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Tribological Behaviors of Inconel 718–Tungsten Carbide Friction Pair with Sulfur Additive Lubrication

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Abstract: This work investigated the lubricating and anti-wear properties of several sulfur additives for a nickel-based superalloy–tungsten carbide friction pair. Compared with PAO40 without any active chemical compounds, the three kinds of sulfur additives could decrease the friction coefficient from 0.2 to 0.1 and the wear volume by 90%. Sulfurized fatty acid ester had the best performance under high temperature and heavy load with COF below 0.1 and the smallest wear volume. Furthermore, the lubricating mechanism was investigated by XPS. The physical adsorptive film and the tribochemical film together enhanced the friction-reducing and anti-wear performances of the lubricants. This effective lubricant for Inconel 718 can be applied to the machining of nickel-based alloy.

Keywords: Inconel 718; sulfur additives; boundary lubrication; wear mechanism

Citation: Yang, Y.; Luan, H.; Guo, S.; Liu, F.; Dai, Y.; Zhang, C.; Zhang, D.; Zhou, G. Tribological Behaviors of Inconel 718–Tungsten Carbide Friction Pair with Sulfur Additive Lubrication. *Metals* **2022**, *12*, 1841. https://doi.org/10.3390/met12111841

Academic Editor: George A. Pantazopoulos

Received: 7 October 2022
Accepted: 24 October 2022
Published: 28 October 2022

1. Introduction

Nickel-based superalloys such as Inconel 718 are widely used in aerospace, gas turbine, nuclear, and automotive industries because of their excellent high-temperature mechanical strength and corrosion resistance [1]. At the same time, nickel-based superalloys are recognized as difficult-to-machine materials. These metals exhibit serious problems during machining such as high cutting force, rapid tool wear, short tool life, and poor surface quality of the machined surface due to the physical, chemical, and thermal properties of metals [2–5]. Tungsten carbide tools are the first choice for their good thermal conductivity, high strength, and poor affinity with nickel [6]. It is of great importance to understand the relationship between nickel-based alloy and carbide material, especially the tribological behaviors, to reduce tool wear and improve tool life. The use of suitable cutting fluids can effectively improve the machining conditions of nickel-based superalloys [7–9]. The cutting fluid acts as a lubricant to reduce friction and as a coolant to cool the temperature at the cutting zone. The lubricating ability of a cutting fluid greatly influences the quality of the machined surface, as well as the tool life [10,11].

The chemical additives in the cutting fluid can act with the metal surface at high temperature and pressure; thus, a lubricant film is formed to reduce friction between the rake face and chips, the flank face, and machined surfaces. Sulfur additives are well known for their extreme-pressure performance and anti-wear characteristics [12–14]. The sulfur compounds, under extreme-pressure conditions, undergo chemical decomposition causing sulfur release (rupture of the R–S bond) and their reaction with the metallic surface that promotes the formation of an inorganic iron sulfide layer [12,15]. Most of the cutting fluids are designed for ferrous metals [11,16–18]. However, there is a matching problem between the additives and the workpiece materials [19,20]. Whether these additives have the same reaction with nickel-based superalloys is not clear. Few scholars have studied the lubricants for nickel-based alloy–tungsten carbide contacts. Moreover, metal working fluid which consists of several chemically active additives has a very complex chemical composition. The individual actions of each component are not easily identifiable. However, it is necessary for new metals such as nickel-based superalloys whose characteristics are very different from traditional ferrous metals. Therefore, it is important to separate contributions from different types of additives to the lubrication so as to select the most efficient lubricant molecules and optimize the components of the cutting fluid. This work focuses on the lubricating effect of different sulfur additives for the nickel-based superalloy (Inconel 718)–tungsten carbide (YG8) tribopair, and the lubricating mechanism is further investigated.

2. Experimental Details

Inconel 718 is the most widely used superalloy. YG8 (WC-Co) tungsten carbide is the optimal tool material for nickel-based superalloy machining. The specimen used in this paper was bought from Hengshihui company, Jiangsu, China. Table 1 shows the chemical composition of Inconel 718 superalloy. The main component is Ni (60.01 wt.%) followed by Cr (17.28 wt.%) and Fe (15.58 wt.%). The Table 2 displays the mechanical parameters of Inconel 718.

Table 1. Chemical compositions of Inconel 718 superalloy (wt.%).

Ni	C	O	Al	Ti	Cr	Mo	Nb	Fe
60.01	2.31	0.62	0.29	0.51	17.28	1.25	2.15	15.58

Table 2. The mechanical parameters of Inconel 718.

Young's Modulus (GPa at 20 °C)	Poisson's Ratio	Hardness (HRC)	Yield Strength (MPa)
199.9	0.3	26.4	1035

The commercial sulfur additives were bought from Symarin company, Shanghai, China. Each additive contained a different amount of sulfur in the molecule (by weight), and the sulfur bonding mechanism was different in each additive. According to the manufacturer's data, the sulfur content of sulfurized olefin was as much as 40%, more than that of sulfurized fatty acid ester (17%) and sulfurized lard (10%). The kinematic viscosity of sulfurized olefin was much smaller than that of the other two kinds of additives for the smallest molecular weight. Table 3 shows the available information about the lubricants. Furthermore, PAO40, the viscosity of which is 396 mm^2/s, was used as pure oil without any active elements for comparison.

Table 3. The basic parameters of sulfur additives.

Name	Sulfur Content	Kinematic Viscosity mm^2/s (at 40 °C)	Appearance	Flash Point (°C)
Sulfurized olefin	40%	45	pale yellow	150
Sulfurized fatty acid ester	17%	582	tan	210
Sulfurized lard	10%	900–1300	dark brown	>160

The frictional tests were carried out utilizing a ball-on-disc apparatus SRV-IV (Optimol, Munich, Germany) under different lubricating conditions. The schematic diagram of the tester is displayed in Figure 1. The discs were Inconel 718 with a hardness of HRC 35. All of the specimens were polished before frictional tests by an automatic polishing/grinding machine, and a surface roughness (Sa) less than 40 nm was obtained. The counter specimen was tungsten carbide YG8 ball with a diameter of 10 mm and surface roughness (Sa) of 25 nm. The hardness of the carbide ball was 89HRA. The samples were ultrasonically cleaned using acetone and ethanol and then ultrapure water successively, each for 10 min, before tests. The upper ball slid reciprocally against the stationary disc with an amplitude of 2 mm and frequency of 20 Hz for 5 min. Before the frictional test, plenty of lubricant was dropped onto the surface of the disc, and the ball returned to yield a normal load of 100 N.

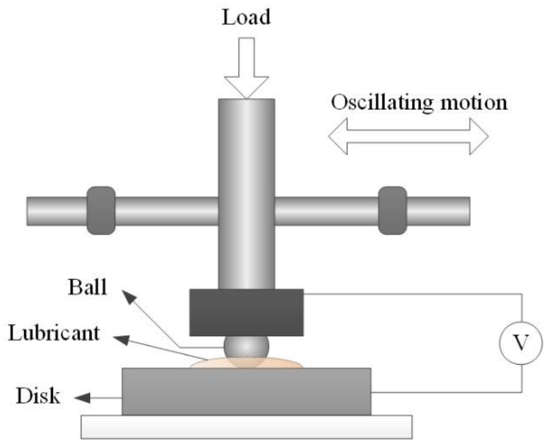

Figure 1. The schematic diagram of SRV-IV tribo-tester.

After the frictional tests, the morphology of rubbing surfaces was observed using a LEXT™OLS5100 laser scanning confocal microscope (Olympus, Tokyo, Japan), and wear volume is calculated. Each test was repeated three times, and the average values were used. The relative errors were on the order of ±5%. A Quanta200 scanning electron microscope (SEM) (FEI, Hillsboro, OR, USA) combined with energy dispersion spectrometry (EDS) (FEI, Hillsboro, OR, USA) was used for the surface analysis of the investigated materials. The chemical compositions of the worn surfaces were characterized using a PHI Quantera SXM X-ray photoelectron spectrometer (ULVAC-PHI, Chigasaki, Japan).

3. Results and Discussions

3.1. Friction and Wear Properties Lubricated with Sulfur Additives

The curves of the friction coefficient and wear volume of the lower samples lubricated by the four kinds of lubricants are shown in Figure 2. For PAO40, the friction coefficient increased to 0.7 rapidly in the running-in period. Then, it decreased gradually and stabilized at about 0.2 after 200 s. When using sulfurized olefin as lubricant, the initial friction coefficient fluctuated to 0.17, before remaining stable at about 0.14 after 100 s. For sulfurized fatty acid ester and sulfurized lard, the friction coefficients remained stable at 0.1 and 0.11, respectively, until the end of the experiment. Compared with PAO40, the three kinds of sulfur-containing additives could reduce the friction coefficient significantly for the nickel-based superalloy. The friction coefficient of sulfurized fatty acid ester was the smallest, while that of sulfurized olefin was 27% larger.

Considering that tungsten carbide is much harder than nickel-based superalloys, wear occurs on the surface of the disc apparently while no wear is observed on the ball. After the frictional tests, the remaining solution and debris on the wear track are washed away by water and alcohol. From Figure 2b, it can be seen that the wear volume of the disc lubricated by PAO40 was 2.4×10^7 μm^3, about ten times that lubricated by the sulfur additives. Thus, basic oil without any active chemical elements failed to lubricate nickel-based superalloy under boundary lubrication. Compared with PAO40, the sulfur additives showed good lubricating and anti-wear performance for the Inconel 718–tungsten carbide friction pair. It is easy to generate inorganic protective films in the tribochemical reaction for an excellent anti-wear effect. For sulfurized fatty acid ester and sulfurized lard, the wear volumes were 2.6×10^6 μm^3 and 2.8×10^6 μm^3, respectively. With sulfurized olefin lubrication, the wear volume was 4.1×10^6 μm^3. The relatively big wear volume is related to the long running-in period during the friction test. A lubricating film was formed, but it was not stable enough to prevent direct contact between Inconel 718 and tungsten carbide completely during the running-in period. It can be concluded that sulfurized fatty acid had the best lubrication for Inconel 718 with the smallest COF and wear volume.

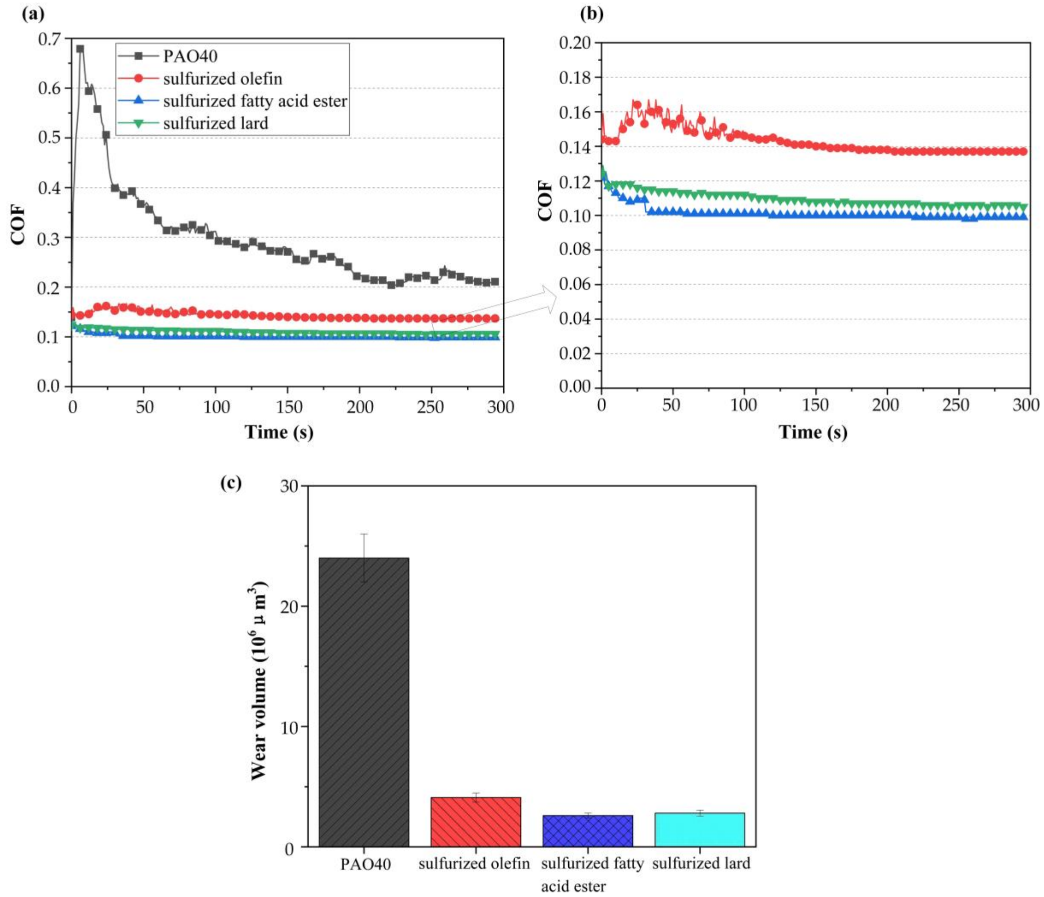

Figure 2. (**a**) Friction coefficients with (**b**) enlarged view, and (**c**) wear volume of the discs lubricated by different lubricants (F = 100 N, f = 20 Hz, L = 2 mm).

The micro-morphology of the friction pairs was obtained by SEM as shown in Figure 3. It can be seen that there were furrows on the surface of the superalloy discs and irregular block materials on the surface of the balls lubricated by the three kinds of additives. From Figure 3(a1), delaminated scars and abrasive particles can be seen on the surface of the disc lubricated by sulfurized olefin. There were obvious furrows on the worn surface lubricated by sulfurized lard (Figure 3(c1)). Furthermore, large dark blocky materials were detected on the ball surface (Figure 3(c2)). The worn surface was compared to the smoothest with no adhesive scar when lubricated by sulfurized fatty acid ester (Figure 3(b1)). However, there were still patches on the surface of the ball (Figure 3(b2)). To ascertain the ingredients of the materials on the surface (the red frame in Figure 3), EDS analysis was conducted. The results show that the nickel-based superalloy on the ball surface was transferred from the discs and adhered to the balls (Figure 3d). The materials on the balls were the same; thus, the EDS result is listed once. The main wear mechanism of the superalloy was significant adhesion, delamination, and ploughing. From the EDS results of the worn surface of the discs, the sulfur element was detected, which did not belong to the alloy. This demonstrates that sulfides formed on the metal surface during the friction process. However, the sulfur contents on the surface of the discs lubricated by the additives were different, seen from Table 4. The sulfur content of the worn surface lubricated by sulfurized olefin was the highest (12.37%), while the adhesion on the ball was relatively mild. It should be considered that the active sulfur content of sulfurized olefin was also the highest. The sulfur content of the worn surface lubricated by sulfurized lard was the lowest (1.34%) with the most adhesive blocky material on the ball. For the sulfurized fatty acid ester, the adhesion on the ball was significantly decreased compared with the sulfurized lard. This indicates that the sulfide generated on the metal surface during the friction process played an important role in reducing the adhesion of nickel-based superalloy to tungsten carbide ball during friction. Sulfurized fatty acid ester contains active sulfur that can react with the metal surface at a lower temperature; hence, the surface quality of the disc and the adhesion of the ball were better than those lubricated by sulfurized lard. Meanwhile, the sulfurized fatty acid ester molecule has a long carbon chain which helps to separate the two surfaces; thus, direct contact was avoided compared with sulfurized olefin.

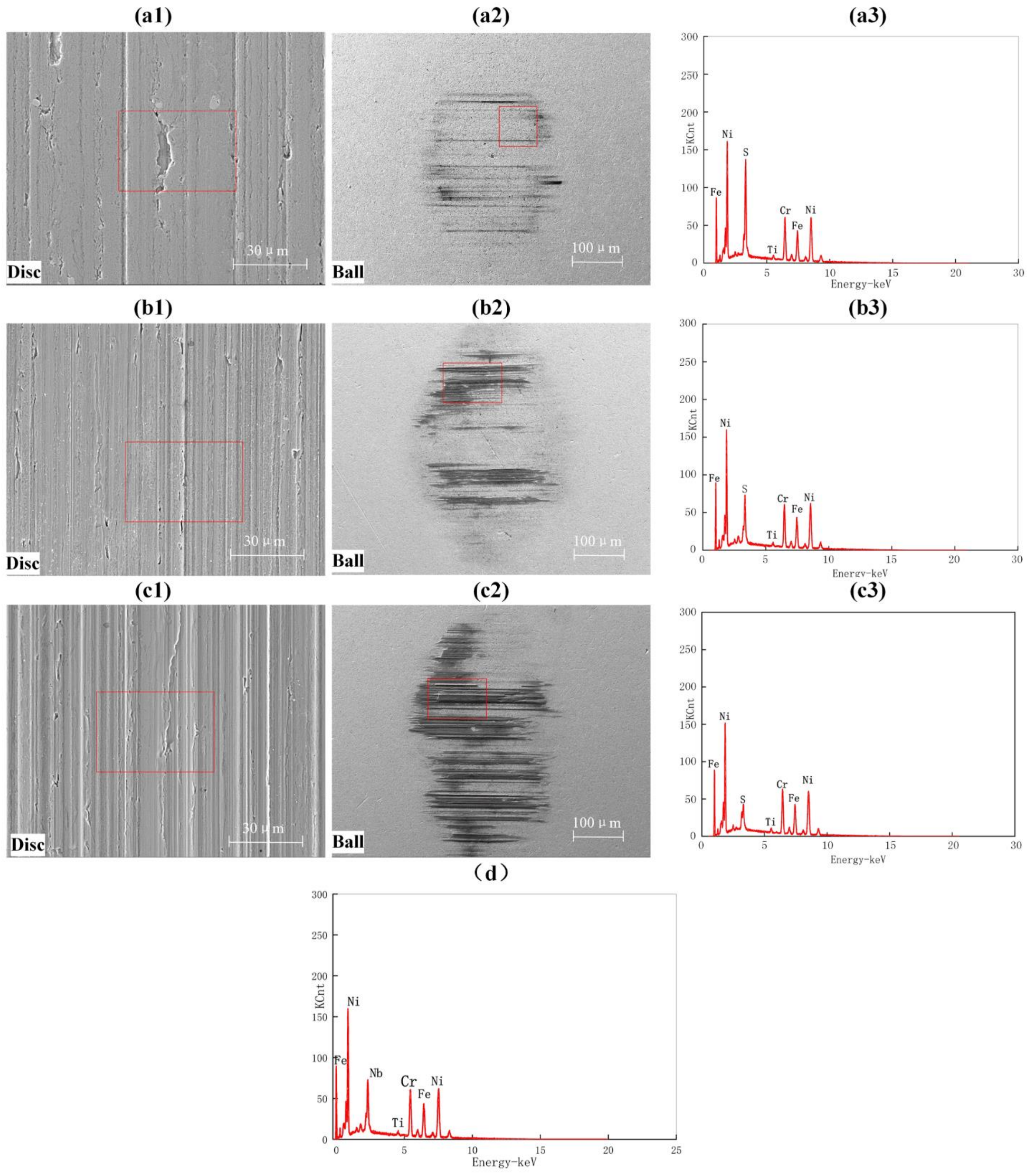

Figure 3. SEM morphology of the tracks on Inconel 718 and tungsten carbide ball lubricated by (**a1**,**a2**) sulfurized olefin, (**b1**,**b2**) sulfurized fatty acid ester, and (**c1**,**c2**) sulfurized lard; (**a3**,**b3**,**c3**) EDS spectrum of the red box in (**a1**,**b1**,**c1**); (**d**) EDS spectrum of the red box in (**a2**).

Table 4. The elements content of the above EDS analysis.

Element (wt.%)	Ni	Fe	Cr	S	C	Al	Si	Ti	Nb
(a3)	45.23	18.2	16.8	12.37	6.09	0.36	0.16	0.96	–
(b3)	43.71	17.67	16.75	2.71	6.75	0.37	0.97	0.87	10.19
(c3)	46.44	18.93	18.36	1.34	4.54	0.52	0.14	1.35	8.38
Element (wt.%)	**Ni**	**Fe**	**Cr**	**Nb**	**Ti**	**C**	**W**	**O**	–
(d)	33.92	16.54	16.01	8.54	1.13	7.31	9.04	1.5	–

3.2. Temperature Effect

Temperature is very important for the chemical reaction of additives with tribo-surface. The influence of temperature on the friction and wear behavior of Inconel 718 sliding against WC-Co under the lubrication of sulfur additives was investigated. The experimental temperature was increased to 150 °C with the other experimental parameters the same as those in Section 3.1. Figure 4a shows the friction coefficient curve at 150 °C. For sulfurized olefin, the fluctuation of the friction coefficient was reduced at the beginning of the test compared with that at 30 °C. Furthermore, the friction coefficient stabilized at about 0.18 after 20 s. For sulfurized fatty acid ester and sulfurized lard, the friction coefficient remained at 0.1 from the beginning to the end of the test without running-in time at 150 °C. This indicates that the lubricant film formed rapidly and remained more stable under high temperature as expected. The wear volumes of the Inconel 718 discs are shown in Figure 4b. Compared with that at 30 °C, the wear volume of the discs with different sulfur lubricants did not change much. The wear volume lubricated by sulfurized fatty acid ester was slightly smaller compared to the other two kinds of additives. It can be seen that the sulfur lubricants had very good temperature stability for nickel-based superalloys.

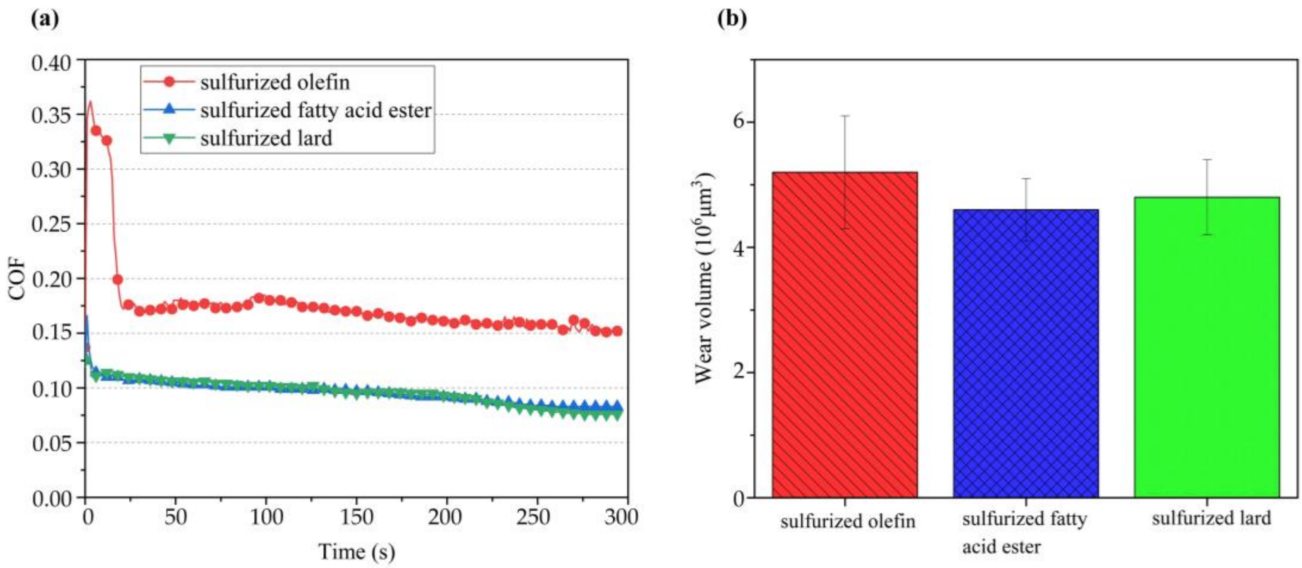

Figure 4. (**a**) Friction coefficients and (**b**) wear volume when lubricated by sulfur additives at 150 °C (F = 100 N, f = 20 Hz, L = 2 mm).

The morphology of the lower disc and the upper tungsten carbide ball after frictional tests at 150 °C was observed by SEM, and the pictures are shown in Figure 5. Compared with 30 °C, the furrows on the surface of the discs decreased, especially for sulfurized fatty acid ester and sulfurized lard (Figure 5(b1,c1)). This indicates that high temperature promoted the formation of a lubricant film, thus increasing the surface quality of the nickel-based superalloy. With respect to the surface of the balls, the adhesive materials were also decreased when lubricated by sulfurized fatty acid ester and sulfurized lard (Figure 5(b2,c2)). However, the block material on the surface of the ball lubricated by sulfurized olefin increased significantly, indicating that adhesion was aggravated, as seen in Figure 5(a2). EDS analysis was performed on the wear scar of the nickel-based superalloy and the adhesion area on the tungsten carbide (the red box in Figure 5). The dark materials on the balls were the same; thus, the EDS results are listed once (Figure 5d). A nickel-based superalloy was transferred from the disc and adhered to the ball. It can be speculated that, under high temperature, the main wear mechanism of the superalloy was adhesion. The elements content of the EDS analysis is listed in Table 5. From the EDS spectrum of the surface lubricated by sulfurized fatty acid ester (Figure 5(b3)), it can be seen that the peak of sulfur in the wear scar increased significantly and the sulfur content increased from 2.71% (30 °C) to 16.33% (150 °C). The sulfide acted as an effective lubricant film, which significantly decreased the adhesion on tungsten carbide balls and the ploughing on nickel-based superalloy discs. For sulfurized lard, the sulfur content increased from 1.34% (30 °C) to 5.43% (Figure 5(c3)), and the surface quality of the frictional pairs improved. As for sulfurized olefin, the sulfur peak did not change very much, and the content of sulfur was 11.63%. This indicates that high temperature did not promote the tribochemical reaction of sulfurized olefin with nickel-based superalloy. Considering that the flash point of sulfurized olefin is 150 °C, when the experimental temperature increased to 150 °C, the sulfurized olefin began to volatilize continuously, and the material left in the friction area was unstable. This led to the aggravation of adhesion at 150 °C lubricated by sulfurized olefin. The flash points of the other two sulfur lubricants were both higher than 150 °C, and the molecules remained stable with temperature increasing. Moreover, the tribochemical reaction was promoted, leading to better surface quality and less adhesion.

Table 5. The elements content of the above EDS analysis.

Element (wt.%)	Ni	Fe	S	C	Ti	Cr	Nb	—
(a3)	41.8	16.12	11.63	6.22	0.72	16.26	3.68	–
(b3)	43.71	17.67	16.33	6.45	0.71	15.4	10.19	–
(c3)	47.56	17.7	5.43	4.35	0.81	17.27	4.16	–
Element (wt.%)	**Ni**	**Fe**	**Nb**	**Cr**	**Ti**	**C**	**W**	**O**
(d)	45.29	14.14	3.73	13.7	0.7	6.22	10	3.51

3.3. Load Capacity

The extreme-pressure performance of the lubricants is also an important index used to measure the lubricating property. To illustrate the performance under extreme pressure, the load slope test results of the three kinds of sulfur additives are shown in Figure 6a. The test load increased from 100 N to1000 N in steps of 100 N. The friction coefficient of sulfurized olefin fluctuated significantly with each load increase. For sulfurized fatty acid ester and sulfurized lard, the friction coefficients remained stable at about 0.1 before the load increased to 600 N. When the load was more than 600 N, the friction coefficients had a little fluctuation. Thus, the load-bearing capacity of sulfurized fatty acid ester and sulfurized lard was better than that of sulfurized olefin.

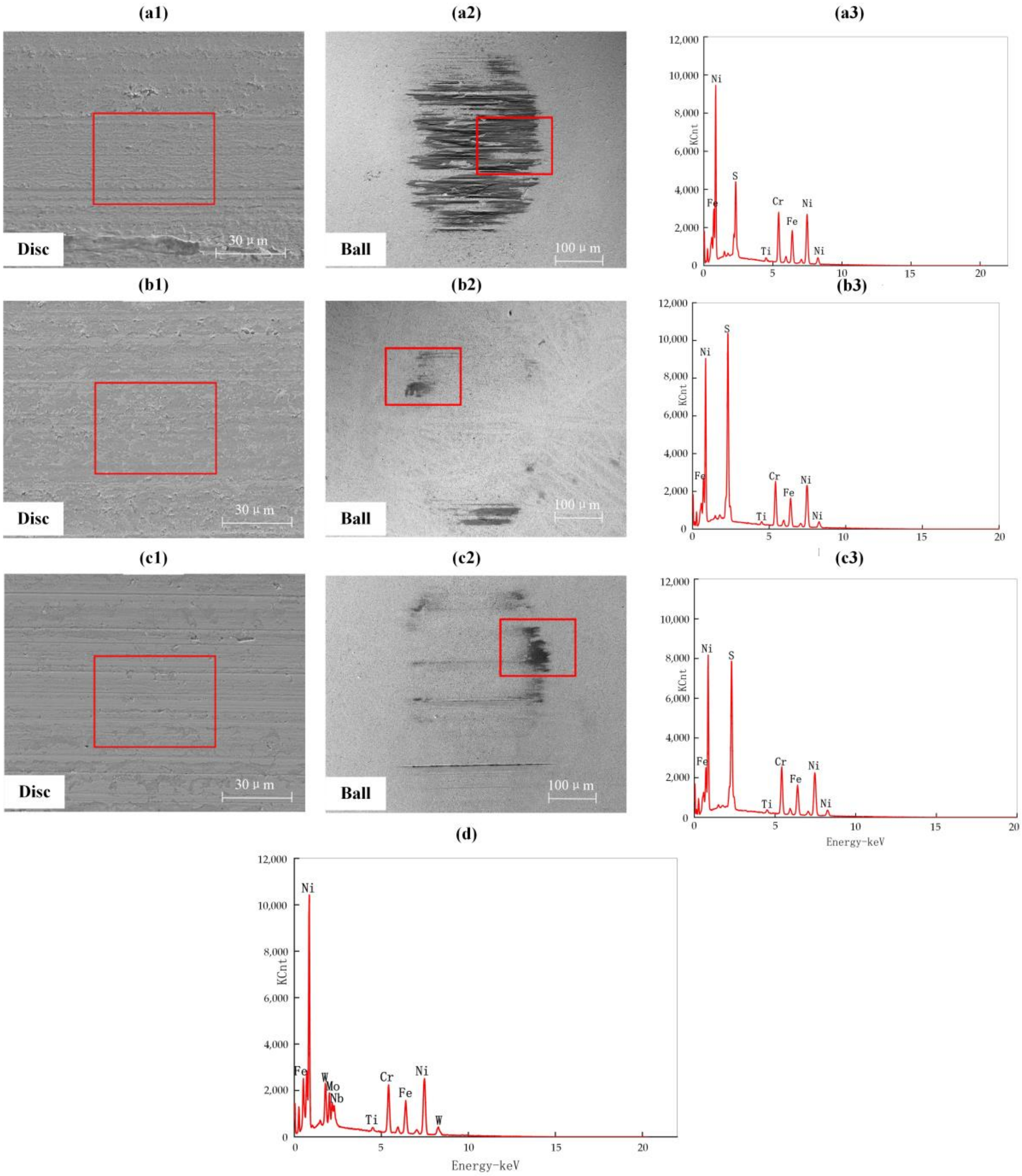

Figure 5. SEM morphology of the tracks on Inconel 718 and tungsten carbide ball lubricated under 150 °C lubricated by (**a1**,**a2**) sulfurized olefin, (**b1**,**b2**) sulfurized fatty acid ester, and (**c1**,**c2**) sulfurized lard; (**a3**,**b3**,**c3**) EDS spectrum of the red box in (**a1**,**b1**,**c1**); (**d**) EDS spectrum of the red box in (**a2**).

The wear volumes of sulfurized fatty acid ester and sulfurized lard on nickel-based superalloy under different loads were further tested. The experimental load was set to 300 N, 500 N, 700 N, and 900 N, and the results are shown in Figure 6b. When sulfurized fatty acid ester lubricated the nickel-based superalloy, the wear volume increased steadily with the increase in load. As for sulfurized lard, the wear volume had a larger increase compared to sulfurized fatty acid when the load increased to 700 N. The big wear volume was consistent with the large friction coefficient, indicating unstable lubricant condition.

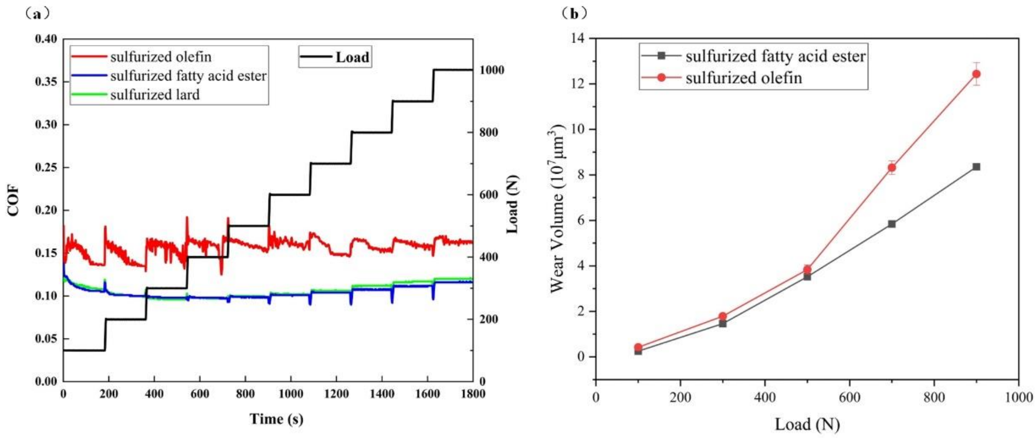

Figure 6. (**a**) Friction coefficient vs. time during a load slope test from 100 N to 1000 N and (**b**) wear volume under different loads (f = 20 Hz, L = 2 mm, t = 25 °C).

3.4. Exploration of the Adsorption Characteristics

XPS is a practical method to clarify the chemical states of elements within the adsorption film on the surface of tribopair. To further explain the lubrication mechanism of sulfur additives for nickel-based superalloy, the worn surfaces were tested by XPS. Figures 7 and 8 show the spectra of the several elements lubricated by sulfurized fatty acid ester and sulfurized olefin. It can be observed that the peak shapes and binding energies of the corresponding elements were similar. Therefore, the tribochemical reaction processes were the same when sulfurized fatty acid ester and sulfurized lard were used as lubricants for the nickel-based superalloy. Figure 7a shows typical XPS survey scans inside and outside the wear track over a binding energy at the range of 0–1400 eV with the lubrication of sulfurized fatty acid ester. The values were shifted 400,000 upward from the second line to show a clear contrast. The peak intensities of Ni2p and Fe2p inside the wear track are lower than those outside the wear track. Furthermore, a sulfur peak at 168 eV appeared at the position inside the wear track, while no sulfur was detected outside the track. This demonstrates that sulfide compounds remained after the frictional test when lubricated by sulfurized fatty acid ester. Ni2p inside the wear track was apparently lower than the substrate, which further demonstrates that some film existed on the wear track. To further investigate the way in which the sulfide compounds acted with nickel-based superalloy, detailed high-resolution XPS scans of Ni2p, Fe2p, S2p, and O1s were recorded, and the results are shown in Figure 7b–e. The peak at 852.8 eV is Ni–S, and the peak at 855.2 eV is Ni–SO_4 (Figure 7b). The peaks at 710.11 eV and 723.4 eV correspond to Fe–S and $FeSO_4$, respectively (Figure 7c) [11]. The S2p spectrum is shown in Figure 7d. The peak at 161 eV–162 eV corresponds to Fe–S, the peak at 162.8 eV corresponds to Ni–S, and the peak at 169.7 eV is the metal sulfate [21]. The O1s spectrum is shown in Figure 7e. The peak at 531.7 eV corresponds to S–O in –SO_4, and the peak at 530.2 is metallic oxide. Combining the Ni2p, S2p, and O1s data, it can be inferred that $NiSO_4$ may have existed on the surface of the wear track. Compared with the S2p spectra on the surface lubricated by sulfurized fatty acid ester, the peak intensities of Ni2p and Fe2p inside the wear track were higher when lubricated by sulfurized olefin (Figure 8a). Moreover, the peak of metal sulfates was much lower than that lubricated by sulfurized fatty acid ester (Figure 8c). It can be speculated that the tribochemical reaction film on Inconel 718 surface lubricated by sulfurized olefin was thinner than that lubricated by sulfurized fatty acid ester, leading to a higher COF and bigger wear volume.

According to the XPS results, the lubricating mechanism of the sulfur additives for Inconel 718–tungsten carbide contact can be summarized. In the frictional process, the molecules adsorb on the metal surface to form a physical adsorption protective film. The active element S reacts with the metal matrix Inconel 718. A tribochemical protective film composed of high-toughness inorganic salts such as nickel sulfide and nickel sulfate is formed, playing a role in lubrication. The physical adsorptive film and the tribochemical film together enhance the friction-reducing and anti-wear performances of the lubricants.

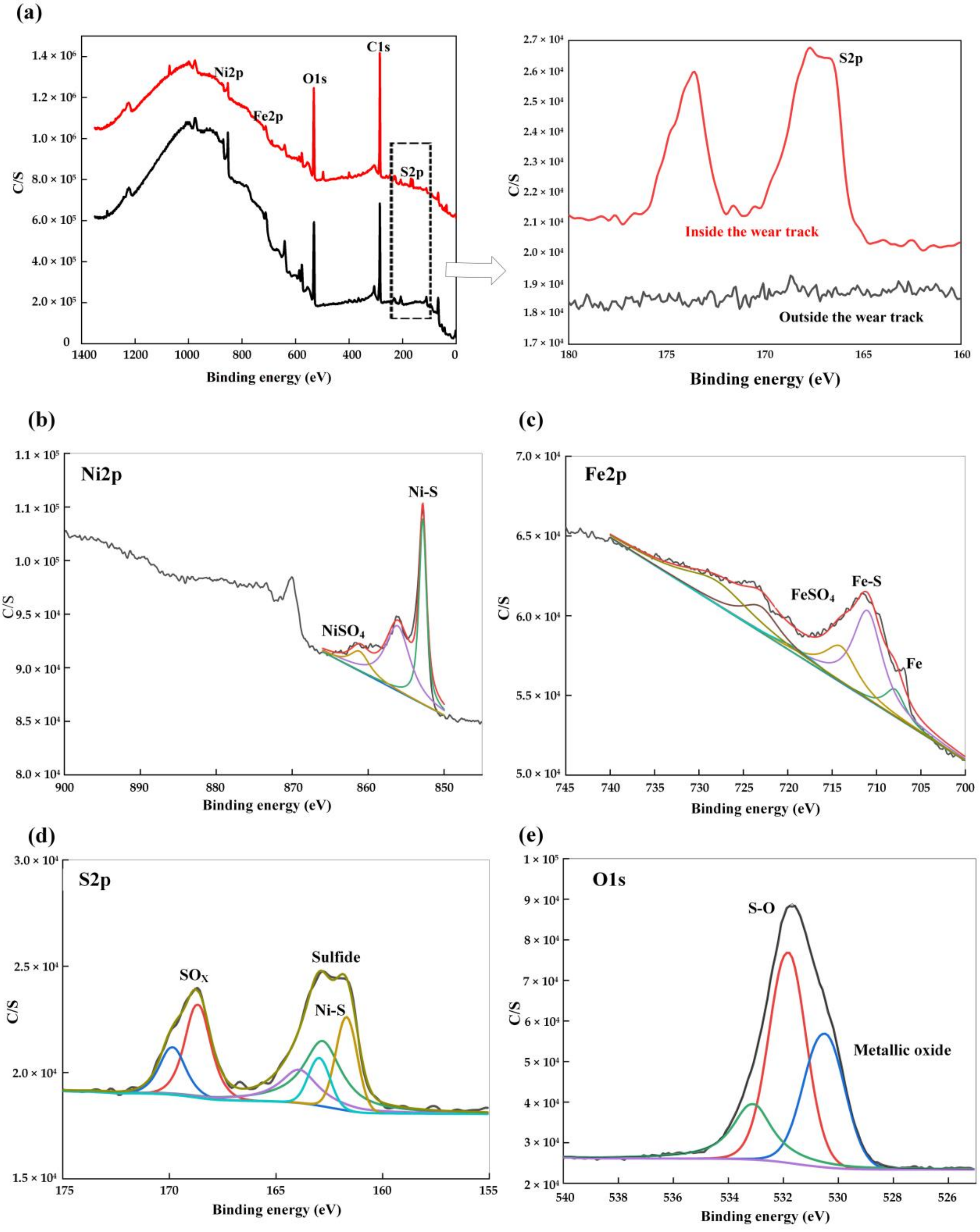

Figure 7. (**a**) XPS survey scans of Inconel 718 surface after tribological test: (**b**) Ni2p, (**c**) Fe2p, (**d**) S2p, and (**e**) O1s inside the wear track lubricated by sulfurized fatty acid ester.

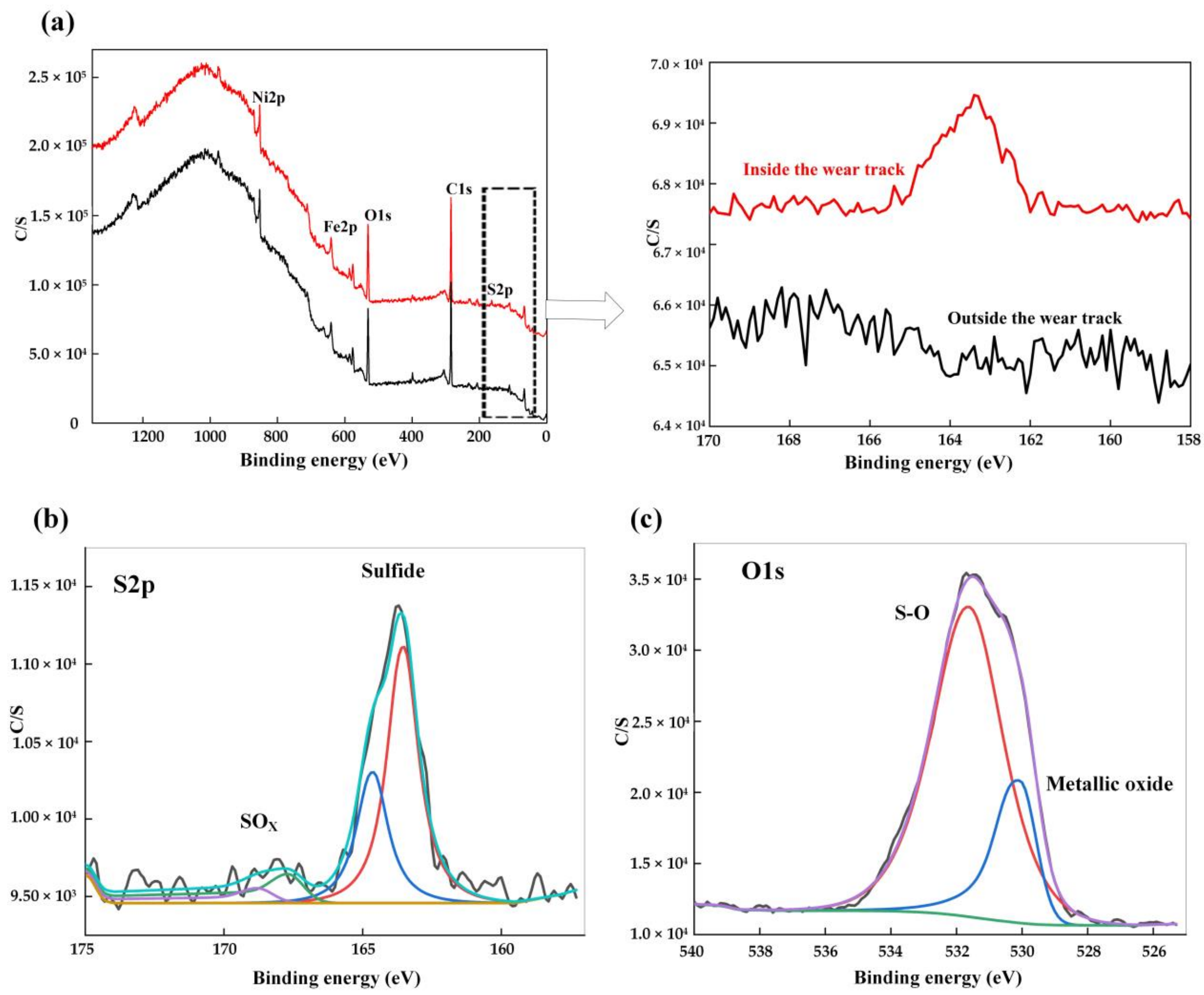

Figure 8. (**a**) XPS survey scans of Inconel 718 surface after tribological test, (**b**) S2p, and (**c**) O1s inside the wear track lubricated by sulfurized olefin.

3.5. The Improvement of the Cutting Fluid

The experimental results show that, for the nickel-based superalloy–tungsten carbide friction pair, sulfurized fatty acid ester had the best lubricating and anti-wear performance among the three kinds of extreme-pressure agents. The effect of sulfurized fatty acid ester on the lubricating performance of some kind of cutting fluid without sulfur-containing additives was tested. The cutting fluid was diluted to 5 wt.% with different content of sulfur additives. The frictional experiments were completed, and the friction coefficients are shown in Figure 9. The results show that the friction coefficient dropped to 0.133 with the concentration of sulfurized fatty acid ester at 1 wt.%. When the concentration of sulfurized fatty acid ester increased to 2%, the friction coefficient further dropped to 0.127. The ester concentration was further increased to 3%, and the friction coefficient did not continue to decrease and remained stable at 0.127. It can be seen that 2 wt.% sulfurized fatty acid ester could successfully decrease the friction coefficient of the cutting fluid.

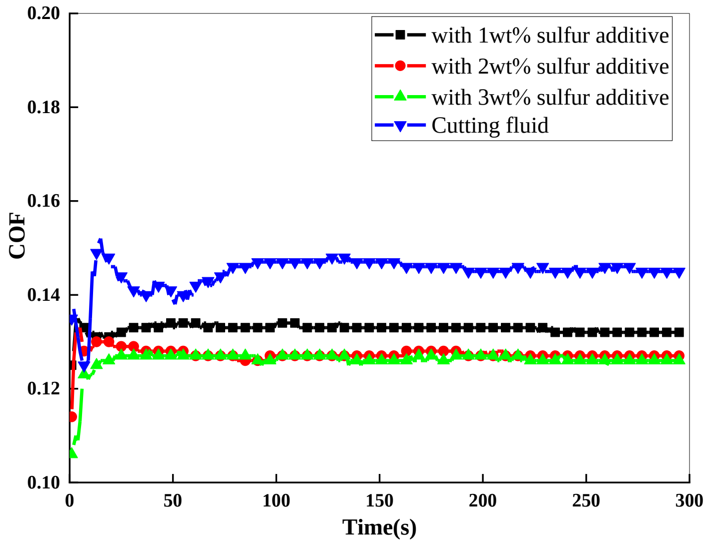

Figure 9. The COF of some cutting fluids with different contents of sulfurized fatty acid ester (F = 100 N, f = 20 Hz, L = 2 mm, t = 25 °C).

4. Conclusions

In the present work, the tribological performance of three kinds of sulfur additives for the Inconel 718–tungsten carbide friction pair was investigated. The friction experiment results showed that sulfurized fatty acid ester possessed excellent antifriction (COF 0.1) and anti-wear performance (wear volume 90% smaller than that lubricated by PAO 40), particularly at the high temperature of 150 °C and at heavy load. The lubrication mechanism of the sulfur additives for the Inconel 718–tungsten carbide friction pair was investigated using XPS. The physical adsorptive film and the tribochemical film together enhanced the friction-reducing and anti-wear performance of the lubricants. This effective lubricant for Inconel 718 can be applied to the machining of nickel-based superalloy.

Author Contributions: Conceptualization, Y.Y. and C.Z.; methodology, Y.Y. and Y.D.; validation, F.L.; formal analysis, F.L.; investigation, H.L., S.G. and Y.Y.; resources, C.Z., Y.Y. and G.Z.; data curation, H.L. and S.G.; writing—original draft preparation, H.L.; writing—review and editing, Y.Y.; visualization, Y.Y.; supervision, F.L.; project administration, F.L. and D.Z.; funding acquisition, C.Z., Y.Y. and G.Z. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by National Key Research and Development Program of China grant number 2018YFB2002204 and National Natural Science Foundation of China grant number 52005010 and U1837602.

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

Conflicts of Interest: The authors declare no conflict of interest.

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