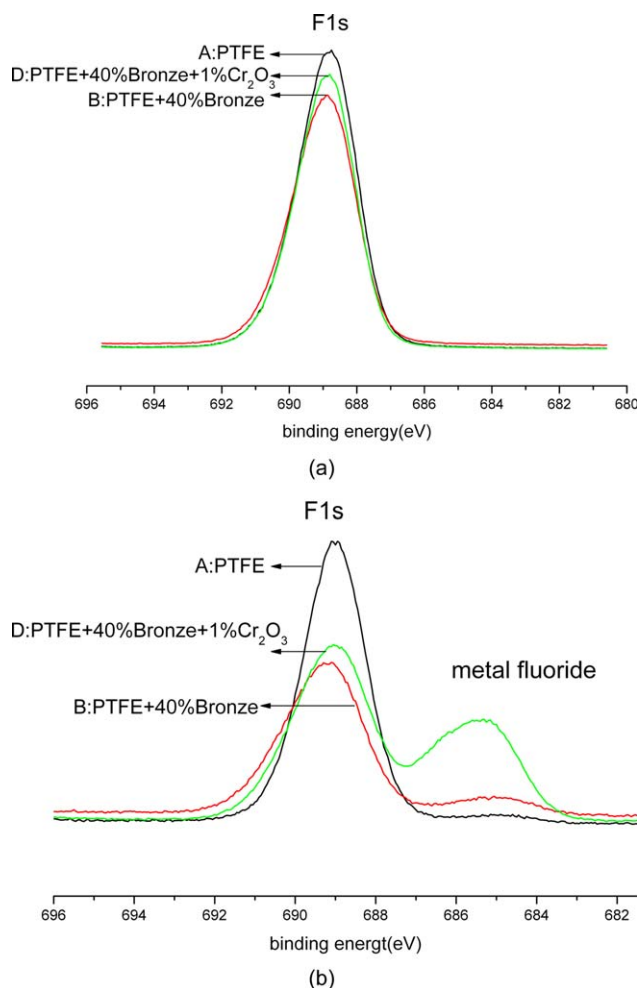
In the F1s XPS spectrum of PTFE composites in Fig. 7, results showed that for the unworn surfaces in Fig. 7. a, there was only one peak for  $-\text{CF}_2-$ , which implies no reaction between fillers and PTFE. There was a new peak of metal fluoride (685 eV) appeared for worn surfaces, especially for composites D, the ratio of metal fluoride to  $-\text{CF}_2-$  was higher than that of B composite. We may infer that there were tribochemical reactions during sliding for composites, and  $\text{Cr}_2\text{O}_3$  may advance the tribochemical reactions. This peak suggested a tribochemical degradation of the polymer and generation of a new species at the interface, which

may be responsible for the improvement of wear resistance of PTFE composites. In Fig. 7.b compare the PTFE composites B to D, XPS results showed only composite D with  $\text{Cr}_2\text{O}_3$  formed metal fluoride, which should be  $\text{Cr}_2\text{O}_3$  acted as a catalyst in situ formation of metal fluoride in the transfer film formation process. The possible tribochemical mechanism was shown as in Fig. 8. Therefore, it could be inferred that a metal fluoride was easily formed when the  $\text{Cr}_2\text{O}_3$  filled to bronze-based composite, and it worked as a lubricating film to improve tribological performance.

#### Transfer Film of Counterparts

On dry sliding, transfer film of PTFE can form on the counter surface. Transfer film was important for wear on dry condition. During sliding, when transfer film formed, it effectively improved the tribological condition, PTFE ran against transfer film of itself other than metal surface, then the friction coefficient would be lower and wear rate would be less.

Figure 8 shows the transfer films of PTFE and different PTFE composites. It is interesting to notice that the transfer film of  $\text{Cr}_2\text{O}_3$ -filled bronze-based PTFE composite (D) seemed to be more compact, thick, and smooth than that of  $\text{Cr}_2\text{O}_3$ -unfilled bronze-based PTFE composite (B). This was mainly because



**Figure 7.** XPS spectra of F1s PTFE and composites surface: (A) unworn and (B) worn. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



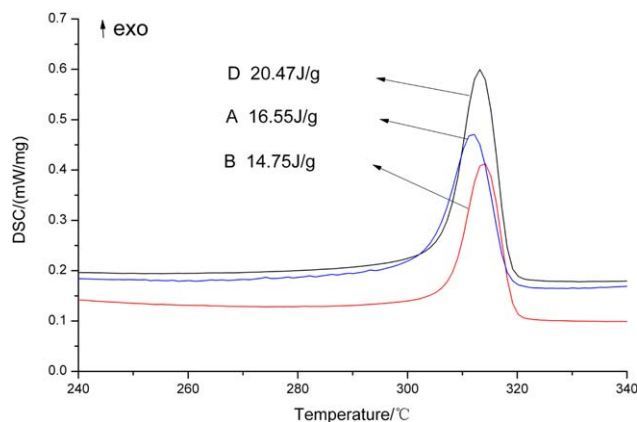
**Figure 8.** Transfer film on the steel ring. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

$\text{Cr}_2\text{O}_3$  helped to form transfer film more easily during the course of sliding. Results showed that excellent performance in the surface was caused by in situ formation of metal fluoride in the transfer film formation.

This thick, compact, smooth, and unique transfer film helped to reduce the friction coefficient as the PTFE composite ran against transfer film of PTFE composite and hence the wear rate was small. Therefore, it could be inferred that  $\text{Cr}_2\text{O}_3$  was transferred on the surface, the addition of  $\text{Cr}_2\text{O}_3$  to bronze-based composite was essential for low friction and wear rate.

#### DSC Analysis

Figure 9 and Table II show the DSC analysis of PTFE and PTFE composites. DSC studied the pure PTFE, bronze-filled PTFE composite, bronze- and  $\text{Cr}_2\text{O}_3$ -filled PTFE composite. From the



**Figure 9.** DSC curves of PTFE and PTFE composites. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

**Table II.** DSC Analysis

Samples	$T_c$ ( $^{\circ}\text{C}$ )	$\Delta H_m$ (J/g)	$X_c$ (%)
PTFE	311.5	16.55	38
PTFE + 40% bronze	313.6	14.75	45
PTFE + 40% bronze + 1% $\text{Cr}_2\text{O}_3$	313.2	20.47	52

DSC curve, it can be inferred that there was no reaction between the filler materials and the PTFE matrix, as there was no other peak appeared and no major variation in the position of the crystal peak. Results showed that bronze filled in PTFE would increase the degree of crystallinity. The crystallinity<sup>17</sup> of pure PTFE was 38%, while the crystallinity of 40% bronze-based PTFE composite was 45%.  $\text{Cr}_2\text{O}_3$  is a kind of inorganic material that can play the role of nucleating agent for PTFE, inducing the increase of crystallinity. Results showed that bronze- and  $\text{Cr}_2\text{O}_3$ -filled PTFE composite had a higher crystallinity than pure PTFE and bronze-filled PTFE composite. The higher values exhibited by composite more than likely depend on factors such as increased bonding strength between the matrix and filler materials and higher degree of crystallinity induced by the filler materials.<sup>18</sup> This resulted in higher stability at a given temperature as developed during sliding at interface thereby reducing adhesive wear. Sviridyonok et al.<sup>19</sup> and Ueda et al.<sup>20</sup> had found the relationship between crystallinity and wear rate, when the crystallinity is between 25% and 64%, as crystallinity increases, the wear rate decreases. According to these, 1%  $\text{Cr}_2\text{O}_3$  increased the crystallinity and had a positive effect on wear resistance.

#### CONCLUSION

Addition of small amount (1 wt %) of  $\text{Cr}_2\text{O}_3$  improved wear resistance of bronze-based PTFE composite significantly while it affected the friction coefficient slightly.  $\text{Cr}_2\text{O}_3$  was better than traditional lubricants such as  $\text{MoS}_2$  and graphite for improving wear resistance of bronze-based PTFE composites. Adding  $\text{Cr}_2\text{O}_3$  to bronze-based PTFE composite promoted to form a thick and unique transfer film on the counterpart and it worked as a lubricating film to improve wear resistance. XPS results showed that a metal fluoride was formed when  $\text{Cr}_2\text{O}_3$  was filled into bronze-based PTFE composite after wear testing.  $\text{Cr}_2\text{O}_3$  affected tribochemical reactions during sliding against ring; these may be responsible for the formation of transfer film and lowered wear rate. DSC results suggested that  $\text{Cr}_2\text{O}_3$  increased the crystallinity of bronze-based PTFE composites from 45% to 52%, which was very effective in improving the wear properties of the composites.

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