

A Formation Mechanism and Tribological Properties of PTFE Transfer Film

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Abstract

Polytetrafluoroethylene (PTFE), as one of the most widely used solid lubricating materials, is thought to be very efficient at forming transfer film because of terrific low intermolecular force. This work investigates the tribological properties of PTFE transfer film under dry friction, and further explores its formation process and evolution mechanism. The friction coefficient is the lowest when the transfer film thickness is in range of 8-12 μm . An evaluation method for transfer film is proposed. The results show that the quality of the transfer film is closely related to the test load and velocity. This method is expected to be a general evaluation method for polymer transfer film, which can further directly evaluate the wear state of polymers, and even directly predict the tribological properties of polymers and abrasion life.

1. Introduction

Transfer films can be formed when sliding by the solid lubricant [1] on the substrate surface. It not only prevents direct contact on the substrate surface, but also decreases the shear strength of the contact layer for frictional reduction [2]. When compared with the traditional method of adding oil-based liquid or semi-solid grease friction, it is more suitable for the service requirements under the environment of modern vacuum [3], high temperature, high pressure and radiation along with more attractive attention. From the perspective of materials, the research hotspots of transfer film mainly focus on polymers, coatings [4] and fiber composites [5].

The tribological properties of polymers and their composites are attributed to the ability of forming a transfer film such as polyphenylene [6], polybutylene terephthalate (PBT) [7], ultrahigh molecular weight polyethylene (UMWPE) [8], polyethersulfone (PES) [9]. Pure polymer materials are difficult to apply to actual working conditions due to their high friction coefficient or low wear resistance, thus resulting in the more frequent use of modified polymeric materials in practice [10]. Nanomaterials have very large specific surface areas, and a very strong interface interaction with the polymer matrix, which significantly improve the performance of pristine polymers. Tribofilm formation is continuously achieved based on complex nanocomposites with the major difficulty in enhancing the load-bearing ability [11]. The nanostructures could contribute to the tribophysical and tribochemical actions for the formation of resulting transfer films [12-13]. However, some hard particles seemed to restrict the stacking of the transfer film [14]. In particular, the transfer film containing metal particles sometimes increases the surface roughness as a result of the decreased tribological properties [15]. Wang et al. [16] studied the different fillers on the effect of friction coefficient and wear rate by thermal conductivity, hardness and transfer films. The transfer film on the nitrogen-containing steel surface was thicker, smoother and stronger as opposed to that on common steel surface alone [17].

The solid lubricating coating is used to disperse various solid lubricants, reinforcing fillers, etc. in an organic or inorganic bonding system to form a special coating. Furthermore, a coating can be formed with strong adhesion after spraying or deposition and curing. It enables to improve the lubrication condition of mechanical parts, reduce friction and extend component life. The layer of assembled

graphene in the graphene coating has been reported to be compacted by its counterpart to form a dense transfer film [18], which is similar to Cp/AlSn coatings [19]. The incorporation of metal ions in the graphene oxide (GO) coating increases structure compactness to achieve high efficiency of load transfer [20]. Most popular fibrous materials are carbon fibers, glass fibers [21], *polytetrafluoroethylene* (PTFE) fibers, Kevlar fibers [22], flax fabrics [23], etc. PTFE/Kevlar fabric composites can generate transfer films on the friction surface in different sliding directions [24]. Moreover, carbon fibers can improve thermal conductivity of PTFE/Kevlar fabric composites in order to minimizing the negative effect of friction heat on the transfer film [25].

The lubrication mechanism of transfer film is somehow complex, which may vary in different materials and environments. The transfer film of PTFE coatings modified with polymethyl methacrylate (PMMA) in possession of O–C^{1/4}O and O–C bonds can effectively reduce the shear force whilst increase the wear resistance [26]. Graphene and carbon fillers are embedded within the PTFE matrix to obtain the transfer film via the formation of metal chelates [4]. Li et al. further proved carboxylic acid end-groups are chelated to the metals, thus developing robust transfer films [27]. Besides, the debris of rubbed ferromagnetic fillers are readily transferred to the counterface under the effect of tribomagnetization in order to promote the adherence [28]. Internal factors of friction pair have the influence on transfer film like surface roughness [29-30]. External environmental factors also affect the transfer film. More adsorbed H₂O molecular help to form a high-quality transfer film [31]. The continuity and thickness of the transfer film formed at various temperatures are quite different [32-33]. High temperatures tend to facilitate the formation of transfer films [34-35]. A robust transfer film provides the low wear rate and steady coefficient of friction (COF) in a temperature range from 25°C to 300 °C [36]. This is because the increase in temperature is conducive to the occurrence of tribochemical reactions, as well as to the formation of transfer films [37]. Transfer films could generally reduce friction and wear [38–40] in dry sliding, as well as in lubricated contacts such as water [41-42], sea water [43], serum lubricants [44].

PTFE is regarded as one of the most popular material in tribology today. Additionally, PTFE transfer film anchors to the counterface by means of the tribochemical synthesis of carboxylate salts [45]. At present, the consensus can be reached by various studies that the transfer film has been in a dynamic destruction-formation process [46-47], which is accompanied by irregular fluctuations in the steady stage [48]. In this work, PTFE and PTFE/Kevlar fabric composite are selected as focused material systems, and the transfer films of these two materials are prepared separately. A new standard method to evaluate polymer transfer film under simulated conditions was proposed. The formation and damage process model of PTFE transfer film was established. Finally, the tribological properties of the polymer enables to be assessed directly by evaluating the state of the transfer film in the future.

2. Experimental Details

2.1 Materials and preparation

PTFE balls (Diameter: 6mm) were purchased from Zhuotai ball co., Ltd., Shaoxing, China. The stainless-steel ball balls with the same diameter were obtained from Qiangda metals co., Ltd., Suzhou, China. PTFE/Kevlar fabric composites with a 8/3 twill-weave pattern were supplied by Shanghai Bearing Technology Research Institute, Shanghai, China. The bonding and curing process is given as follows: (i) The back of PTFE/Kevlar fabric composites is evenly coated with phenolic resin, and then adhered to the steel sheet (i.e. stainless steel) polished with sandpaper and cleaned with alcohol; (ii) Use a torque wrench to ensure that the bonded test pieces are firmly bonded, and the resin impregnation thickness is consistent;(iii) Place it in a high and low temperature (alternating) damp heat test box for the curing purpose.

2.2 Preparation of transfer film

The preparation tests of PTFE transfer film were conducted on the TRB reciprocating sliding tribometer (Anton Paar, Austria) under dry friction conditions and CETR-UMT3 tribometer (Bruker Nano Surfaces, USA). The pure PTFE transfer film was prepared by ball-on-disk wear tests, as shown in Fig. 1(a). The setup in relation to PTFE/Kevlar fabric composites by using pin-on-disk wear tests are also illustrated in Fig. 1(b).

The morphologies of worn surfaces, the thickness and macroscopic morphology of the transfer films prepared were analyzed with the aid of an Ultra Nanoindentation Tester (UNT, Anton Paar, Austria). Scanning electron microscopy (ZEISS, Sigma-500, Germany) was employed to evaluate morphological structures of transfer films. The composition and proportion of transfer films were determined by means of energy spectrum analysis (EDS). The specific wear rate of the materials was calculated using the following equation:

$$\omega = \frac{\Delta m}{\rho FL} \quad (1)$$

where ω is the wear rate ($\text{mm}^3(\text{N}\cdot\text{m})^{-1}$), Δm is the mass loss of the sample before and after the test (mm^3), ρ is the density of the specimen ($\text{g}\cdot\text{mm}^{-3}$), F is the applied normal force (N), and L is the total sliding distance (m).

3. Results And Discussion

3.1 Preparation method for transfer film

3.1.1 Friction and wear

Fig. 2(a) shows the friction coefficient and wear rate of PTFE against steel at different friction distances. With increasing the friction distance, the COFs increased rapidly and then decreased sharply, with a further stably maintained trend. From the 0 to 50 m during the accelerated wear period, the COF quickly rose to 0.2. As the friction distance continued to increase, the COF dropped over a short period of time. When the distance reached 100 m, it entered the stable period and remained at approximately 0.1. The wear rate is

basically consistent with the trend of COF. When the friction distance reaches about 50 m, the wear rate of materials continues to increase rapidly and particularly reaches a peak when the friction distance exceeds 100m. Whereas, the wear rate decreases and tends to become more stable. The wear rate decreased from the highest value of $191 \times 10^{-6} \text{mm}^3 (\text{N}\cdot\text{m})^{-1}$ to approximately $100 \times 10^{-6} \text{mm}^3 (\text{N}\cdot\text{m})^{-1}$ at a well-maintained level.

When the test distance is 25m (Fig. 2 (b)), a relatively clear friction region has appeared, but there is no obvious transfer film and wear debris. When the test distance is 25m, a relatively clear wear area has appeared, but there is no obvious transfer film and wear debris. The intermolecular force of PTFE is very small, peeling easily occurs, sporadic transfer film appears in the friction region, and wear debris accumulation occurs at the boundary of the friction region (Fig. 2 (c)). The constant friction forms "island" debris near the edge of friction region leading to greater frictional resistance [24]. As a result, the coefficient of friction and the wear rate are the highest at 50m. When the test distance reaches 75-100 m, PTFE debris, the feeding material for the transfer film to ensure the continuous formation of the transfer film gradually, appears in the friction region which edge are full of debris continuously (Fig. 2 (d)-(e)). COF and wear rate have decreased without reaching the minimum of the stable period [41]. As friction continues, the friction region is basically filled with transfer film and debris.

At this stage, the destruction and formation of the transfer film reached a dynamic balance [46-47], the COF dropped to approximately 0.1 into a stable state, while the wear rate was maintained at a low level. Ultimately, a stable and continuous transfer film is generated. This period is broadly defined as a period of stability.

Fig. 3 shows the SEM micrographs and EDS analysis diagram of the transfer film on the steel surface with different friction distances (test load:10 N and test velocity: 0.2 m/s). The main chemical components of stainless steel consist of Fe, C, Cr, Ni, Si, Mn, as well as other elements, and the characteristic element of PTFE is known to be fluorine. The comparison of element composition can determine whether a transfer film is formed on the steel surface or not. When the friction distance is 50 m, PTFE debris can be manifested in Fig. 3(a), but the EDS shows that there is no sign of F element. When considering the measurement error, there should be very little fluorine. As the friction distance reaches 100 m (Fig. 3(b)), there are 20.4 wt% of F elements, indicating the formation of a large area transfer film at this time. When the friction distance is up to 200 m (Fig. 3(c)), there are 41.1 wt% of F elements identified, and the transfer film with high coverage is formed on the surface of the steel. Since the volume of fluorine atoms are larger than that of hydrogen atoms, F-C bond appears to be shorter and negative charge of fluorine atoms for adjacent macromolecules can repel one another, resulting in elongated spiral (or twisted zigzag) arrangement. As such, fluorine atoms tend to densely pack around the carbon-carbon chain skeleton. The helical conformation just surrounds the carbon chain bonds of PTFE, which are vulnerable to chemical erosion to create a tight and complete "fluorine" protective layer, which makes the main chain of PTFE free from any external reagent. Therefore, when increasing the fluorine content, carbon content also yields an increasing trend. Fig. 5(d) shows that there are 14.8, 17.4 and 21.4 wt% of C elements at 50, 100 and 200 m. The specific process detailed in Fig. 2(b) was confirmed by the wear

surface morphology. The wear scar becomes wider, and accordingly the friction pair changes from line contact to surface contact, which creating facilitating conditions for the formation of the transfer film. The transfer film formation was similar to other polymer with good tribological properties against steel [49]. The immediate attachments of PTFE wear debris can be rapidly formed by subsequent accumulation and expansion.

3.1.2 Velocity and load

Fig. 4(a)-(b) show the COFs of PTFE-steel under different velocities and loads. Overall, the COFs began to rise extremely rapidly, which were accompanied by apparent fluctuations, and then slowly fell to a certain range and remained relatively stable. The entire process is consistent with the formation process of the transfer film. It indicates that high-quality transfer films are formed around 1500 s. Fig. 4(a) illustrates that COF is extremely unstable under low load and high velocity conditions. High velocity leads to the a relatively high increase in friction temperature. The sphere contact surface continuously generates abrasive debris, some of which are pushed to both sides of the wear scar. This phenomenon results in the transfer film being constrained by the abrasive debris on both sides during the formation process, which makes it difficult to form high-quality transfer film. However, due to the small load, the positive pressure is low and meanwhile the COFs of the three curves are not high during the stable period as well.

According to COF curve at a test velocity of 0.8 m/s shown in Fig. 4(b). The basic evolution mechanism of pure PTFE transfer film under the low speed and light load can be summarized as follows (i) The rough peaks of the steel for high hardness sliding against the counter surface can scratch the PTFE surface with low hardness with the island-shaped abrasive debris output, which causes the increase in both wear resistance and COF; (ii) The grinding chips constantly deform, squeeze and pile up, thus forming transfer film or transfer film fragments, which gradually occupy the friction interface with a clear sign of the downward trend in friction coefficient and wear rate; (iii) experienced at least once before reaching the state period. The periodicity of transfer film is in good accordance with previous work [50], which used transfer film area fractions at sliding cycle. BC in triangle explain that wear debris and transfer film fragments would stabilize the interface to provide sufficient time and energy for tribochemical reaction products to occur and accumulate in order to generate the transfer film [51].

Fig. 4(c) shows the thickness of the transfer film at different speeds under 10N. The COF has the lowest value of 0.1 at 0.8 m/s. Fig. 4(d) is the COF of transfer films with different thickness. It can be seen that the thickness range of the transfer film with better tribological properties is between 8-14 μm . The COF increases from 0 to 4 μm in thickness, tends to be reduced for greater thickness over 4 μm , and becomes stable at 8 μm . The thickness-COF curve of the transfer film is consistent with the COF-time curve, which proves that the transfer film directly affects the tribological properties [33-34]. The thickness of the transfer film continues to increase after reaching a certain level, which does not induce a greater impact on the friction coefficient.

As the speed increases, the COF decreases (Fig. 4(b)) while the thickness of the transfer film increases (Fig. 4(c)). It can be concluded that high velocity is conducive to the formation of transfer film. It can be

seen from the transfer film area and elemental analysis that increasing the test velocity leads to a higher coverage of the transfer film. Note that the main characteristic element of PTFE transfer film is fluorine according to the elemental analysis.

Fig. 5 shows the SEM morphologies and EDS analysis of PTFE transfer films at different friction velocities under a 10N load. PTFE has no branches on main macromolecular chains, and cross-links cannot be formed as a whole though its molecular contour is quite smooth. Such a contour makes PTFE have a small friction coefficient so that it is easy to transfer to the dual surface during the sliding process to form transfer film.

Under the same test conditions, the transfer film formed at different speeds requires different time. Moreover, the coverage of the formed transfer film is also different. Higher sliding speed is beneficial to the formation of transfer film. When the velocity reaches 0.8m/s, the coverage area of transfer film appears to be the largest and most uniform. EDS spectra (Fig. 5(d)) demonstrate that fluorine content is 38.8, 44.5 and 59.3% at different velocities of 0.3, 0.5 and 0.8m/s. Fluorine and carbon bond in possession of repulsive force causes the surface material to be easily sheared, thereby resulting in a low friction coefficient. Fig. 6 shows FT-IR analysis of PTFE and PTFE transfer film.

FT-IR of pure PTFE and PTFE transfer film were revealed in Fig. 6. The intense absorption peak of both of pure PTFE and PTFE transfer film at 1642 cm^{-1} , 1211 cm^{-1} and 1155 cm^{-1} arises from C=O stretching, -CF₂- antisymmetric stretching and -CF₂- symmetric stretching, respectively [52]. These same wavenumber transmittances of PTFE transfer film are lower than pure PTFE. Nonetheless, the characteristic band of -CH₂- antisymmetric stretching at 2930 cm^{-1} and -CH₂- symmetric stretching at 2849 cm^{-1} are invisible, improving that more carbon is involved [53]. These phenomena illustrate there is reaction from the friction of PTFE and steel in air. The disappearance of -CF₂- characteristic peak indicates that fluoride was worn off. The characteristic band of C=O stretching vibration band at 1664 cm^{-1} indicates that oxidation reaction has occurred at the contact surface [54-55].

3.2 Evaluation method used for transfer film

Fig. 7 shows the friction coefficient curve of the transfer film (condition: 10 N, 0.8 m/s, 200 m) against steel.

According to the COF analysis in Fig. 7, contact load and sliding speed can play a significant role in the tribology performance of the transfer film. When the contact load was 2 N, the friction coefficient increased sharply from 0.235 to 4.753 m after the beginning of the experiment and returned to the dry friction coefficient of the metal.

It is concluded that a higher test speed can effectively increase the life of the transfer film. When the test load is 2N, the maximum and minimum values of the inflection point are 0.477 m (0.3m/s) and 3.176 m (0.8m/s) respectively. When the test load is 5N, the maximum and minimum values of the inflection point are 0.390m (0.3m/s) and 1.194m (0.8m/s) respectively. When the test load is 10N, the maximum and

minimum values of the inflection point are 0.179m (0.3m/s) and 0.758m (0.8m/s) respectively. According to the average calculation, the test load is increased by 1.5 times, and the life of the transfer film is reduced by 62.41%, which is doubled and reduced by 36.52%. The test linear velocity increased from 0.3m/s to 0.5 m/s, the average life of the transfer film increased by 92.77%, and the test linear velocity increased from 0.5 m/s to 0.8 m/s, the life increased by 145.32%. The test results show that the transfer film has a certain ability to resist the load. However, the higher the load, the more obvious the damage to the transfer film. The experimental results show that the higher test velocity and load speed up to the failure of the transfer film can lead to the increase of the friction coefficient. The failure mode of the transfer film is very regular. Therefore, it can be inferred that the failure process of the transfer film relies on a large area to fall off instantly, and then the remaining part to break into small pieces and gradually fall off. However,

4. Conclusion

In summary, the formation process of PTFE transfer film under dry friction was elucidated. The influence of transfer film formed under different conditions on tribological properties was analyzed. An innovative evaluation method for the performance of transfer film was proposed with the major results summarized below:

- The formation model and damage model of the transfer film have been established. In terms of the formation process, the large transfer film can be generated whose formation directly results in the highly likely initiation for a stable period of friction coefficient. With respect to the failure process, the transfer film suddenly peeled off in a large area, and the remaining part fell off slowly into small pieces until they completely disappeared.
- The thickness of the transfer film gradually increased. The friction coefficient is the lowest when the transfer film thickness is in range of 8-12 μ
- The results show that the higher test velocity and a large load speed up the failure of the transfer film, leading to increasing the friction coefficient.
- The test load is increased by 1.5 times, and the life of the transfer film is reduced by 62.41%, which is doubled and reduced by 36.52% on average. The test velocity increased from 0.3m/s to 0.5 m/s, the average life of the transfer film increased by 92.77%, and the velocity increased from 0.5 m/s to 0.8 m/s, its life increased by 145.32%.
- This method is expected to be a general evaluation method for polymer transfer film, which can further directly evaluate the wear state of polymers, and even directly predict the tribological properties of polymers.

Declarations

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Figures

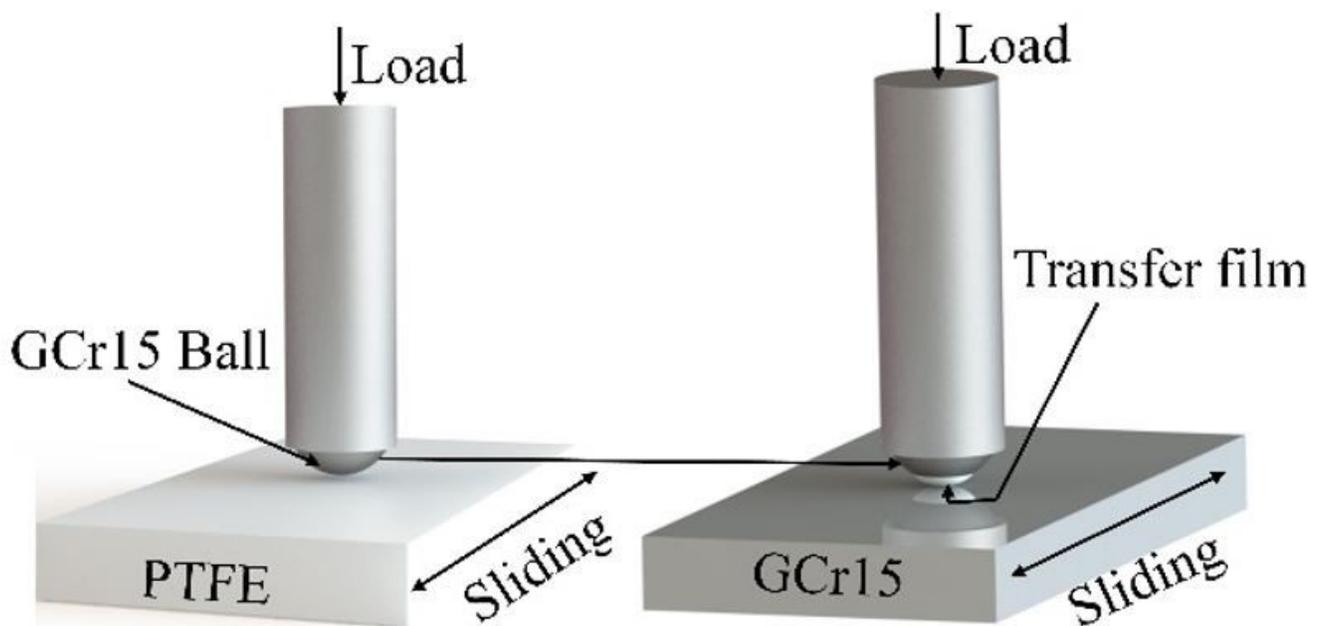


Figure 1

Schematic diagram of the evaluation method for a transfer film

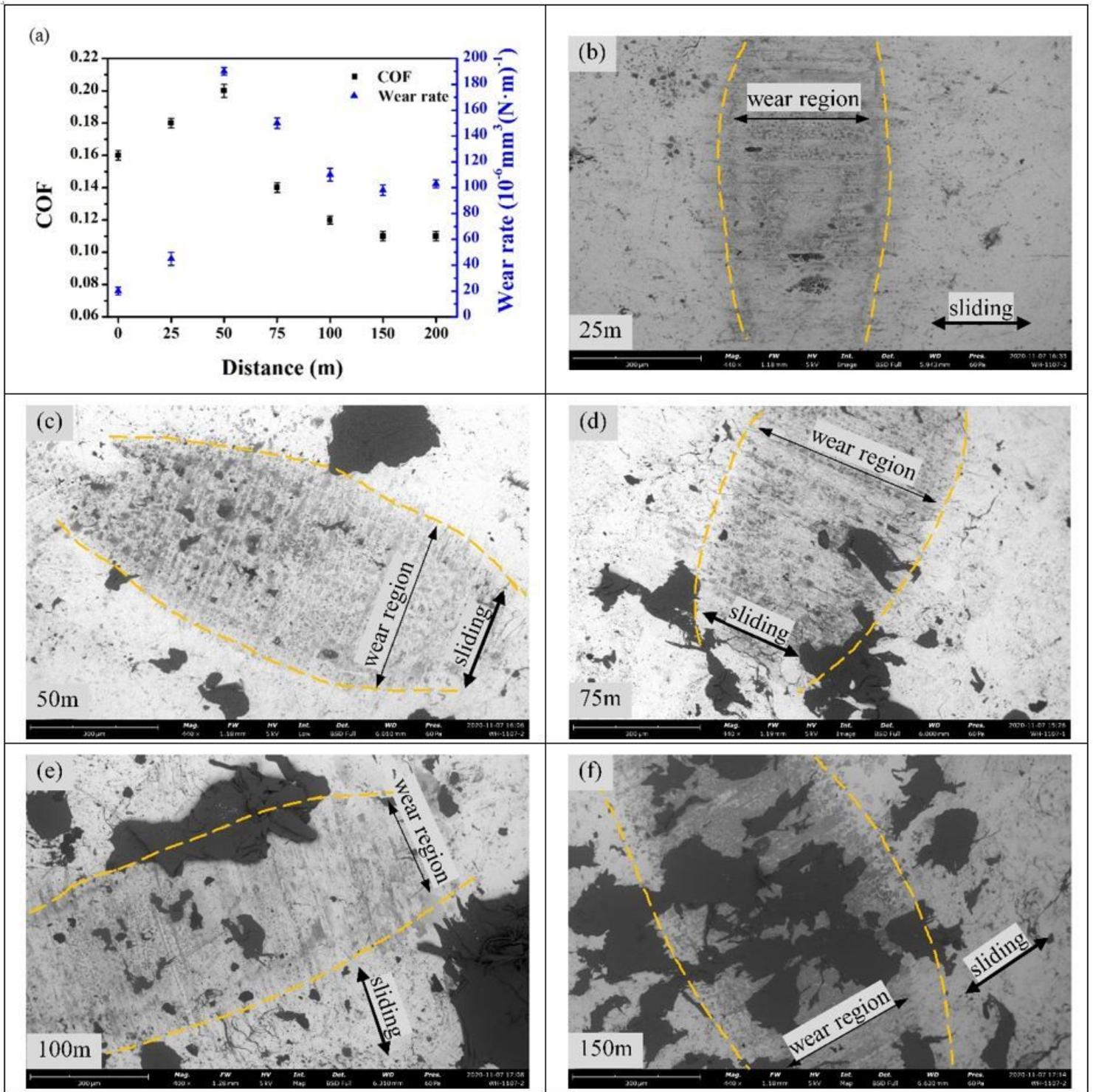


Figure 2

(a) COF and wear rate of PTFE against steel with different test distances, (b) surface morphology of ball at 25m, (c) 50m, (d) 75m, (e) 100m and (f) 150m

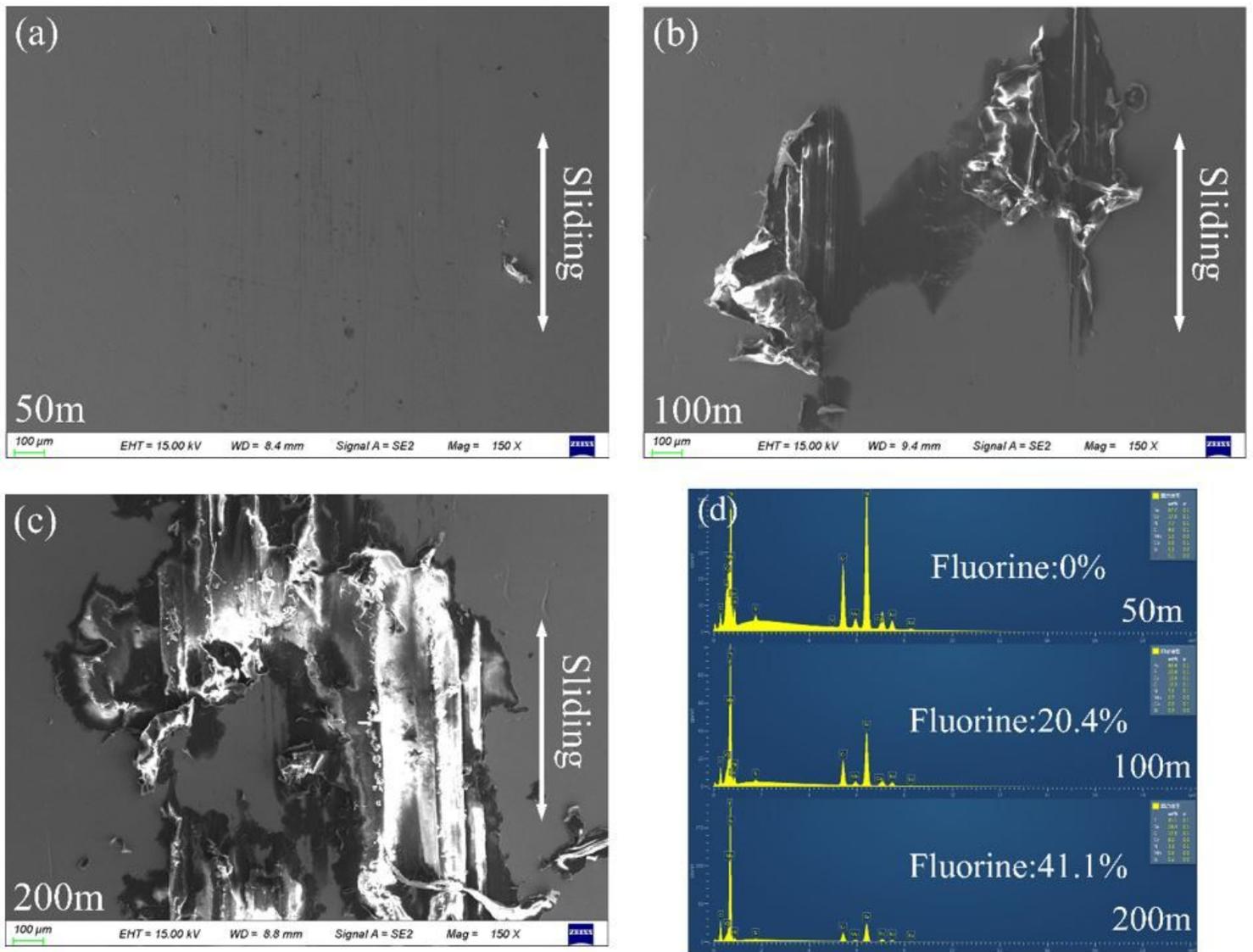


Figure 3

SEM images at different test distances: (a) 50m, (b) 100m, (c) 200m and (d) EDS of worn surfaces

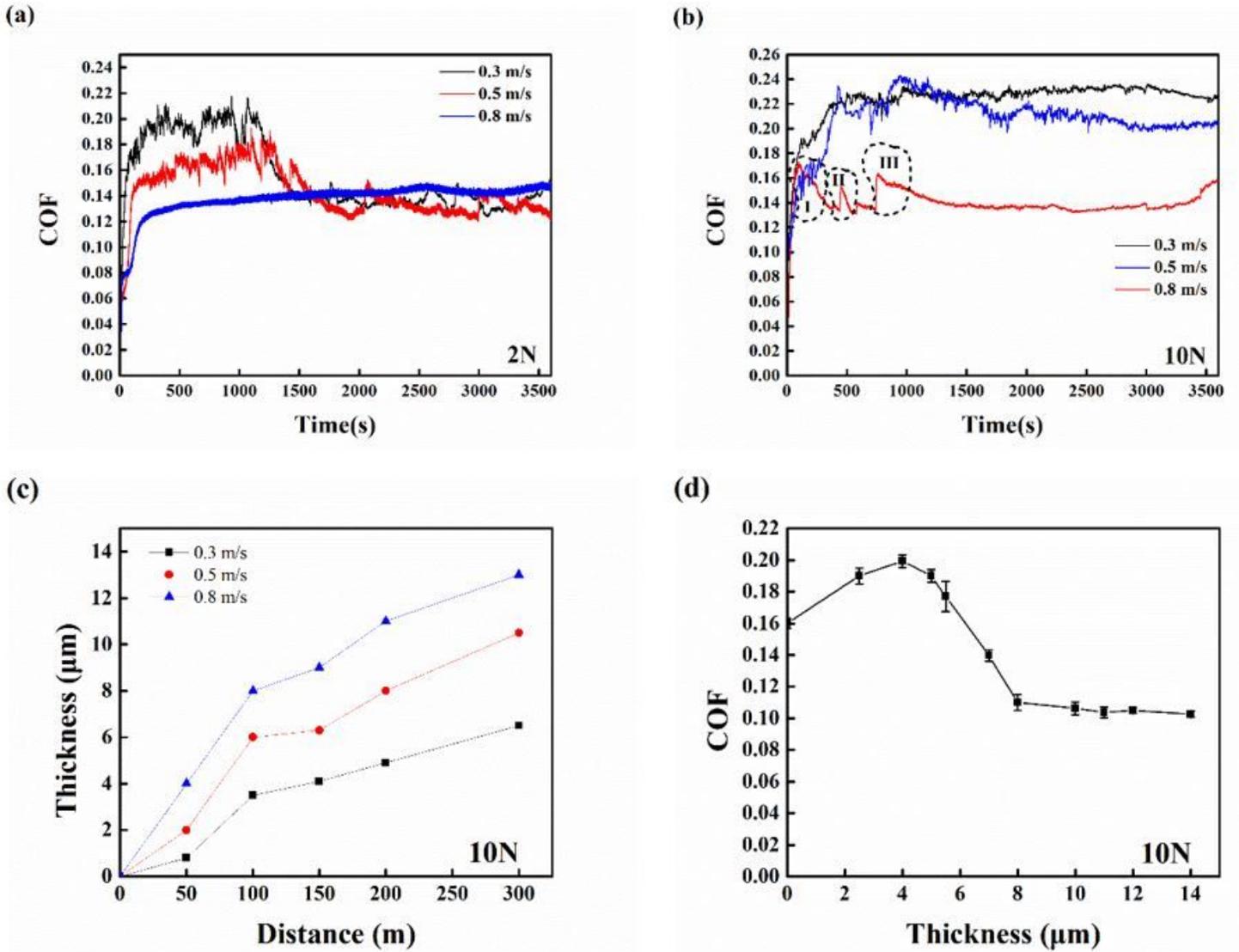


Figure 4

COF under different loads of (a) 2 N and (b) 10 N, as well as (c) different test velocities and (d) different thicknesses of transfer film

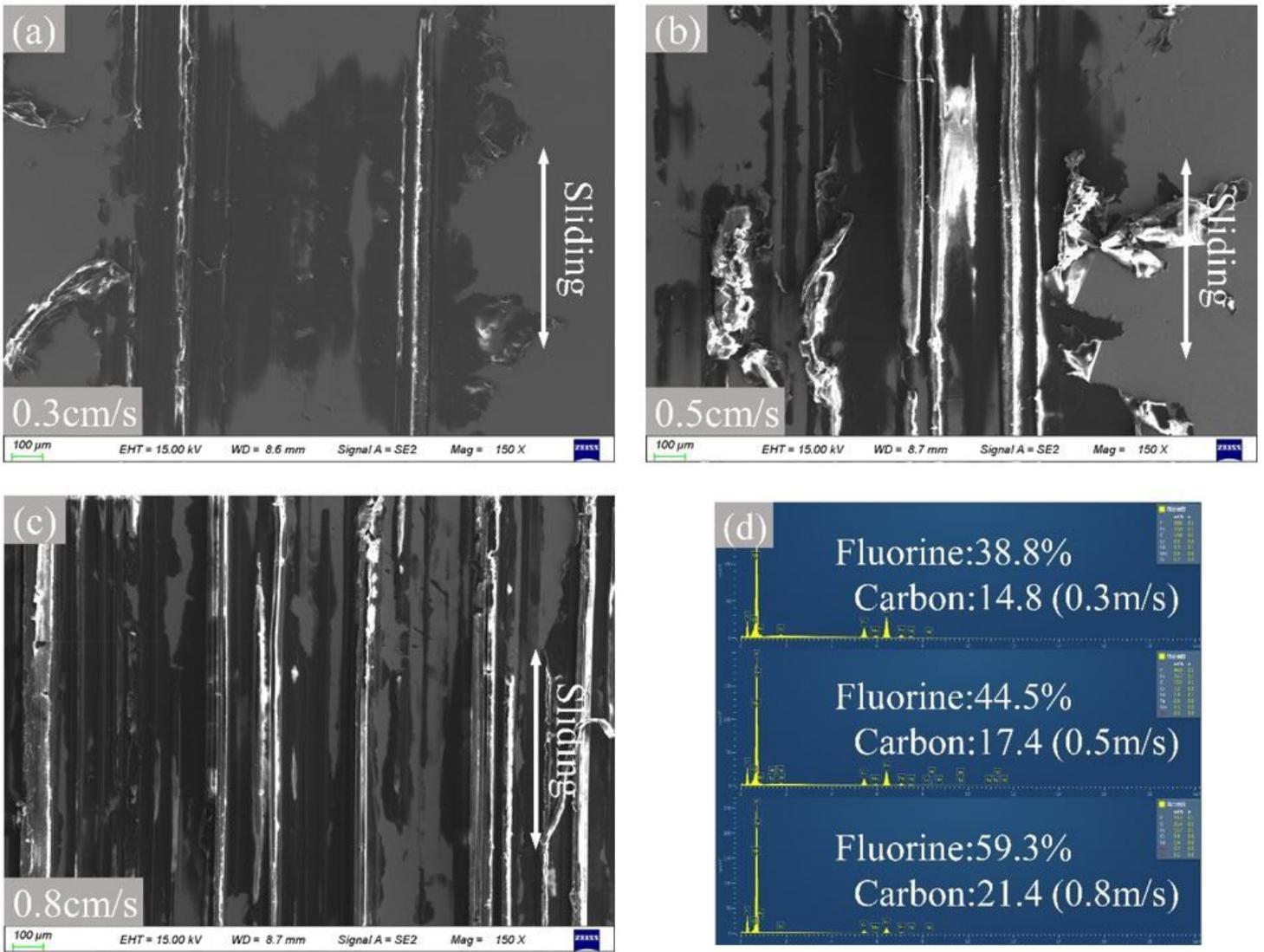


Figure 5

SEM micrographs of PTFE transfer film under 10N at different velocities: (a) 0.3m/s, (b) 0.5m/s, (c) 0.8m/s and (d) EDS spectra

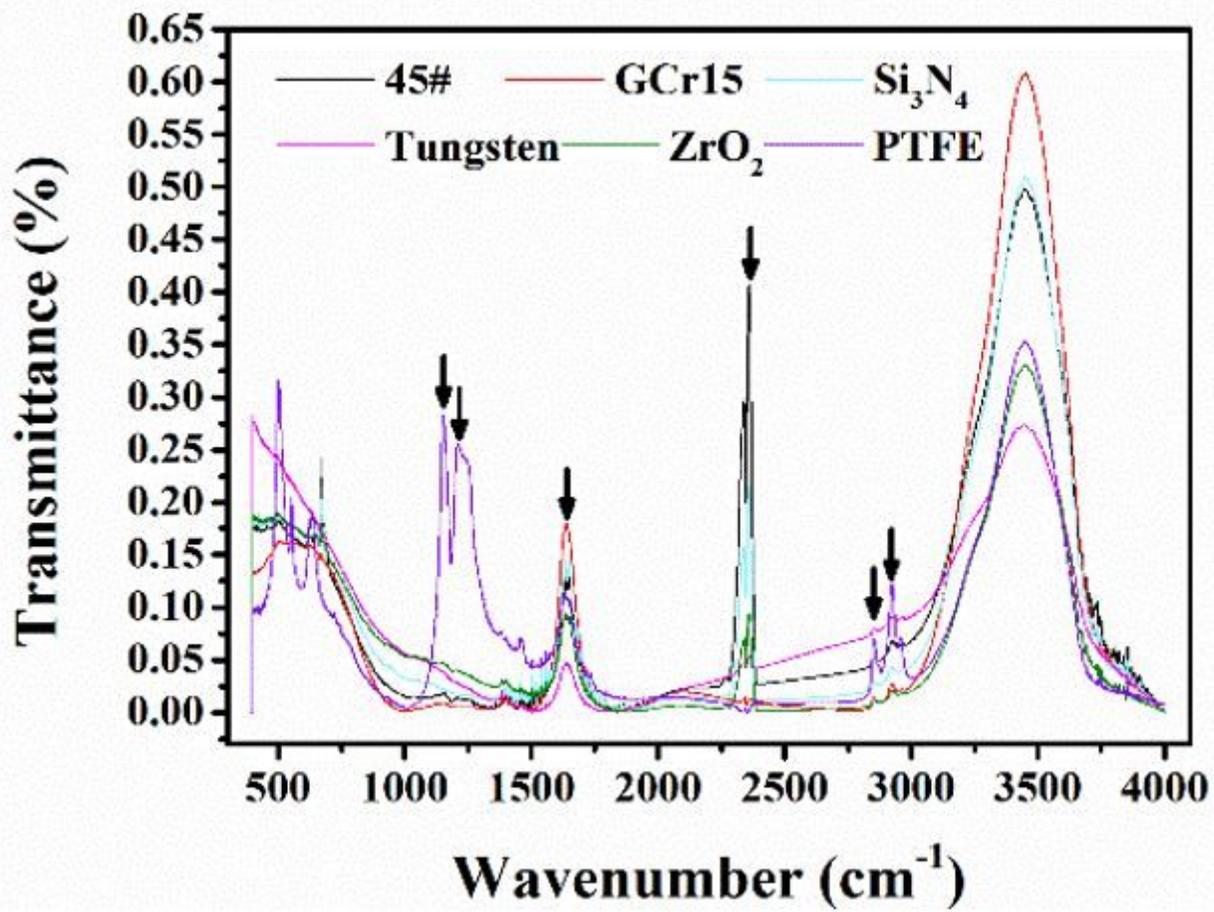


Figure 6

FT-IR analysis of PTFE and PTFE transfer film

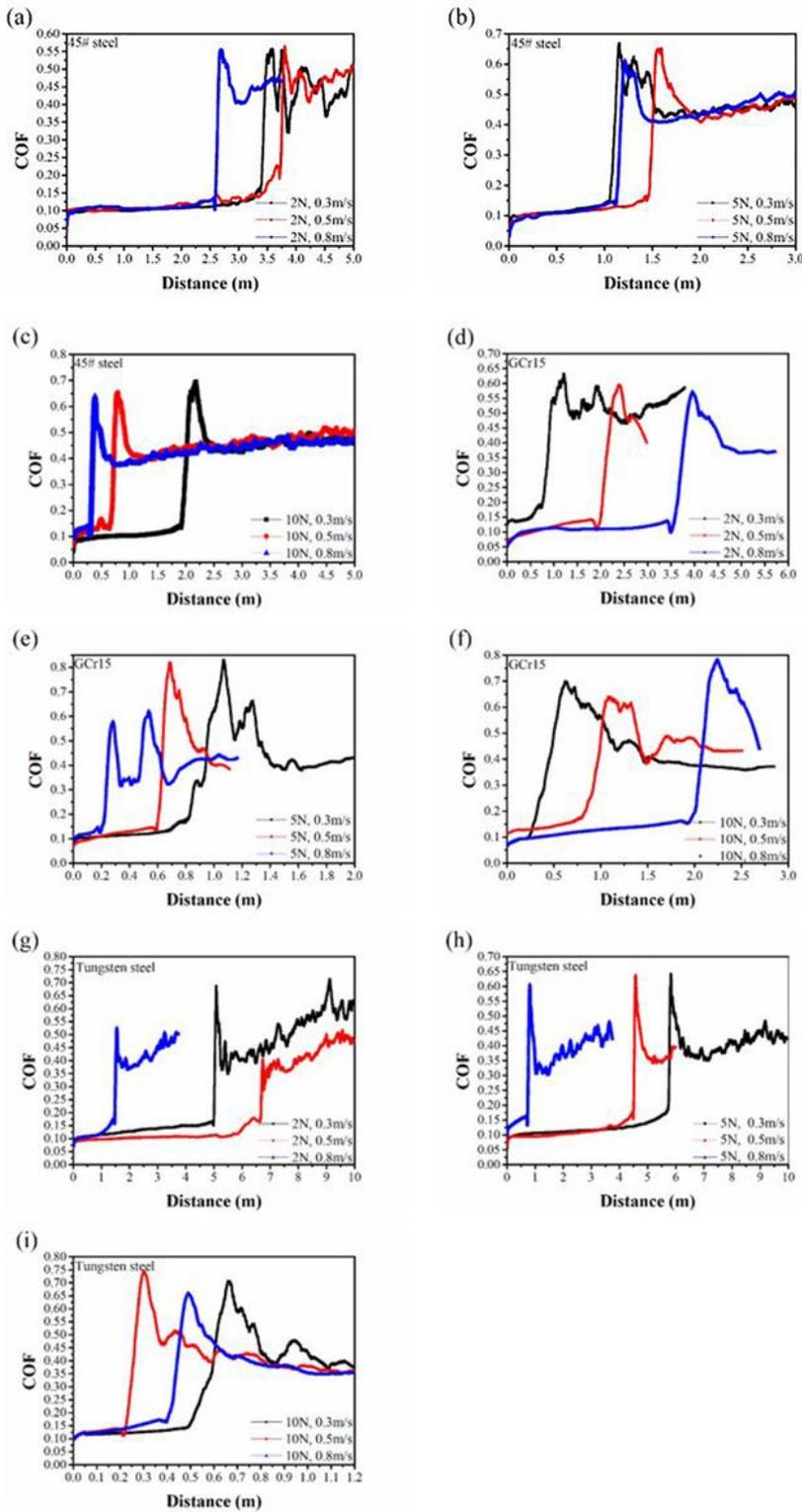


Figure 7

COFs of transfer film against metal materials at different loads and velocities: (a-c) 45#, (d-f) GCr15 and (g-i) tungsten steel

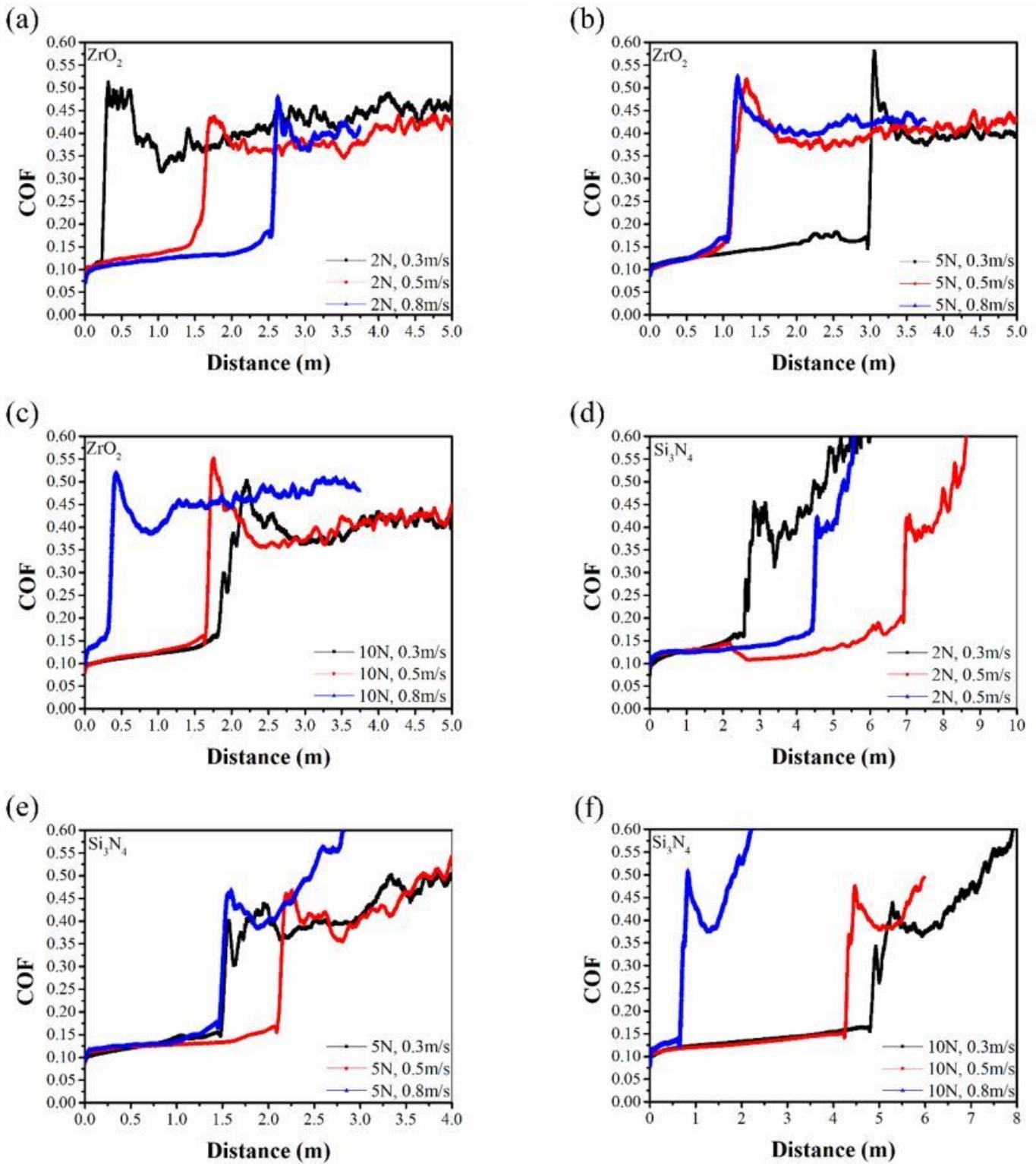


Figure 8

COFs of transfer film against ceramics materials at different loads and velocities: (a-c) ZrO₂ and (d-f) Si₃N₄