

Fabrication of Mechanically Robust Self-lubricating Polyphenylene Sulfide Composites for High PV (Pressure × Velocity) Applications

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Electronic Supplementary Information

Abstract To address the poor mechanical performance and improve the tribological properties of self-lubricating polyphenylene sulfide/irradiation treated polytetrafluoroethylene (PPS/i-PTFE) blends, different aspect ratio carbon fibers (i.e., PSCF: 50, SCF: about 429) were introduced as reinforcement fillers. The results showed that the hybridizing of PSCF and SCF at certain mass ratios exhibited simultaneous enhancement of mechanical and tribological performance for PPS/i-PTFE blend through the construction of synergistic lubrication and mechanical interlocking network. Specifically, the flexural strength and modulus of PPS/i-PTFE were increased by 125.6% and 389.3%, the friction coefficient and specific wear rate were decreased by 13.9% and 95%, respectively. It was worth noting that PPS composites possessed excellent integrated performance which were able to withstand sliding action under high PV (≥ 10 MPa·m/s) conditions, as assessed by a customized pin-on-disc tester. This work demonstrated that the formation of intact lubricating film combined with the enhanced thermal and mechanical properties were favorable for improving the tribological properties of PPS-based composites, which makes them suitable for advanced engineering applications.

Keywords Polyphenylene sulfide; Irradiated polytetrafluoroethylene; Synergistic reinforcement; Tribological property

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INTRODUCTION

Tribological behavior is frequently encountered in industrial sectors and daily lives. Very high friction and wear can cause severe energy loss and materials loss that may result in malfunction and shortening service life of machinery components.^[1–5] The development of high-performance self-lubricating polymer composites that suit for industrial applications is receiving attention thanks to their lightweightness, ease of processing as well as tailorable tribological performance by adding different types of solid lubricants.^[6–8] Polyphenylene sulfide (PPS) is regarded as the sixth special engineering thermoplastic due to its outstanding thermomechanical performance, dimensional stability and chemical resistance. Thus, PPS is widely used to prepare the structural components in the areas of aviation, transportation, electronic devices among others.^[9,10] However, the friction and wear performance of pure PPS is quite poor which cannot be directly used for lubrication applications.^[11–13] It thus becomes imperative to enhance the tribological performance of PPS to broaden its application in the industrial sectors.

Recently, research efforts including blending with the other polymers,^[14] loading different solid lubricants such as poly-

tetrafluoroethylene (PTFE),^[15,16] graphite,^[17,18] molybdenum disulfide,^[19,20] inorganic particles such as titanium dioxide,^[21,22] silicon carbide,^[23,24] carbon fibers and fabrics^[25,26] were adopted to improve the tribological performance of PPS. Among the above-mentioned methods, incorporating PTFE was commonly employed because of its intrinsically low friction coefficient, ease of forming a lubricating film on the surface of friction pair, high-temperature resistance and excellent chemical stability.^[16,27–29] In a previous study, it was reported that utilizing irradiated-PTFE (i.e., i-PTFE) nanopowders was effective in improving the tribological performance of PPS due to its better compatibility with the host substrate which favored the development of intact and continuous F-rich transfer film on friction pair.^[30] Nevertheless, the introduction of PTFE particles undermined the mechanical performance. In a recent work,^[15] we reported that the addition of PTFE fillers led to a concurrent reduction of both tensile and flexural strengths of PPS. In addition, Zhang *et al.*^[31] reported that the mechanical performance of PTFE-filled polyoxymethylene deteriorated with increasing PTFE concentrations. Similar findings were reported by Chen *et al.*^[32] Therefore, reinforcement fillers^[33] such as carbon fibers (CF),^[34] glass fibers (GF)^[35] and natural fibers^[36] are adopted to enhance mechanical and tribological performance of polymer composites.

Conventionally, the tribological and mechanical properties of CF-containing composites are superior to those of GF-con-

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taining counterparts thanks to the self-lubricating nature and high specific modulus of CFs.^[37,38] Cho and Bahadur^[39] studied the influence of copper oxide (CuO) nanoparticles and CFs on the tribological behavior of PPS-based composites. They reported that PPS/CF/CuO composites presented superior tribological characteristics in comparison with single filler-containing counterparts, which indicated a synergistic role arising from the incorporation of nanoparticles and reinforced fibers in tailoring the tribological performance. According to previous studies,^[38,40] the addition of CFs was favorable for concurrently meliorating the mechanical and tribological performance of PPS-based composites; however, the effect of using CFs with different aspect ratios and the hybridization of different aspect ratio CFs on the properties of PPS-based composites were not comprehensively studied.

In this present work, CFs with different aspect ratios (*i.e.*, PSCFs: 50, SCFs: about 429) were employed as the reinforcement fillers to meliorate the mechanical and tribological performance of PPS/i-PTFE blend. The findings demonstrated that the hybridization of PSCF and SCF at certain mass ratios led to a simultaneous enhancement of mechanical and tribological properties. The effect of using multi-scale CFs on improving tribological performance and the related mechanism were reported. Additionally, the tribological behavior of PPS-based composites which was evaluated under rigorous conditions ($PV \geq 10$ MPa·m/s) was performed using a customized pin-on-disc tester. To conclude, a feasible method was provided to manufacture plastic parts with excellent mechanical and tribological characteristics that exhibit prospective industrial utilization in the aerospace, automotive and machinery sectors.

EXPERIMENTAL

Materials Details

PPS powders were obtained from Deyang Keji High-Tech Material Co., Ltd., (China). Irradiation-treated PTFE (i-PTFE) were purchased from Sichuan Golden Nuclear Irradiation Technology Co., Ltd., (China). Polyacrylonitrile-based short carbon fiber powders (PSCF, diameter: 6 μm , length: 0.3 mm, aspect ratio: 50) and short carbon fiber (SCF, diameter: 7 μm , length: 3 mm aspect ratio: about 429) were available from Shanghai Lishuo Composite Material Technology Co., Ltd., (China). Additionally, the detailed information of PPS, i-PTFE and CFs was provided in the supplementary file.

Fabrication of Samples

All materials were dried thoroughly prior to processing. Afterwards, they were mixed in accordance with the mass ratios specified in Table 1 and introduced into a twin-screw extruder (Chengdu Tarise Chemical Engineering Co., Ltd., China) for melt compounding. The processing temperatures were set at 230, 275, 285, 285, 290, 290, 285 and 285 $^{\circ}\text{C}$, respectively, and the screw rotation speed was 70 r/min. Then, an injection molding machine (Ningbo Haitian Plastics Machinery Co., Ltd., China) was used to fabricate specimens for testing. The processing temperatures were set at 290, 300, 300, 300 and 280 $^{\circ}\text{C}$, respectively. The injection pressure and speed were 120 MPa and 75 mm/s, respectively.

Table 1 The material designation and formulation of various composites.

Sample ID	Weight fraction (wt%)			
	PPS	i-PTFE	PSCF	SCF
P/i-PTFE	70	30	0	0
P/i-PTFE/PS20	56	24	20	0
P/i-PTFE/PS15-S5	56	24	15	5
P/i-PTFE/PS10-S10	56	24	10	10
P/i-PTFE/PS5-S15	56	24	5	15
P/i-PTFE/S20	56	24	0	20

Characterization

The tensile and flexural properties were assessed using a universal testing machine (Instron 5567, USA) as per GB/T 1040.2-2022 and GB/T 9341-2008, respectively. Five specimens were tested for each sample. A Shore durometer (Yueqing Handpi Instruments Co., Ltd., China) was employed to evaluate the surface hardness. The tribological properties were measured using a M-200A block-on-ring testing rig (Beijing Guance Instrument Co., Ltd., China). The applied load, sliding velocity and duration were 200 N, 0.42 m/s and 3600s, respectively. Three specimens were tested for the above measurements. The friction coefficient (μ) and specific wear rate (W) was calculated as per the following formula:^[23,30,38]

$$\mu = \frac{P}{F \times r} \quad (1)$$

$$v = d \times \left[\frac{\pi r^2}{180} \arcsin\left(\frac{w}{2r}\right) - \frac{w}{2} \sqrt{r^2 - \frac{w^2}{4}} \right] \quad (2)$$

$$W = \frac{v}{F \times L} \quad (3)$$

where, P is friction torque (N·m), F is the applied load (N), r is the radius of steel ring (m), v is the wear volume (mm^3), d is the width of the specimen (mm) and w is the length of worn tracks (mm), L is the sliding distance (m).

The *in situ* friction temperature during tribological test was recorded by a multi-path thermometer (Changzhou AnBai Precision Instrument Co., Ltd., China). A JSM-9600 scanning electron microscope (JEOL, Japan) was used to capture the cryo-fractured morphology and worn surface. The elemental composition and mapping analysis of transfer film was characterized using an energy-dispersive X-ray spectrometer (EDS) equipped with SEM. A 3D profilometer (VR6200, Keyence, Japan) was employed to capture the topography of worn tracks. The chemical state of specimens after friction tests was characterized by an X-ray photoelectron spectrometer (XPS, AXIS Supra, Kratos Instruments Co., Ltd., UK). The electrical resistance of each sample was examined by a SM711 resistance meter (HIOKI, Japan) and five replicates were measured. Then, the electrical conductivity (φ) was obtained in accordance with the following equation:

$$\varphi = \frac{1}{\delta} = \frac{H}{\varepsilon \times S} \quad (4)$$

where, δ is volume resistivity ($\Omega\cdot\text{m}$), ε is electrical resistance (Ω), H is the length of the specimen (m), S is sectional area (m^2).

The thermal conductivity was characterized using a Hot disk TPS-2500S thermal constant analyzer (Hot Disk AB, Sweden). The tribological performance of samples under high PV

(≥ 10 MPa·m/s) conditions was tested by a homemade pin-on-disc tester. The applied load and sliding velocity were 200 N and 2 m/s, respectively. The duration for each test was 210 s. Three specimens were tested for the above measurements. The calculation of μ and mass wear rate (MWR) was given as follows:

$$\mu = \frac{P}{F \times R} \quad (5)$$

$$m = m_a - m_b \quad (6)$$

$$\text{MWR} = \frac{m}{F \times L} \quad (7)$$

where, P is friction torque (N·m), R is the radius of testing plate (m), F is the load (N), m is wear loss (mg), m_a is the initial specimen's weight (mg), m_b is the specimen's weight after tribological test (mg), L is the testing distance (m).

The dimensions of samples for friction and wear tests were 30 mm $\times$ 6 mm $\times$ 7 mm, and the testing setups for both methods were depicted in Fig. 1.

RESULTS AND DISCUSSION

Morphology

Fig. 2(a) shows that areas where are distinct holes (as circled by the red contour) and i-PTFE agglomerates (as pointed out by the red arrow) were observed in P/i-PTFE blend, which were related to the small particle size of i-PTFE and the limited interfacial bonding with PPS substrate.^[41]

Figs. 2(b)–2(f) show that CFs were randomly interspersed and interwoven within PPS, presenting different degrees of “reinforced concrete structure”. In addition, traces of destruction and exfoliation of CFs were observed in the composites. However, less disruption and holes were observed in the cross-section of samples with increasing SCF content. When the mass ratio of PSCF:SCF is 1:3, Fig. 2(e) shows that P/i-PTFE/PS5-S15 exhibited the densest filler packing structure. This is likely attributed to the fact that lower AR PSCFs were uniformly distributed in PPS, which favored the formation of a loosely stacked filler network. In addition, the presence of a certain content of lower AR PSCFs effectively filled the voids among discretely distributed SCFs, thus reducing the fiber-deficient region and constructing a dense filler packing structure to improve the mechanical performance. Fig. 2(f) shows

that the higher aspect ratio (AR) SCFs were interspersed and interwoven with each other to form a compact filler network in P/i-PTFE/S20.

Mechanical Properties

Fig. 3 demonstrates that the mechanical properties of P/i-PTFE blend increased significantly with the addition of CFs. Particularly, as shown in Fig. 3(a), the flexural strength and modulus of the composites reached as high as 191.1 MPa and 13.7 GPa when the mass ratio of PSCF:SCF is 1:3, which were 125.6% and 389.3% higher than those of P/i-PTFE, respectively. Under such circumstances, the formation of a dense CFs packing network within PPS is regarded as the contributing factor.

Similar to flexural properties, the tensile strength of P/i-PTFE/CF composites improved consistently with increasing fraction of SCFs. This was due to the better load transfer of higher AR CFs^[38]. In addition, Fig. 3(b) illustrates that the surface hardness of PPS composites was enhanced by adding CFs, which was related to the formation of entangled CF network that led to an increase of surface hardness.^[42] The increase of the mechanical properties of P/i-PTFE/CF composites was favorable for enduring external friction damage, thereby improving tribological properties.

Tribological Properties

Fig. 4(a) shows that a great fluctuation of transient μ was observed for PPS-based composites with the addition of CFs. The above observation indicated that the fractured CFs would act as hard abrasives amid sliding interface which caused instability of transient μ during running-in period.^[43] The fluctuation in transient μ of P/i-PTFE/CFs composites was gradually decreased with increasing proportion of SCFs. This was related to the development of 3D reinforced filler packing structure arising from the presence of higher AR SCFs that reduced the likelihood of peeling off during friction process.

The average μ and W of P/i-PTFE with different content of CFs were presented in Fig. 4(b). The results showed that the values of both μ and W of P/i-PTFE blend were greatly reduced by adding CFs. By comparing the μ and W between P/i-PTFE/PS20 and P/i-PTFE/S20, the presence of PSCFs was effective in reducing μ and the presence of SCFs was able to greatly reduce W . Thus, the tribological properties of P/i-PTFE/CF composites were affected by the AR of CFs. Lower AR CFs

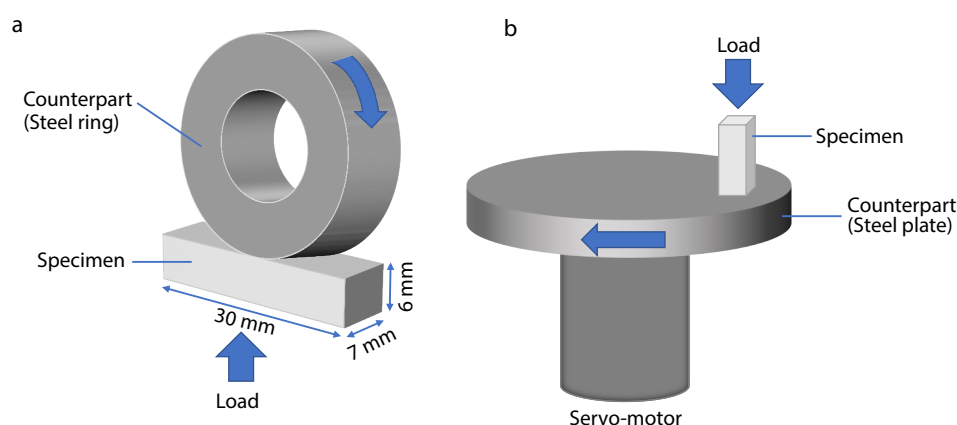


Fig. 1 The schematic illustration of (a) block-on-ring and (b) pin-on-disc tests.

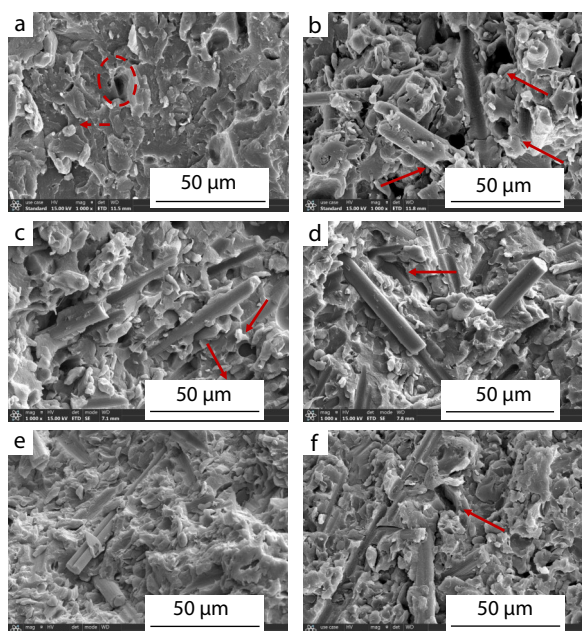


Fig. 2 The morphology of (a) P/i-PTFE, (b) P/i-PTFE/PS20, (c) P/i-PTFE/PS15-S5, (d) P/i-PTFE/PS10-S10, (e) P/i-PTFE/PS5-S15 and (f) P/i-PTFE/S20.

were more likely peeled off from the sample block and therefore participated in the formation of a continuous transfer film on the surface of friction pair, thereby reducing the direct contact area between the surface of sample block and friction pair, and contributing to reducing μ .^[44]

When a small amount (5 wt%) of PSCFs were replaced by higher AR SCFs, *i.e.*, P/i-PTFE/PS15-S5, both μ and W were increased when compared with P/i-PTFE/PS20, which was related to cutting effect of higher AR SCFs. Under such circumstances, the fragmentation of SCFs formed wear debris amid sliding interface, which would scrape the surface of specimens and resulted in an increase of both μ and W . However, the μ and W were decreased with increasing the proportion of SCFs. In this scenario, the development of intact reinforced

network structure within PPS was instrumental in improving mechanical properties and thermal conductivity that led to an improvement of tribological performance.

When the filling ratio of PSCFs and SCFs is 1:3, the μ and W of P/i-PTFE/PS5-S15 reached as low as 0.142 and $1.63 \times 10^{-6} \text{ mm}^3/(\text{N} \cdot \text{m})$, which were 13.9% and 95% lower when compared with P/i-PTFE blend. According to a previous study, the average μ and W of pure PPS were as high as 0.564 and $3.55 \times 10^{-3} \text{ mm}^3/\text{Nm}$, respectively.^[40] As a result, the μ and W of P/i-PTFE/PS5-S15 were 74.82% and 99.95% lower than those of pure PPS, respectively. The above results indicated that a hybrid loading of CFs with different ARs was instrumental in improving the tribological characteristics of PPS.

Wear Mechanism

Fig. 5(a) demonstrates that the worn surface of P/i-PTFE exhibited wear tracks, ploughed furrows and wear debris coupling with the existence of lubricating film, which suggested poor load-bearing ability of sample blocks.^[15] The formed lubricating film was related to the transfer of PTFE chains which was able to develop a smooth transfer film during friction process.

The worn surface of P/i-PTFE/CF with different mass ratios of PSCFs and SCFs was presented in Figs. 5(b)–5(f). Fig. 5(b) shows that obvious wear debris appeared on the surface of sample block which indicated that P/i-PTFE/PS20 was subject to adhesive abrasion because low AR PSCFs were likely scrapped off from the sample surface and formed wear debris by depositing at the sliding interface, thereby forming lubricating film between friction pair and specimens that resulted in a reduction of μ and W (see Fig. 4b).

Fig. 5(c) reveals that P/i-PTFE/PS15-S5 exhibited a coarser worn topography and more debris and grooves, suggesting that the filling of 5 wt% SCFs was not enough to develop interwound network and the discretely distributed SCFs were likely fragmented by the sliding friction pair. Consequently, the wear debris served as hard abrasives that severely scrapped the sample surface. In addition, the protruded remains of SCFs caused a ploughing action on sample surface, which resulted in the formation of rougher worn surface. Under such circumstances, P/i-PTFE/PS15-S5 exhibited the high-

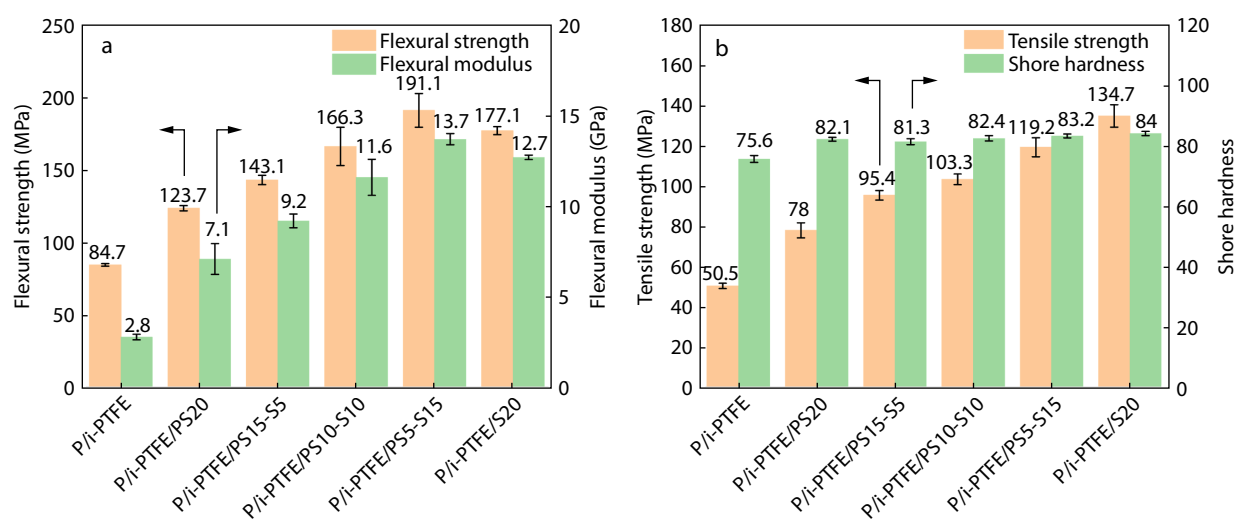


Fig. 3 The (a) flexural properties and (b) tensile strength and surface hardness of PPS-based composites.

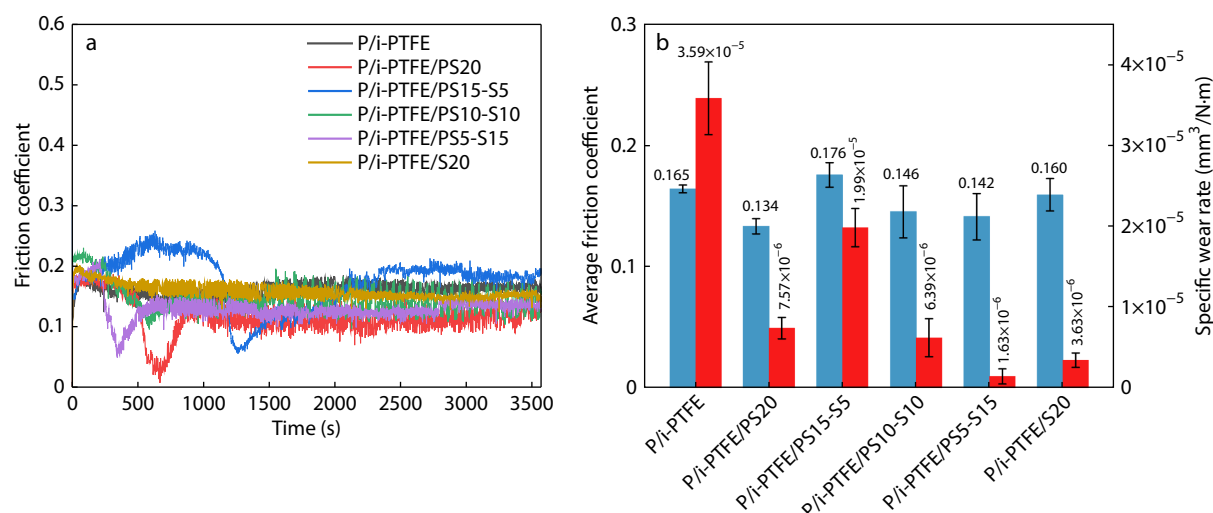


Fig. 4 (a) The transient μ of PPS-based composites as a function of sliding time, and (b) the average μ and W of different PPS-based composites.

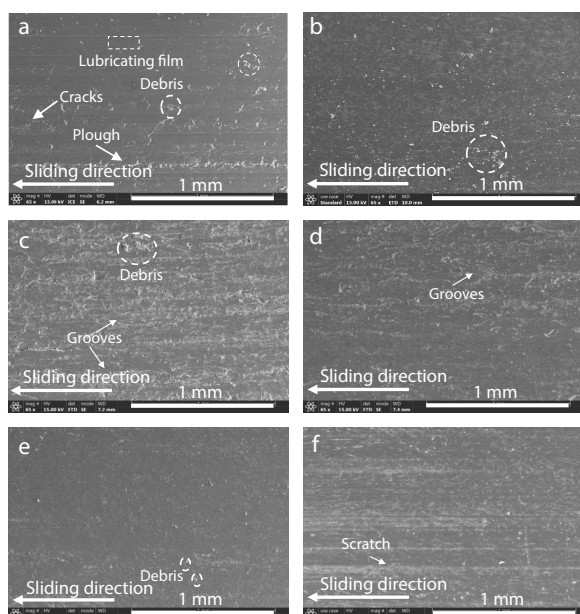


Fig. 5 SEM images of worn surface: (a) P/i-PTFE, (b) P/i-PTFE/PS20, (c) P/i-PTFE/PS15-S5, (d) P/i-PTFE/PS10-S10, (e) P/i-PTFE/PS5-S15 and (f) P/i-PTFE/S20.

est μ and W among the studied systems. Therefore, the wear mechanism of P/i-PTFE/PS15-S5 was mainly abrasive wear accompanied by slight adhesive wear.

Fig. 5(d) displays that P/i-PTFE/PS10-S10 had smoother worn surface when compared with that of P/i-PTFE/PS15-S5, suggesting that increasing the addition of SCFs was favorable for constructing reinforced network within PPS, thereby improving the tribological properties. The worn topography of P/i-PTFE/PS5-S15 became even better with only traces of wear debris, as detected in Fig. 5(e). The above observation showed that the wear mechanism of P/i-PTFE/PS5-S15 was mainly adhesive wear, and the formation of reinforced filler network within PPS in conjunction with the formation of continuous transfer film were believed to be the contributing fac-

tors. Although an intact reinforced CF structure was formed within P/i-PTFE/S20, a lack of lower AR PSCFs was unfavorable for the development of high-quality lubricating film. As a result, the tribological performance of P/i-PTFE/PS5-S15 was the best among the studied systems.

The profile of the worn tracks for specimens was observed using a 3D profilometer and the images were presented in Fig. 6. The characteristic parameters including worn width (R_w) and depth (R_v) of the worn tracks were displayed in Table 2. Fig. 6(a) shows that P/i-PTFE had a wide and deep worn track, which was attributed to the poor mechanical performance of sample block. According to literature,^[32,40] the introduction of i-PTFE reduced the surface hardness of sample block. Consequently, the polymer substrate was easily scrapped off by sliding friction ring. Fig. 6(b) demonstrates that, in comparison with P/i-PTFE, the worn track of P/i-PTFE/PS20 became shallower and narrower and this was linked with the formation of transfer film at the sliding interface that reduced both μ and W .

Similar to the observations in Fig. 5, P/i-PTFE/PS15-S5 possessed the deepest and widest worn track among the studied CF-containing composites. Fig. 6(e) and Table 2 show that P/i-PTFE/PS5-S15 had the lowest values in terms of both R_w and R_v . This was consistent with the tribological properties in Fig. 4(b) and SEM observations in Fig. 5. Thus, the results further corroborated the synergistic effect of adding CFs with different ARs in improving the tribological performance of PPS-based composites.

As shown in Figs. 7(A)–7(F), EDS elements analysis of transfer film on the friction pair for the PPS-based composites was conducted. Then, the optical images and element distribution on the surface of friction pair for the P/i-PTFE and P/i-PTFE/PS15-S5 after testing in Figs. 7(a, a1–a3)–7(e, e1–e3), respectively. Figs. 7(A) and 7(a2) demonstrate that a higher proportion of F element was present in the transfer film of P/i-PTFE, indicating that PTFE chains were transferred and covered to the surface of friction pair, which was favorable for reducing the μ and wear loss.^[32]

Figs. 7(B)–7(F) show that the proportion of F element re-

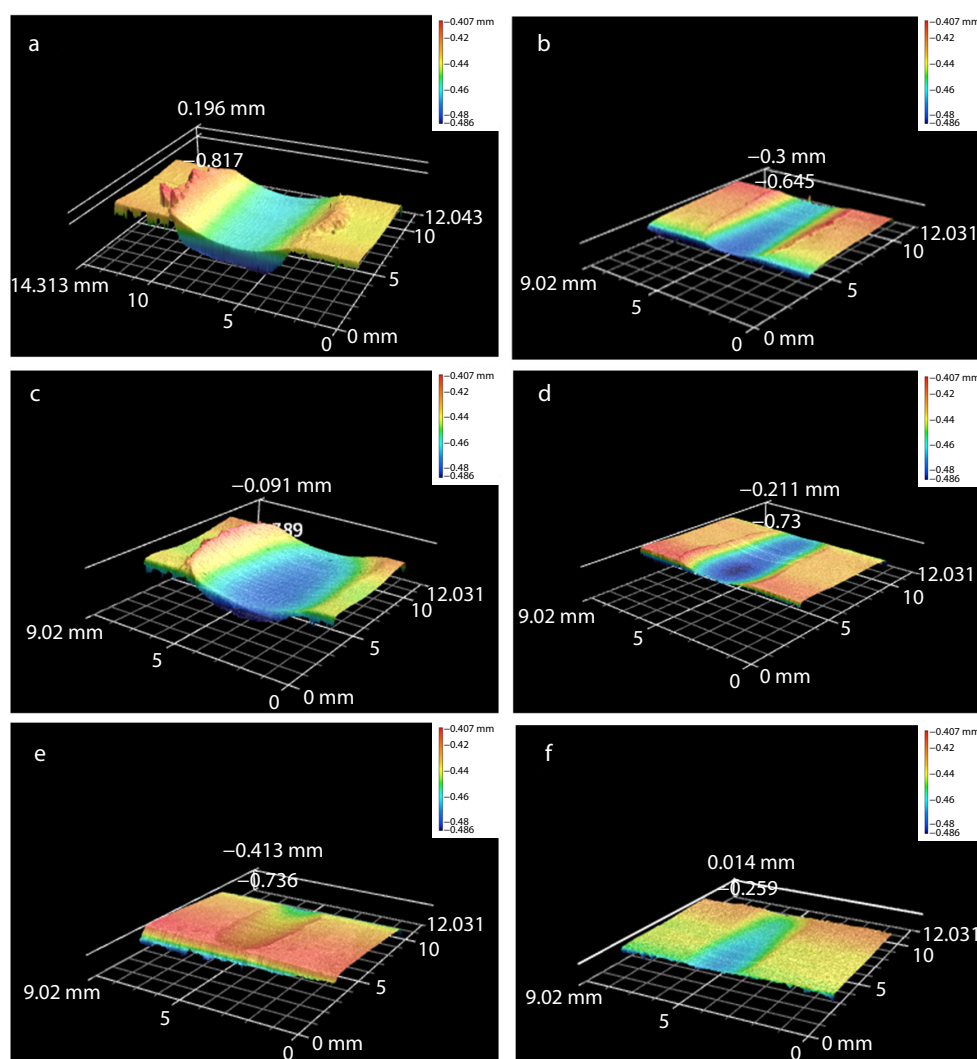


Fig. 6 3D profile of worn tracks: (a) P/i-PTFE, (b) P/i-PTFE/PS20, (c) P/i-PTFE/PS15-S5, (d) P/i-PTFE/PS10-S10, (e) P/i-PTFE/PS5-S15 and (f) P/i-PTFE/S20.

Table 2 The R_w and R_v of the worn track of specimens.

Sample ID	R_w (μm)	R_v (μm)
P/i-PTFE	6927	318
P/i-PTFE/PS20	4292	99
P/i-PTFE/PS15-S5	6630	412
P/i-PTFE/PS10-S10	3677	117
P/i-PTFE/PS5-S15	2652	29
P/i-PTFE/S20	2823	33

duced significantly in CF-containing composites when compared with that of P/i-PTFE counterpart, which was a result of the co-adhesive effect of CF debris and PTFE on the surface of friction pair. Moreover, the ratio of O/F element in the transfer film was the lowest for P/i-PTFE and it was increased greatly with the addition of CFs. For example, the ratio of O/F for P/i-PTFE was 0.439 whereas it was increased to about 1 for P/i-PTFE/PS20. This indicated that a severe friction-induced oxidation reaction was likely occurred for CF-containing composites due to the presence of CF debris and the intensive sliding action. Afterwards, the O/F ratio of P/i-PTFE/PS15-S5

was increased to 4.76 which suggested that the addition of a small amount of SCFs provided more CF debris or fragments at the sliding interface that greatly destroyed the transfer film, thereby deteriorating the tribological properties.

Furthermore, the ratio of O/F for P/i-PTFE/PS5-S15 was reduced to 3.01 and this indicated that friction-induced oxidation reaction was alleviated with the generation of continuous transfer film. The above finding was consistent with the results of μ and W as well as the morphology observations of the specimen. In addition, Fig. 7(F) shows that the transfer film of P/i-PTFE/S20 possessed a much lower level of F element when compared with the other CF-containing counterparts. This was probably attributed to the following reasons: (1) higher AR SCFs formed interconnected filler network within PPS, and the development of intact PTFE transfer film on the sliding interface was, to some extent, impaired; (2) higher AR SCFs were not feasibly transferred to the surface of friction pair and the fragmented CF debris acted as hard abrasives at sliding interface, which was unfavorable for the formation of intact and continuous transfer film.^[45] Consequently, the tribological performance of P/i-PTFE/S20 was inferior

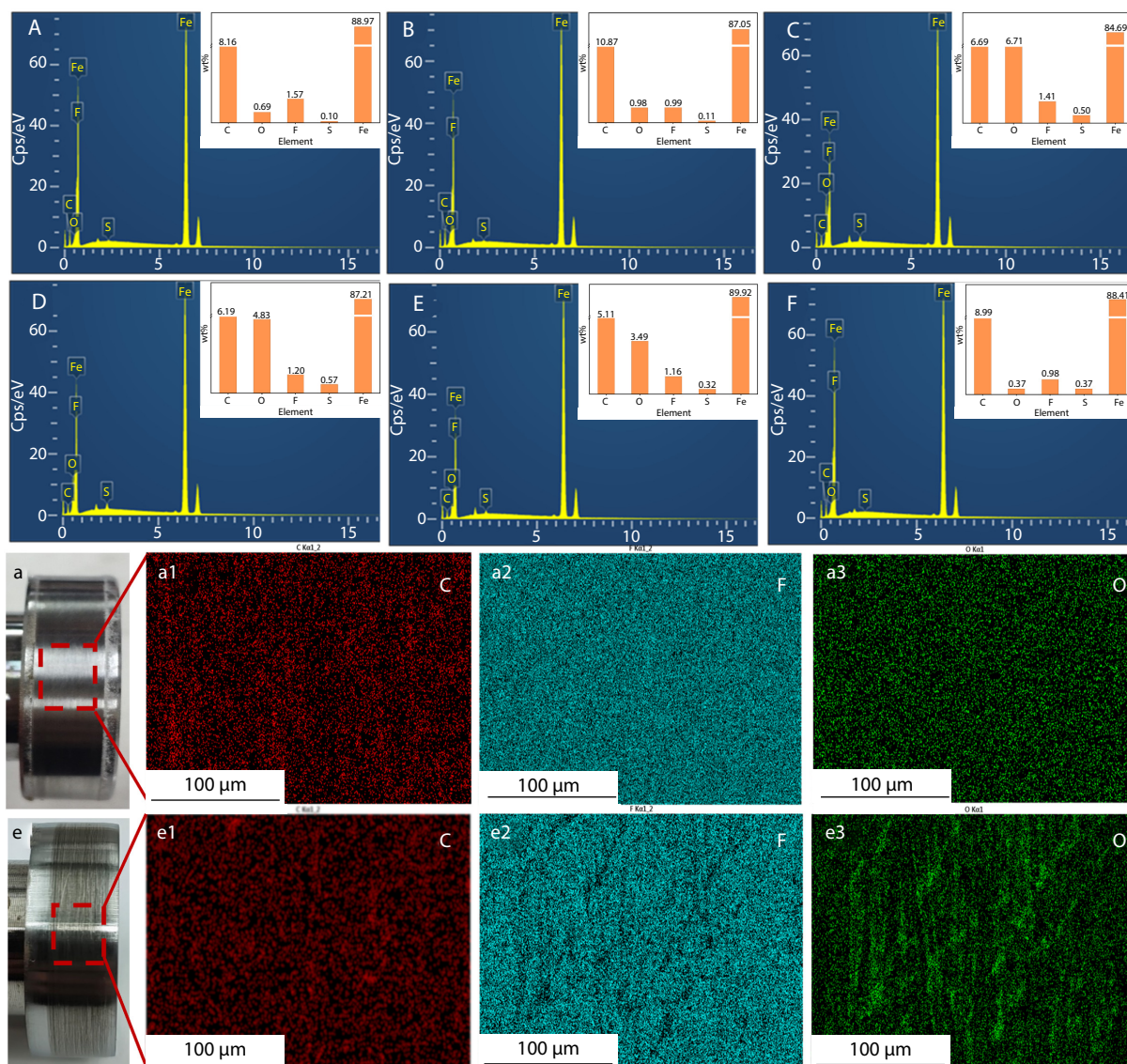


Fig. 7 EDS mapping analysis of transfer film on the friction pair: (A) P/i-PTFE, (B) P/i-PTFE/PS20, (C) P/i-PTFE/PS15-S5, (D) P/i-PTFE/PS10-S10, (E) P/i-PTFE/PS5-S15 and (F) P/i-PTFE/S20; Optical images and element distribution on the surface of friction pair: (a, a1–a3) P/i-PTFE, (e, e1–e3) P/i-PTFE/PS5-S15.

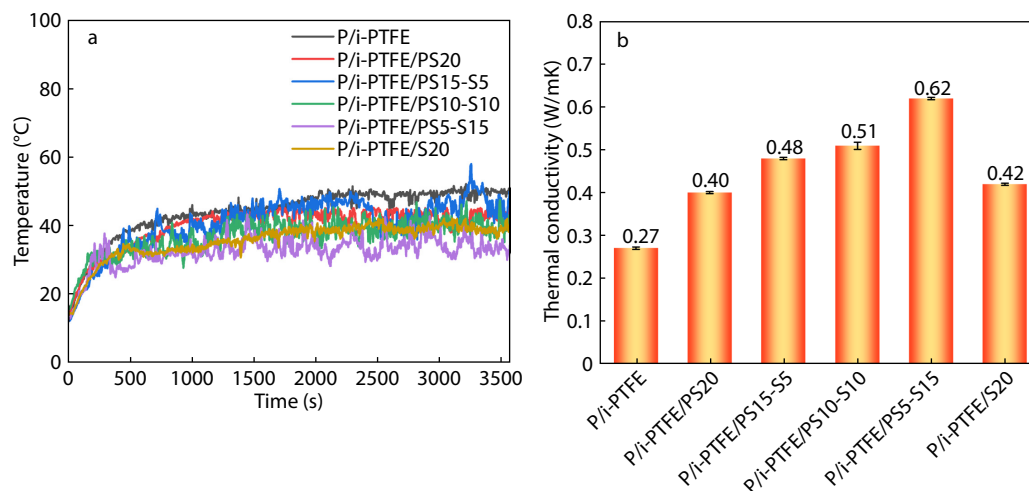


Fig. 8 (a) The instantaneous friction temperature during friction process and (b) the thermal conductivity of P/i-PTFE/CF composites.

to that of P/i-PTFE/PS5-S15.

Fig. 8(a) shows that there was a significant increase of friction temperature for all samples before 500 s, which was associated with the accumulation of frictional heat during running-in stage. In this scenario, frictional heat started to generate and accumulate at the sliding interface once the sample block came into contact with the friction pair, thereby resulting in an increase of instantaneous friction temperature.

Moreover, the friction temperature of sample blocks during the stable stage was quite different. For instance, P/i-PTFE demonstrated the highest instantaneous friction temperature among the investigated systems, and the friction temperature was reduced with the introduction of CFs in P/i-PTFE

blends, as displayed in Fig. 8(a). The above observation was related to the influence of thermal conductivity of sample blocks.^[46,47] Fig. 8(b) shows that the thermal conductivity of PPS-based composites was increased through the addition of CFs, which was related to the formation of interconnected filler network within the substrate.

According to literature,^[46,47] the higher the thermal conductivity of sample block, the easier dissipation of frictional heat. It is worth noticing that P/i-PTFE/PS5-S15 presented the lowest values of friction temperature among the studied system, which was attributed to the following factors: (1) P/i-PTFE/PS5-S15 showed a higher thermal conductivity when compared with the other composites, which was favorable for

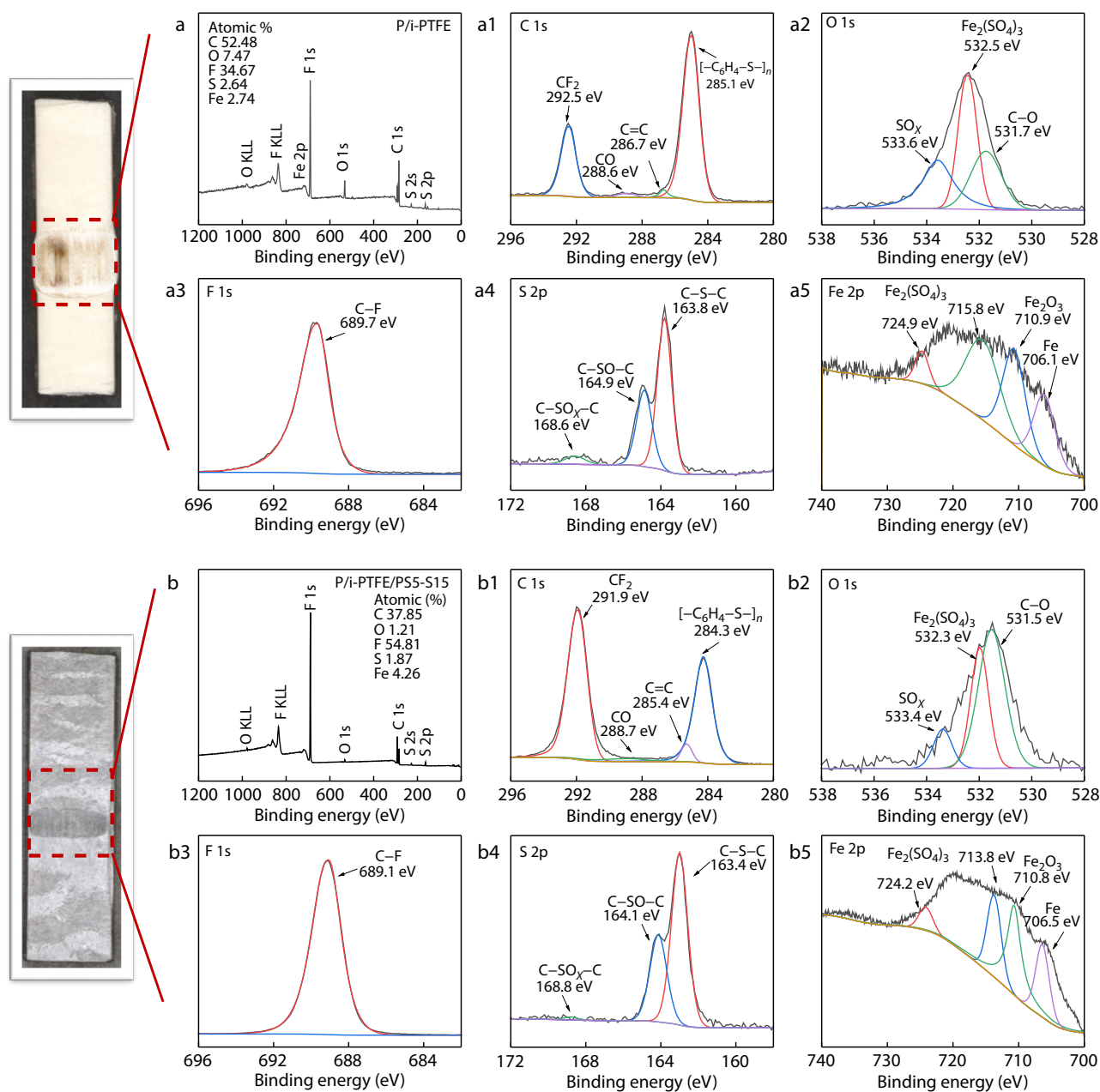


Fig. 9 (a, b) Full XPS spectra, (a1, b1) C 1s, (a2, b2) O 1s, (a3, b3) F 1s, (a4, b4) S 2p and (a5, b5) Fe 2p energy spectra correspond to the worn track of P/i-PTFE (top) and P/i-PTFE/PS5-S15 (bottom).

timely dissipating frictional heat during sliding process; (2) the generation of a well-developed transfer film reduced the direct contact between the sample block and metallic friction pair, thus reducing the friction work at sliding interface.

The bonding state and the proportion of corresponding peak fittings of P/i-PTFE and P/i-PTFE/PS5-S15 were presented in Fig. 9. The results suggested that different degrees of chemical reactions occurred on the surface of sample blocks during friction test. As displayed in Figs. 9(a) and 9(b), the relative content of O in P/i-PTFE/PS5-S15 was less than that of P/i-PTFE counterpart, while the F content was higher. In addition, a comparison of Figs. 9(a1) and 9(b1) reveals that the content ratio of $-\text{C}_6\text{H}_4-\text{S}-$ and CF_2 in the C1s spectrum of P/i-PTFE was much higher than that of P/i-PTFE/PS5-S15. It was worth noting that the relative content of $\text{C}-\text{SOx}-\text{C}$ in the S2p spectrum of P/i-PTFE/PS5-S15 was significantly lower. The results indicated that the oxidation reaction on the worn surface of P/i-PTFE/PS5-S15 was greatly mitigated than that of P/i-PTFE, coupling with less thermo-oxidative degradation of PPS chains.^[49,50]

The above finding is likely attributed to the fact that the formed dense CF network inside P/i-PTFE/PS5-S15 provided excellent resistance to external sliding force and efficient heat dissipation, which prevented the composite from being violent damaged during friction test. In addition, by comparing

the ratio of F content between P/i-PTFE and P/i-PTFE/PS5-S15, a more continuous and intact lubrication film existed on the surface of P/i-PTFE/PS5-S15. This was probably due to the softening of PPS substrate for P/i-PTFE blend arising from the accumulation of frictional heat, thereby leading to severe adhesion transfer of P/i-PTFE and resulting in higher μ and W .^[38] Under such circumstances, P/i-PTFE/PS5-S15 showed better tribological performance when compared with the other samples which were related to its excellent mechanical performance and the generation of intact lubrication film under the continuous action of friction pair.

Measuring the electrical conductivity (φ) can be used as an indicator to reflect the compactness of filler packing structure in CF reinforced polymer composites.^[51] Fig. 10(a) shows that the φ of P/i-PTFE composites exhibited a small enhancement with the addition of PSCF. However, with the introduction of higher AR SCF, the φ of corresponding composites showed a significant increase. This was due to the fact that higher AR CFs created more contact points that facilitated the transport of electrons.^[52] Notably, Fig. 10(a) shows that P/i-PTFE/PS5-S15 exhibited the highest φ among the studied systems, which validated the construction of internal dense CF network arising from the hybrid addition of CFs with different ARs.

The wear mechanism for representative samples was

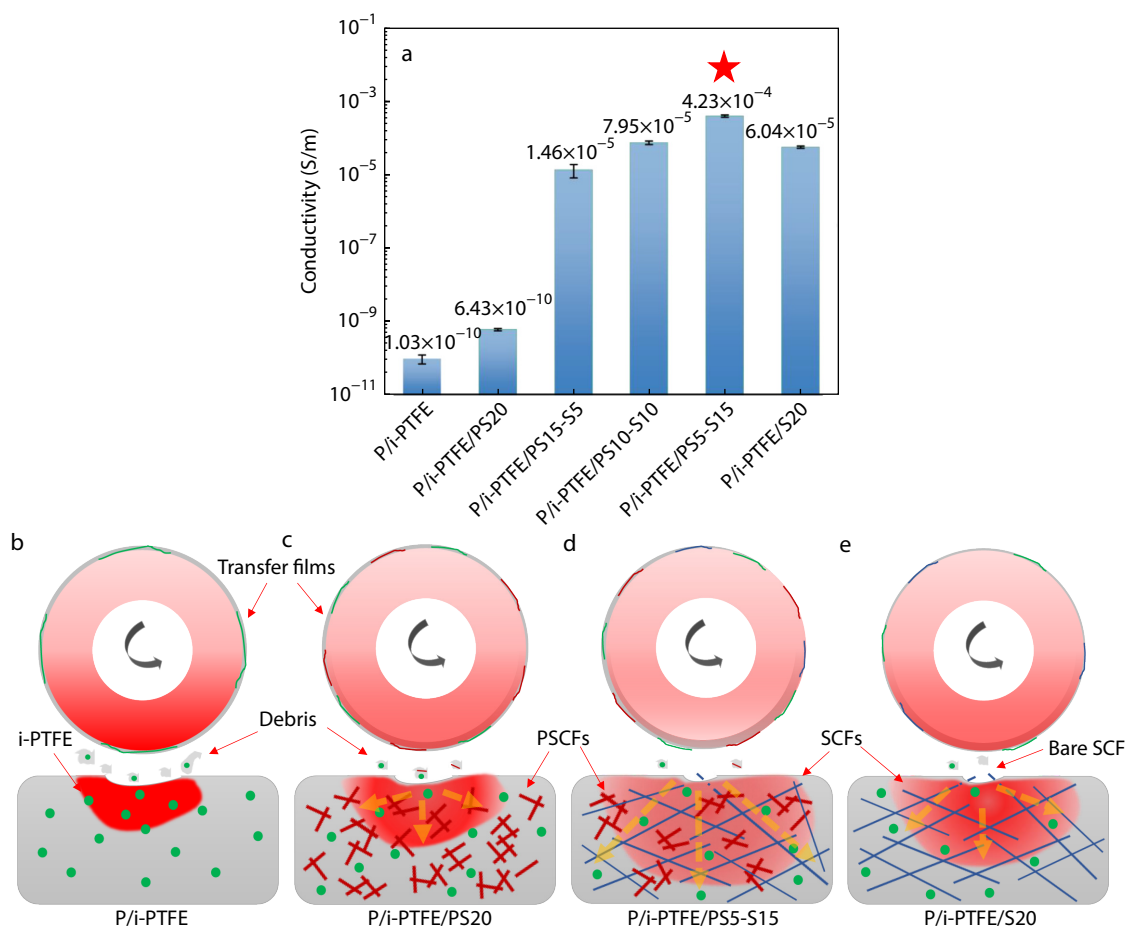


Fig. 10 (a) The electrical conductivity of PPS-based composites with different combinations of CFs; Schematic diagram of the friction process for (b) P/i-PTFE, (c) P/i-PTFE/PS20, (d) P/i-PTFE/PS5-S15 and (e) P/i-PTFE/S20.

schematically depicted in Figs. 10(b)–10(e). P/i-PTFE could be easily deformed and peeled off from the sample block under the shearing influence of friction pair owing to its poor mechanical properties. In addition, the low thermal conductivity caused the build-up of frictional heat, resulting in softening, melting and deformation of P/i-PTFE blend. As a consequence, the partial transfer of P/i-PTFE wear debris on the surface of friction pair developed a thin layer of transfer film, which helped to reduce μ and the wear loss. In addition, more substrate was scraped off and formed wear debris amid the sliding interface that led to a heavy mass loss, as presented in Fig. 4(b). The addition of CFs essentially enhanced the surface hardness, mechanical performance and thermal conductivity of samples by forming a reinforced CF network within PPS, which greatly improved the resistance against sliding friction pair and improved the tribological performance.

As depicted in Fig. 10(c), lower AR PSCFs tended to be peeled off and adhered to the surface of friction pair during tribological measurement. As a result, the transfer of PTFE and PSCFs on the surface of friction pair facilitated the development of a hybrid transfer film which played a synergistic role in reducing both μ and W . In addition, the addition of higher AR SCFs facilitated the development of a more robust filler network, and SCFs could not be easily scraped off from sample block. Therefore, the protruded SCFs were likely present due to the continuous action of sliding ring, as illustrated in Fig. 10(e). Consequently, P/i-PTFE/S20 had a much higher μ but lower W when compared with P/i-PTFE/PS20. Nevertheless, the tribological properties of P/i-PTFE/S20 were

better than those of P/i-PTFE.

A more compact filler packing structure was formed within PPS due to the hybrid loading of PSCFs and SCFs, as depicted in Fig. 10(d). Under such circumstances, the thermal conductivity of P/i-PTFE/PS5-S15 was greatly enhanced which was favorable for timely dissipating frictional heat during tribological test,^[46,48] thereby leading to a dramatic improvement of friction and wear performance. A comparison of the average μ of P/i-PTFE/PS5-S15 with several commercial products was displayed in Table 3. The results revealed that the average μ of P/i-PTFE/PS5-S15 was quite comparable to or lower than that of existing commercial products, which holds promise for extensive application in the advanced engineering fields.

Tribological Performance of Samples under High PV Conditions

To satisfy the operational requirements of equipment under demanding conditions, it is essential to evaluate the tribological performance of PPS-based composites under high PV (pressure $\times$ velocity) conditions.^[53] The μ and MWR of specimens which were evaluated using a customized pin-on-disc tester were displayed in Fig. 11. The testing duration for high PV measurements was 210 s, and it was divided into five testing cycles with each cycle lasting for 42 s. Fig. 11(a) shows that the high PV μ of all samples decreased with increasing testing cycles, which was associated with the gradual formation of transfer film on steel plate. Similar to block-on-ring tests, P/i-PTFE/PS5-S15 showed the lowest μ and MWR among the investigated systems.

Fig. 12 displays the optical images of the steel plate and SEM images of the worn surface of different samples after

Table 3 A comparison of average μ : this work versus commercial products.

Sample ID	Average μ	Manufacturer
P/i-PTFE/PS5-S15	0.146	This work
Gepla®X1177 (PPS/CF/PTFE)	0.25	Jiangsu NFD Composites Co., Ltd. (China)
DURAFIDE®6345A4 HF2000 (PPS/GF/PTFE)	0.23	Polyplastics Co., Ltd. (Japan)
FORTRON®6345L4 (PPS/GF/PTFE)	0.28	Celanese Corporation (USA)
FZL-4033 (PPS/GF/PTFE)	0.22	DIC Corporation (Japan)
A515 (PPS/GF/PTFE)	0.15	Toary Industries, Inc. (Japan)

Herein, the values of commercial products were taken from the datasheet from the manufacturers.

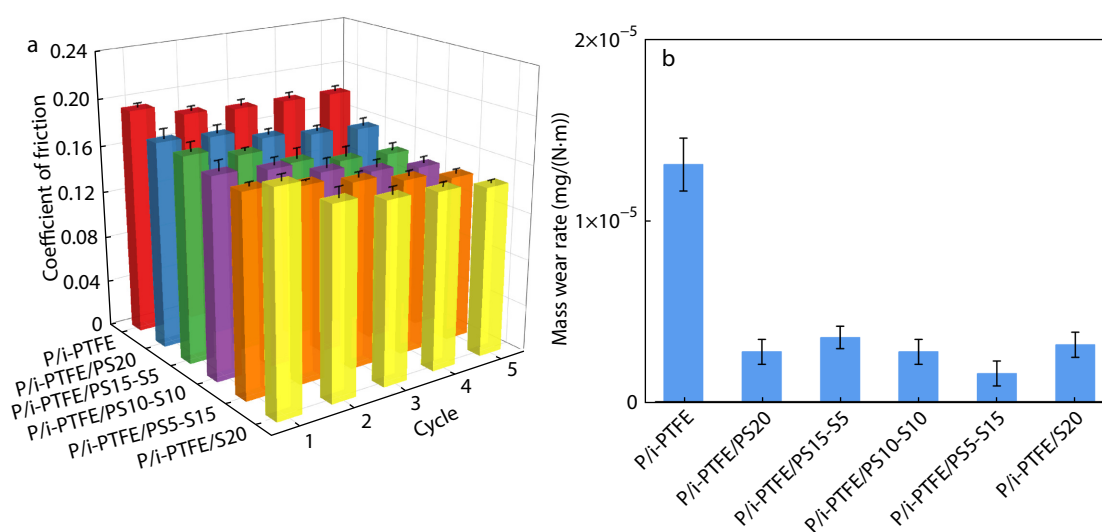


Fig. 11 The (a) μ and (b) MWR of specimens under high PV conditions.

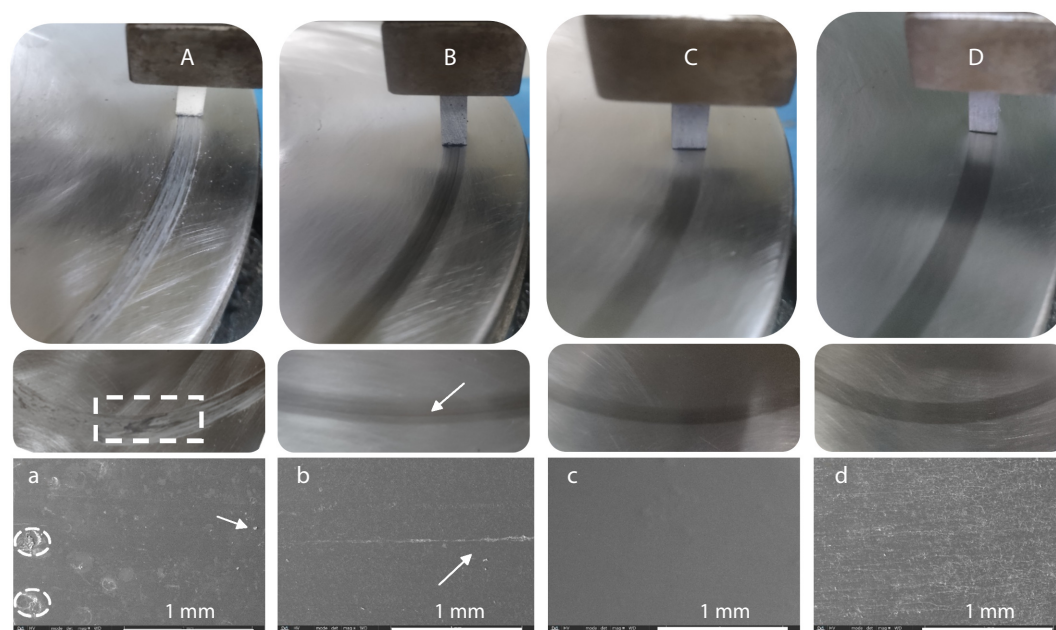


Fig. 12 The optical images of friction plate and SEM images of the worn surface of sample blocks after high PV friction tests: (A, a) P/i-PTFE, (B, b) P/i-PTFE/PS20, (C, c) P/i-PTFE/PS5-S15 and (D, d) P/i-PTFE/S20.

high PV friction tests. Fig. 12(A) shows that severe abrasion wear loss was observed for P/i-PTFE which was related to its poor mechanical properties and thermal conductivity. Moreover, the mechanical properties were greatly enhanced with the introduction of CFs. Under such circumstances, the sample blocks were able to endure the sliding action of friction counterpart. Moreover, the formation of PTFE transfer film along with the presence of CF debris was observed, which was favorable for reducing μ and mass wear loss.^[49]

Fig. 12(a) reveals the extensive collapse of sample substrate and the presence of wear debris, indicating severe adhesive wear of P/i-PTFE during friction process. This also explained the higher MAR of P/i-PTFE, as shown in Fig. 11(b). Furthermore, the worn track of P/i-PTFE/PS5-S15 was more uniform when compared with that of P/i-PTFE/PS20 and P/i-PTFE/S20, which suggested that there was a synergistic effect of adding CFs with different ARs in tuning the tribological performance by developing an intact transfer film. Notably, the worn surface of P/i-PTFE/PS5-S15 exhibited a smooth and flat appearance, indicating its excellent self-lubricating performance under high PV conditions. Thus, it can be concluded that the hybrid addition of PSCFs and SCFs was favorable for developing high-quality transfer film, thereby contributing to improving the tribological properties of PPS-based composites under high PV conditions.

CONCLUSIONS

In this work, a series of self-lubricating PPS-based composites with integrated mechanical and tribological properties were fabricated by a combined addition of CFs with different aspect ratios (PSCF: 50, SCF: about 429) along with irradiated PTFE (i-PTFE) powders. P/i-PTFE/PS5-S15 with a PSCF:SCF mass ratio of 1:3 demonstrated the best tribological performance among the studied systems. The average friction coefficient and specific

wear rate of P/i-PTFE/PS5-S15 reached as low as 0.142 and $1.63 \times 10^{-6} \text{ mm}^3/(\text{N} \cdot \text{m})$, respectively, as assessed by block-on-ring test. SEM observations revealed that the construction of a denser CF network consisting of PSCFs and SCFs was crucial for improving the mechanical and thermal properties. In the meantime, the synergistic lubrication effect of i-PTFE and CFs promoted the formation of high-quality transfer film that led to a significant improvement of tribological performance. Additionally, the high PV ($\geq 10 \text{ MPa} \cdot \text{m/s}$) friction test was conducted using a customized pin-on-disc tester, and the results revealed that the friction coefficient and wear mass loss of PPS/i-PTFE/CF composites were reduced with the synchronically addition of different aspect ratio CFs. This work provided a facile strategy for fabricating self-lubricating PPS-based composites by concurrently adding i-PTFE and different aspect ratio CFs through constructing a dense filler packing structure, which has promising applications in high-end industrial sectors.

Conflict of Interests

The authors declare no interest conflict.

Electronic Supplementary Information

Electronic supplementary information (ESI) is available free of charge in the online version of this article at <http://doi.org/10.1007/s10118-025-3486-4>.

Data Availability Statement

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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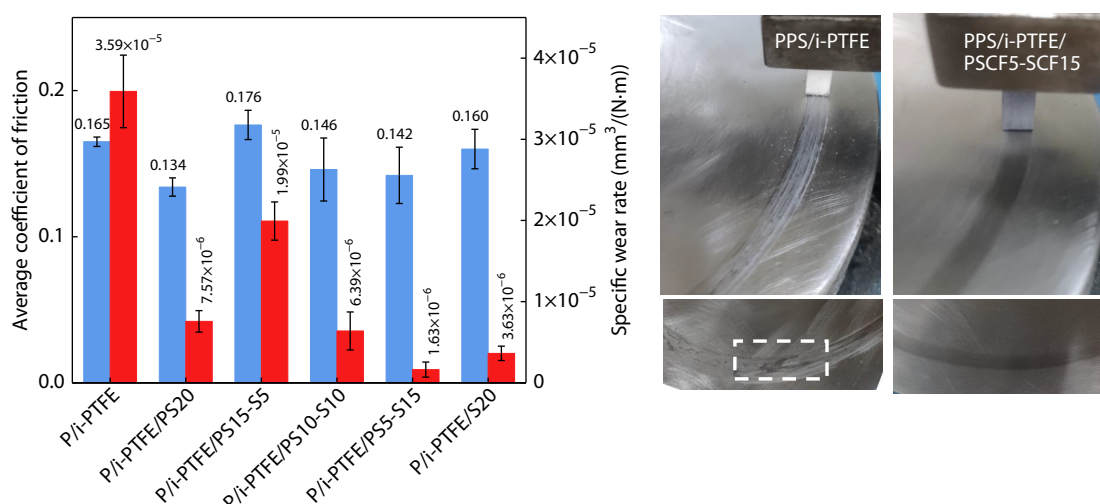
Graphical Abstract

Fabrication of Mechanically Robust Self-lubricating Polyphenylene Sulfide Composites for High PV (Pressure × Velocity) Applications

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Multi-scale carbon fibers (CFs) can give full play to their respective advantages under certain ratios, thus achieving a significant improvement in the comprehensive performance of polyphenylene sulfide/irradiation treated polytetrafluoroethylene (PPS/i-PTFE). Tribological tests at high PV (pressure × velocity) verified the viability of the composite in a demanding service environment.



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