

Friction-condition-dependent sulfide and sulfate evolution on dialkylpentasulfide tribofilm studied by XANES

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Abstract: The effects of friction conditions, such as rotational speed, frictional time, and applied load, on the evolution mechanism of sulfide and sulfate on the top and bottom layers of tribofilm were investigated by total electron yield (TEY) and fluorescence yield (FY) mode X-ray absorption near-edge structure (XANES) spectra in the same beam line (4B7A). The results demonstrated that the top and bottom layers of tribofilms were covered by sulfide and sulfate. The addition of dialkylpentasulfide (DPS) could form complex nonuniform tribofilm. In addition, the friction condition (speed, load, or time) has its unique role in the generation of sulfide and sulfate at a specific depth on the tribofilm surface. The enhancement of friction conditions could promote the sulfur tribochemical reaction in a comparatively large range and alter the relative intensity of sulfurization and the sulfur-oxidizing process.

Keywords: sulfur; X-ray absorption near-edge structure (XANES); friction condition; tribochemical process

1 Introduction

Zinc dithiophosphate (ZDDP) is one of the most successful lubricant additives, and it could be widely employed to promote the friction reduction, wear reduction, extreme pressure, and antioxidant performance of base oil [1]. However, for decades, many researchers have focused on its action mechanism [2]. Some investigated the formation mechanism of ZDDP-tribofilm, and several modes were presented for a comprehensive understanding of tribofilm evolution [3]. Bell et al. [4] revealed a ZDTP-tribofilm combined with iron sulfide and/or oxide using X-ray photoelectron spectroscopy (XPS). Smith and Bell [5] noticed patches containing sulfur, zinc, and oxygen signals below the phosphorous signal by XPS, and they deduced that a dense zinc/iron sulfide/oxide forms a unique layer beneath the phosphate layer. Martin et al. [6] proposed a two-layer structure of ZDDP-tribofilm composed of

long-chain poly(thio) phosphate at the top and mixed Fe/Zn short-chain (poly)phosphate at the bottom with metal sulfide precipitates embedded in its structure. Spikes [1] described a Fe/Zn sulfide at the interface of a steel substrate and glassy Fe/Zn phosphate on ZDDP-tribofilm. Although the structure of ZDDP-tribofilm has been widely studied, the mechanism regarding the tribofilm consisting of a single P or S additive is not well understood. Sulfur additives are the second most widely used lubricant additives for industrial lubricants [7]. They possess excellent extreme pressure, antiwear, and friction-reducing characteristics, and many researchers have revealed that sulfur additives could form protective film at the frictional interfaces [8–10]. The majority of studies focused on the design of nanoparticle [11, 12] or heterocyclic compounds by sulfur [13, 14]. Therefore, the role of sulfur in the lubrication process was not explicit because of the diversity of the active element in their molecular

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structure [15, 16]. Because of the limited molecular-level research on the sulfur-containing additive/substrate interface underneath thick tribofilm, it is desirable to investigate the chemical composition of Sulfur-tribofilm.

Friction is a dynamic process, and the model of static tribofilm may be incapable of explaining the performance variation of the dynamic friction process. The dramatic difference of lubricating performance cannot be simply attributed to the structure of tribofilm or certain tribochemical products [17], indicating the unknown mechanism governing the lubrication effectiveness of tribofilm. In addition, some important facts and viewpoints have been heeded after years of research. The tribochemical reaction of ZDDP or phosphate-containing additive could be enhanced with elevated load, interval time, speed, and temperature [18, 19], indicating that frictional conditions play an important role in the lubricating effectiveness of tribofilm. Therefore, a study of the interaction between frictional conditions and the evolution of sulfur-tribofilm is necessary and important for its application in oil.

In previous work, it was reported that the concentration of dialkylpentasulfide (DPS) in synthetic ester and its emulsion played a key role in determining the structure of Sulfur-tribofilm at the lubricant-substrate interface [20]. However, the interaction between the friction condition and tribochemical products contributed to the formation of the above-mentioned tribofilm on a steel surface. In the present study, both total electron yield (TEY) and fluorescence yield (FY) mode X-ray absorption near-edge structure (XANES) spectra were conducted on tribofilm to understand the evolution of sulfur-tribofilm in the same beam line station. The study clearly showed that the friction condition was the driving force for the evolution of nonuniform tribofilm formation. In addition, the excessively harsh friction condition turned the driving force into a destructive force, and the critical point varied with the depth of the tribofilm and the specific friction conditions.

2 Materials and experimental details

2.1 Materials

The composition of the steel ball was 0.95–1.05 wt%

C, 0.15–0.35 wt% Si, 0.20–0.40 wt% Mn, 0.027 wt% P, 0.020 wt% S, 1.30–1.65 wt% Cr, 0.30 wt% Ni, and 0.25 wt% Cu, and the remainder was primarily Fe. DPS was obtained from STARRYCHEM, Shanghai, China. The chemical structure of DPS used in this study is shown in Fig. 1. The additive was diluted with pentaerythritol tetraoleate (PETO) to 1.5 wt%. PETO is a synthetic ester composed of four oleic acids and a pentaerythritol [20]. The physical features of PETO and DPS are shown in Tables 1 and 2, respectively.

2.2 Preparation of tribofilm

A four-ball tester was employed to prepare the tribofilm

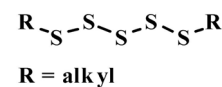


Fig. 1 Molecular structure of DPS.

Table 1 Physical characteristic of PETO.

Parameter	Index
Extrinsic feature	Yellow and transparency liquid
Density (20 °C, g/cm ³)	0.92
Viscosity index	208
Kinematic viscosity (mm ² /s)	
40 °C	65
100 °C	13
Open flash point (°C)	310
Pour point (°C)	–27
Saponification value value (mg KOH/g)	185
Iodine value (g /100g)	84
Acid value (mg KOH/g)	< 2
Hydroxyl value (mg KOH/g)	7

Table 2 Typical properties of DPS.

Parameter	Index
Extrinsic feature	Yellow and transparency liquid
Density (20 °C, g/cm ³)	1.047
Kinematic viscosity 40 °C (mm ² /s)	48.5
Open flash point (°C)	150
Pour point (°C)	–35
Total sulfur content (wt %)	40
Active sulfur content (wt %)	38
Odor	Low smell
Chrominance	2

formed in PETO under variable test conditions (rotary speed, applied load, and time), as in Fig. 2 [21]. The frictional pairs were cleaned with hexane and acetone, and they were ultrasonically cleaned to remove any trace of contaminants on the rings. The steel ball after the test (tribofilm) was gently cleaned with acetone and hexane to remove any debris and remnant oil.

2.3 XANES analysis

Chemical composition measurements were conducted by FY and TEY mode XANES to detect the valence states of the sulfur element on different depths of tribofilm. Compared with the methods reported in previous studies [22–24], the experimental data were obtained at different detection modes of the same beam line station, and the data of different depth surfaces could be compared and analyzed with the K-edge of the sulfur element [25]. An X-ray beam was monochromatized by a fixed-exit double-crystal Si(111) monochromator, and it covered the photon region of 2,460–2,490 eV with 0.30 eV resolution. The maximum analysis depths at the K-edge were approximately 5 μm and 50 nm for FY and TEY mode, respectively [19, 26]. All the spectra were collected with a step size of 1.0 eV on the region of 2,460–2,490 eV, 0.50 eV in the pre-edge region of 2,460–2,469 eV, 0.20 eV in the near-edge region of 2,469–2,485 eV, and 0.50 eV in the post-edge region of 2,485–2,490 eV.

3 Results and discussion

3.1 Effect of load on the evolution of DPS-tribofilm in synthetic ester

The sulfur K-edge FY and TEY spectra for DPS-tribofilm at different loads are shown in Fig. 3. Sulfur can exist in different valence states ranging from -2 to $+6$, with -2 as the reduced state and $+6$ as the oxidized state.

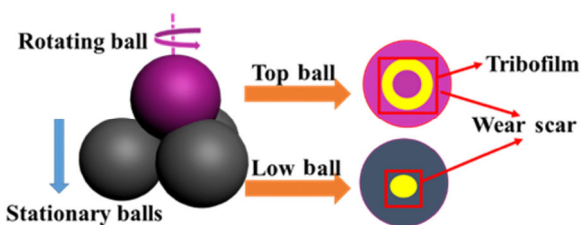


Fig. 2 Contact mode of steel balls in four-ball tester.

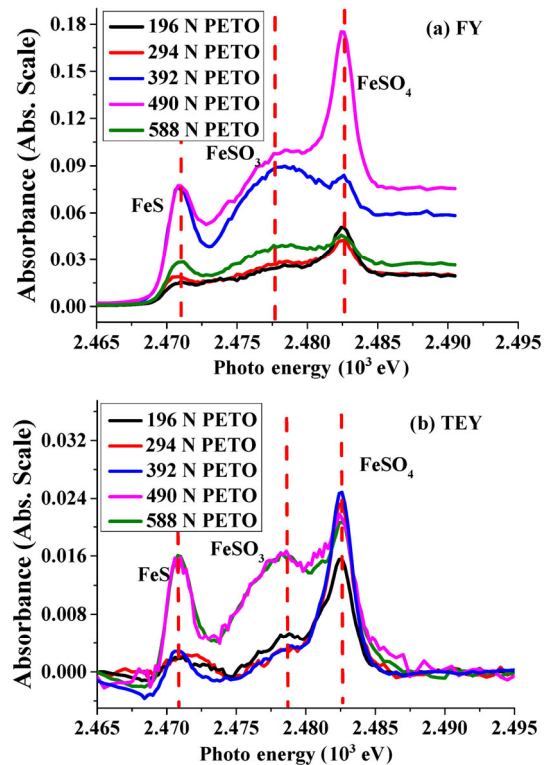


Fig. 3 Sulfur K-edge FY mode (a) and TEY mode (b) XANES spectra for DPS-tribofilm with different applied loads.

The absorption edges of different valence states can be easily distinguished by XANES spectra. The main chemical states of sulfur at all loads are FeS, FeSO₃, and FeSO₄ [10, 20]. Additionally, the change in the peak of the intensity is caused by the variation of the sulfide (FeS) and sulfate (FeSO₃ and FeSO₄) content.

For the 196 N DPS-tribofilm, there are two weak peaks and one strong peak on the bottom layer, while three strong peaks exist on the top layer, indicating that the contents of FeS, FeSO₃, and FeSO₄ in the tribofilm have an uneven distribution along the depth direction. In addition, the FeS/FeSO₄ ratio in sulfur FY spectra is higher than that of the sulfur TEY spectra, suggesting that a low load promotes the vulcanization on the bottom surface. In addition, a low load promotes the sulfur-oxidizing process on the top surface. For the bottom layer of tribofilm, the FeS/FeSO₄ ratio increases to the highest value at 392 N load, sharply decreases to a relative low value, and then has a moderate increase when the load continues to mount. The ratio of FeS to FeSO₄ at the top layer decreases with the increasing load in the low-load range (196–392 N) but increases at the high-load range (490–588 N). This

indicates that FeSO_4 and FeSO_3 are the main products in the tribofilm, but the increasing load can significantly alter the ratio of FeS/FeSO_4 and increase the content of sulfide and sulfate at the bottom and top layers of tribofilm. When the load becomes even higher (588 N), the content of sulfide and sulfate at each depth becomes less than that at a low load. In addition, the bottom layer of tribofilm suffers the greatest decline in products content, indicating that the driving force for tribofilm growth becomes a destructive force, and sulfides are more likely to form under a destructive force.

3.2 Effect of rotary speed on the evolution of DPS-tribofilm in synthetic ester

Figures 4(a) and 4(b) show plots of sulfur K-edge spectra in TEY and FY modes for tribofilm generated from DPS in PETO at different rotary speeds. The plots of Sulfur K-edge FY spectra of bottom-layer tribofilm indicate that a mixture of sulfide and sulfate species is present. The intensities of the sulfate and sulfide peaks are stronger in tribofilm formed during high-speed tests than those in low-speed tests. However, this trend has an inflection point when the rotary

speed is very high (1,760 rpm), and the intensity of the peak becomes weak. The evolution of top-layer tribofilm is different from that of bottom-layer tribofilm. Although there is more oxygen on the top layer of the tribofilm, sulfur is more easily vulcanized to form FeS on the top layer with the rise of rotary speed. The increase of FeSO_4 content at the top-layer of the tribofilm is more significant compared with that of FeS . However, the proportion of FeS becomes larger when rotary speed increases from 1,450 to 1,760 rpm. The results clearly suggest the formation of a nonuniformly layered tribofilm and a significant increase of tribochemical product content with the increase of the rotary speed, and then the ratio of sulfide to sulfate in the products is altered. When the rotary speed becomes even faster (1,760 rpm), the intensity of the adsorption peak becomes weaker, as in Figs. 4(a) and 4(b), which is similar to the situation that occurred for a high load.

3.3 Effect of time on the evolution of DPS-tribofilm in synthetic ester

The spectra were acquired for all tribofilms tested at 10 s, 10 min, 20 min, and 30 min in TEY and FY modes, and TEY spectra (Fig. 5(b)) and FY spectra (Fig. 5(a)) are shown. It is evident that the detected compounds are mainly FeS , FeSO_3 , FeS_2 [27], and FeSO_4 .

Figures 5(a) and 5(b) show that sulfate and sulfide distributed unevenly on the tribofilm of the steel ball surface and the ratio of FeS/FeSO_4 varies with depth. The change of the FeS/FeSO_4 ratio on the bottom layer shows little regularity with time, but the content of FeSO_4 increases significantly with time. This suggests that the bottom layer formed in a short period of time appears still to be underdeveloped, and the development process is easily affected by the adverse factors near the friction interface. Interestingly, the increase of sulfide and sulfate content is time dependent when the rubbing time is within 30 min. In addition, sulfide and sulfate maintain stable growth during the test period and do not reach the turning point.

The intensity of sulfate/sulfide peak is clearly increased with interval time from 10 s to 20 min. After 20-min exposure, the sulfate and sulfide on the top layer show little increase and remain at a relatively high value, while the ratio of FeS/FeSO_4 gradually decreases

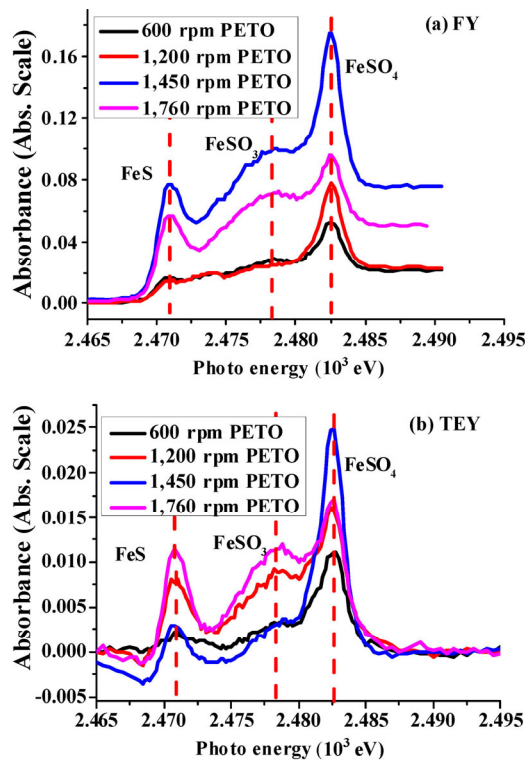


Fig. 4 Sulfur K-edge FY mode (a) and TEY mode (b) XANES spectra for DPS-tribofilm with different rotary speeds.

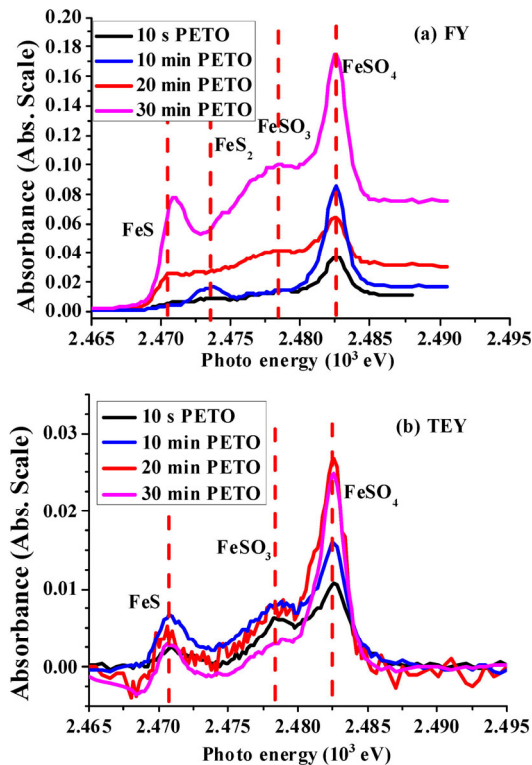


Fig. 5 Sulfur K-edge (a) FY mode and (b) TEY mode XANES spectra for DPS-tribofilm in PETO with different baking times.

over time. The low ratio of FeS/FeSO₄ formed in 30 min on the top and bottom layers of tribofilm is more conspicuous in comparison with other times. Figure 5(b) shows that 30-min tribofilm has a very strong peak of FeSO₄, similar to that on the bottom layer, indicating that the effect of time on the composite is distinct from that of load and speed, and it may take a longer time to reach a turning point.

3.4 Mechanism for the formation of tribofilm under different friction conditions

In the present work, the effect of friction conditions on the evolution of DPS-tribofilm was studied. Considering friction conditions during the frictional process, the following conditions were selected: rotary speed, applied load, and time [10, 28]. The sulfur-containing additive decomposed through the vulcanization and sulfur-oxidizing processes, and their intensity and relative proportion were greatly influenced by the friction conditions. It is well known that chemical reactions are usually affected by the reaction conditions [29], and the tribochemical reaction is no exception. Figures 1–5 show that friction conditions are both a

driving force and a destructive force for the evolution of DPS-tribofilm, and the enhanced friction conditions can transform a driving force into a destructive force.

The TEY and FY spectra of sulfur K-edge XANES suggest that sulfur exists primarily in the form of sulfate (FeSO₄ and FeSO₃) rather than sulfide (FeS) on the tribofilm, and the top layer of tribofilm easily forms FeS compared with the bottom layer (Fig. 6). Thus, sulfur is consumed in the form of sulfate rather than sulfide on the top surface of tribofilm. As friction conditions become more stringent, the shortage of oxygen on the interface may occur because of the increasing consumption of oxygen. Then, the proportion of FeS increases dramatically because of the shortage of oxygen at the frictional interface. The scenario on the bottom layer of tribofilm may also originate from the shortage of oxygen, because this layer is far from the frictional interface. An enhanced friction condition seems to break the limitation of oxygen shortage, leading to the formation of sulfide. Then, one can conclude that the tribochemical reaction is strengthened by the enhanced friction conditions, which could have a significant impact on the lubrication effectiveness on the interface [30].

The increase of load may cause many original noncontact asperities in contact, and this is more likely to result in the melting, shedding of asperities, and three-body abrasive wear of a steel surface and the rise of temperature on the surface [31]. Then, the applied load can promote the vulcanization and sulfur-oxidizing processes simultaneously, but the relative intensity of these processes depends on the depth of the tribofilm and the value of the load. In the low-load range, the tribochemical reaction between the bottom-layer and top-layer surfaces seems to take place with relatively strong vulcanization and sulfur-oxidizing processes, respectively. When the applied load is higher, however, the relative trend reverses on the surface for each depth.

The increase of rotary speed increases the chance of surface shearing, so that there are more asperities in contacts in the same period, resulting in the rapid spalling and regeneration of the asperities [32]. In this process, DPS also has more opportunities to react with the nascent surface. Friction is a dynamic process in which the previous tribofilm is destroyed and the nascent tribofilm is formed at the same time. Finally,

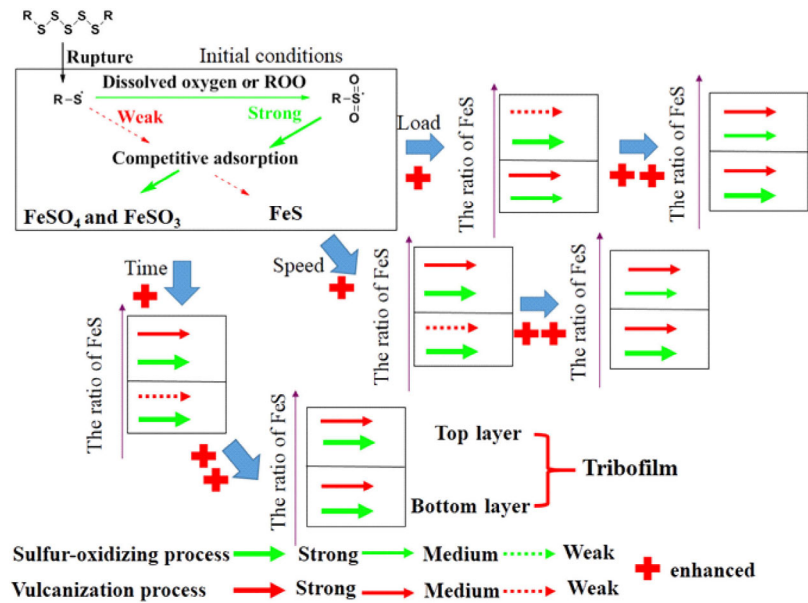


Fig. 6 Schematic explanation of the tribofilm evolution at top and bottom layer.

a dynamic balance of destruction and regeneration of tribofilm is established. In addition, the contents of sulfide and sulfate vary on the tribofilm, but the growth of tribofilm is not simply constrained with the tribofilm thickness, but it is reflected in the relative intensity of the vulcanization and sulfur-oxidizing processes. As shown in Fig. 6, the effects of load on the chemical composition of the tribofilm at the bottom and top layers are not the same, and the effect of load on the tribofilm at the same depth also has a significant difference. There is almost no absorption peak of FeS at a low rotary speed, and FeS begins to appear and occupies a certain proportion with the further increase of the rotary speed. However, the sulfur oxidation process for the formation of FeSO₄ is enhanced more prominently. This situation is slightly different at the top layer of the tribofilm, and the vulcanization process on the top surface is significantly enhanced, as well as the sulfur-oxidizing process.

The prolongation of rubbing time is beneficial to the accumulation of tribochemical products and the formation of stable tribofilm. However, with the gradual thickening of the lubrication film, the contact status can be gradually improved, which in turn affects the tribochemical reaction process. In all cases, the sulfur-oxidizing process is enhanced greatly at the top and bottom tribofilm layers. The vulcanization process is weaker than the sulfur-oxidizing process in

the top layer of tribofilm, but the vulcanization process still occupies a certain proportion. However, the vulcanization process at the bottom tribofilm layer is enhanced with the further increment of rubbing time.

5 Conclusions

The tribochemical interaction of DPS-tribofilm was investigated under various friction conditions. Based on the experimental results obtained, the following conclusions were drawn.

- (1) Simple DPS additive can form complex nonuniform tribofilm composed of sulfate (FeSO₄, FeSO₃) and sulfide (FeS, FeS₂).
- (2) The top layer of tribofilm is more prone to vulcanization than the bottom layer.
- (3) The variation of the friction conditions affects the evolution of sulfate and sulfide on the bottom and top layers of tribofilm.
- (4) The enhanced sliding process stimulates the intensity of the tribochemical reaction and alters the reaction pathways involved in the decomposition of DPS.
- (5) The friction conditions are both the driving force and the destructive force for the formation of tribofilm, indicating a transition from promoting force to destroying force along with the gradually more-rigorous friction conditions.

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