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Influence of Additive Chemistry on the Tribological Behavior of Steel/Copper Friction Pairs

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Abstract: Tribological properties of five anti-wear additives for a steel-copper contact were investigated. It was found that the tribological performances are closely related to the molecular structure of additives. The protic ionic liquid anti-wear additive AW316 exhibits the best tribological performance with the lowest mean friction coefficient of 0.082, and the smallest wear volume, which is more than one order of magnitude smaller than base oil. Transmission electron microscope (TEM) and X-ray photoelectron spectroscopy (XPS) tests reveal that a 10–15 nm thickness uniform boundary lubrication film composed of oxides, phosphates, and cuprous oxide was formed on the copper disc, which was responsible for its outstanding tribological performances.

Keywords: anti-wear additives; ionic liquid; tribology; steel-copper

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1. Introduction

The application of steel and copper alloys as friction materials for sliding machines is very common in modern machinery. Typical applications are worm gears, bearings, spindle drives, hydraulic pumps, and various bushing and guiding devices [1]. These components are ordinarily lubricated with oil; therefore, the tribological performance is a concern for these friction pairs. It is generally accepted that copper is more susceptible to wear due to its low hardness and susceptibility to plastic deformation. In addition, the matching of copper with steel makes them more prone to wear and more challenging to be lubricated. Therefore, the appropriate design of lubricants, especially the selection of lubricating additives, is one of the most critical factors in controlling the tribological performance of copper-based materials [2–4]. Unlike its steel counterparts, whose tribological performance has been extensively studied and the tribological mechanism is well understood [5–7], less attention has been paid to the tribological properties of copper materials. Although several studies have been conducted recently to investigate the friction and wear performances of lubricants for steel/copper friction pairs [2–4,8,9], there still lacks basic knowledge about tribological performance as well as the mechanism of copper-based materials.

It is well recognized that anti-wear additives play a dominant role in sliding contact under a boundary lubrication regime. Traditional anti-wear additives were designed for steel–steel contact and their compatibility with steel–copper, which is not well understood. Some previous works related to copper have studied the influences of additives on the tribological performance of steel/copper contact. Robin Jisa et al. [1]. investigated the tribological properties of ester-based additives on various types of copper alloys and found a remarkable variation of tribological behavior with a clear relation to both the chemistry/composition of lubricant and the type of alloy. Recently, ionic liquids, as novel lubricant additives for steel/copper contact, have been extensively studied [10–15]. Cai [13] reported a multifunctional imidazolium ionic liquid for steel/Cu-Sn alloy and found that a strong interaction between benzotriazole and the surface of the Cu alloy was proposed to account for its outstanding anti-wear and anticorrosion properties. Li [14] studied the corrosion and lubrication properties of 2-mercaptobenzothiazole functionalized ionic liquids for steel/copper contact and found that the synergistic effect of adsorbed ionic liquids film and tribochemical products are responsible for their tribological performances. Our recent study investigated the compatibility between organic friction modifier and various oil-soluble ionic liquids for a steel–bronze contact under a boundary lubrication regime, finding that a protic ionic liquid works well with organic friction modifier in terms of both friction-reducing and anti-wear performances [15]. Very recently, tribological performances of novel nano-lubricant additives, such as graphene, and nanodiamonds for steel/copper pairs, were also studied [16]. Although these studies provide valuable insight into the tribological performance of steel–copper friction, the comparison of conventional additives and emerging ionic liquids in terms of their tribological performances on steel/copper was still merely studied and rarely reported.

In order to comparatively investigate the friction-reducing and anti-wear properties between traditional anti-wear additives and novel ionic liquids (ILs) under steel/copper friction pairs, five anti-wear additives with different chemical structures, such as zinc dialkyldithiophosphate (ZDDP), ashless dialkyldithiophophate, tricresyl phosphate, as well as two ILs, were selected. Their tribological properties were measured using an SRV-IV oscillating reciprocating friction and wear tester, and the tribological mechanism was discussed based on the scanning electron microscopy (SEM), energy dispersive spectrometer (EDS), and X-ray photoelectron spectroscopy (XPS) analysis on worn surfaces. These results can provide a guide for selecting suitable friction-reducing and anti-wear additives for steel/copper friction pairs when formulating high-performance lubricants.

2. Experiment Section

2.1. Materials

A synthetic base oil, poly alpha olefin (PAO4, with a viscosity of around 4 mm^2/s at 100 °C) was purchased from Exxon Mobil Corp. (Irving, TX, USA). Zinc Dioctyl Primary Alkyl Dithiophosphate (ZDDP, trade name RF2203) (Purity > 98.0%) was provided by Xinxiang Richful Lube Additive Co., Ltd., Xinxiang, China. Dialkyl dithiophosphate ester (the trade name is Irgalube 353, purity > 93.0%) was provided by BASF Corporation (Ludwigshafen, Germany). Tritolyl phosphate (TCP, purity > 98.0%) was provided by Zibo Huihua Petroleum Additive Co., Ltd. (Zibo, China). Phosphate amine (a protic ionic liquid, trade name AW316, purity > 98.0%) was provided by Qingdao Lubemater lubrication materials technology Co., Ltd. (Qingdao, China). An aprotic ionic liquid trihexyltetradecylphosphonium bis (2,4,4-trimethylpentyl) phosphinate (abbreviated as [P_{8888}][DEHP]) was synthesized in our lab according to the procedures reported previously [17]. All these additives were added to the base oil with the same concentration of 0.8 wt.%. Schematic chemical structures of these anti-wear additives are depicted in Figure 1.

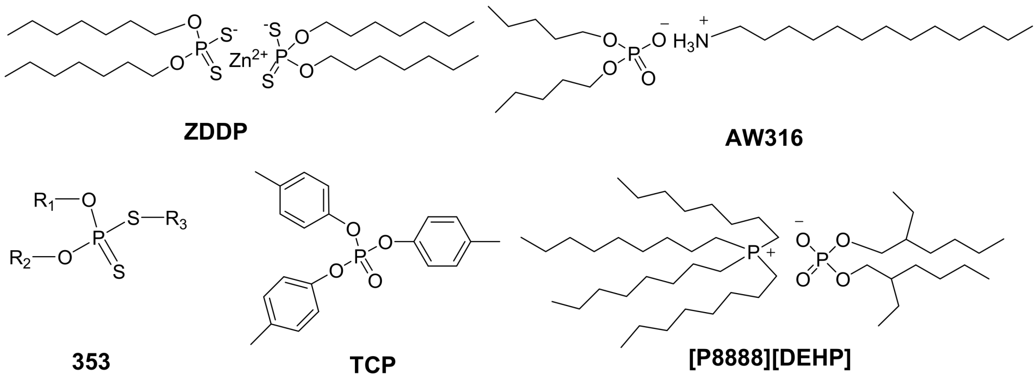

Figure 1. Schematic chemical structures of anti-wear additives.

2.2. Tribological Testing

Boundary lubrication tests were conducted on an SRV-IV oscillating reciprocating friction and wear tester, and a schematic diagram of ball-on-disc sliding is shown in Figure 2. The schematic diagram is of an AISI 52100 bearing steel ball (10 mm diameter) sliding against a bronze plate (25.37 × 25.37 × 6.35 mm). The chemical composition of the test materials is summarized in Table 1. Tribological tests were carried out under a 50 N load (the maximum Hertzian pressure is approximately 959.9 MPa) and an oscillation frequency of 25 Hz, with a stroke of 1 mm, at 100 °C for 1 h. After tests, contact surfaces were cleaned with ethanol and petroleum ether. Each test was repeated at least three times to guarantee accuracy.

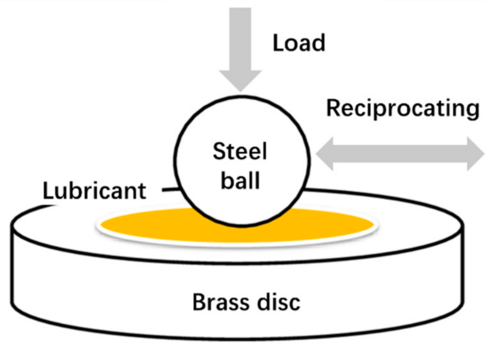

Figure 2. Schematic diagram of ball-on-disc reciprocating sliding.

Table 1. Chemical composition of sliding materials.

Materials	Composition
AISI 52100	C, 1.04%; Mn, 0.11%; Si, 0.25%; Cr, 1.58%; Fe-balance
H62 Brass	Cu, 60.5–63.5%; Fe, 0.15%; Pb, 0.8%; Sb, 0.005%, Bi, 0.002%, P, 0.01%, Zn-balance

2.3. Surface Characterizations

Wear volume of the lower disc was measured using a MicroXAM-800 3D noncontact surface mapping profiler. A Hitachi S-3500N scanning electron microscope (SEM), coupled with energy-dispersive X-ray spectroscopy, was used to examine the morphology and chemical composition of the worn brass surface. X-ray photoelectron spectroscope (XPS) Al Kα X-rays, focused on a 250 μm spot, were used to excite photoelectrons that were measured with a hemispherical electron energy analyzer and 128 channel detectors. Cross-sectional lamellae of the tribofilms were obtained by using a focused ion beam (FIB) in a DualBeam SEM/FIB instrument for further HR-TEM investigation. In order to protect the tribofilm, the area was coated with Au and Pt cap layer before cutting cross-sectional lamellae.

3. Results and Discussion

3.1. Friction-Reducing Behaviors

The mean coefficient of friction (COF, the average value of COF over the test duration) of each test is summarized in Table 2. The variation of COFs with test duration is also displayed in Figure 3. It is observed that the COF of base oil PAO4 experienced a drastic fluctuation during the test and gave the highest mean COF of 0.349. The introduction of anti-wear additives greatly alleviated severe friction conditions on the metal interfaces. Among these additives, AW316, a protic anti-wear additive, achieved the lowest mean COF of 0.082, which is an almost 76% reduction in the COF compared with base oil. Moreover, the COF of ZDDP was 0.093, which indicates that ZDDP works very well on the steel–copper friction pair. Furthermore, 353, an ashless anti-wear additive with a typical DDP (dialkyldithiophosphate) group, also showed a relatively low COF of 0.118. All these three additives share a common characteristic: that they are chemically active additives and have a relatively high total acid number (TAN). TAN of AW316, ZDDP, and 353 were 260 mgKOH/g, 140 mgKOH/g and 136 mgKOH/g, respectively. The high acid value indicates these additives are more prone to absorb onto the metal surfaces and require less active energy to initiate tribochemical reactions; in other words, they are more likely to take part in the tribochemical reactions during sliding. By contrast, the mean COF of TCP and [P_{8888}][DEHP] were 0.170 and 0.199, respectively, which are much higher than their counterparts. TANs of TCP and [P_{8888}][DEHP] are around 0.1 mgKOH/g, which is much lower than the above active additives' means, suggesting that they are chemically inert. Overall, the friction-reducing order of the tested anti-wear additives are AW316 > ZDDP > 353 > TCP > [P_{8888}][DEHP].

Table 2. Mean coefficient of friction of different lubricants.

Item Number	Composition	Mean Coefficient of Friction
a	PAO4 base oil	0.349
b	PAO4 + 0.8% ZDDP	0.093
c	PAO4 + 0.8% AW316	0.082
d	PAO4 + 0.8% [P_{8888}][DEHP]	0.199
e	PAO4 + 0.8% 353	0.118
f	PAO4 + 0.8% TCP	0.170

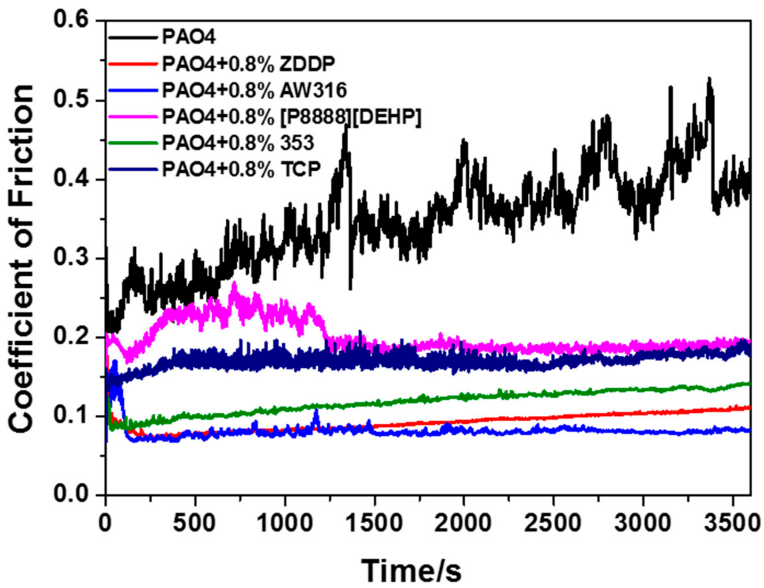

Figure 3. Variation of coefficient of friction with test duration.

3.2. Anti-Wear Performances

Figure 4 shows the wear volume of the copper plate after the tribological tests of different lubricants. PAO4 yields the highest wear volume of 72.9 $\times$ 10^{-3} mm^3. The addition of anti-wear additives greatly strengthens the wear protection performance of the base oil. It is worth mentioning, however, that AW316 produces the lowest wear volume of 6.15 $\times$ 10^{-3} mm^3, which is more than an order of magnitude smaller than base oil. Other anti-wear additives also reduce the wear volume to a great level. The wear protection properties of the selected additive follow the order of AW316 > ZDDP > 353 > [P_{8888}][DEHP] > TCP. The overall trend of anti-wear behavior is similar to their friction-reducing properties.

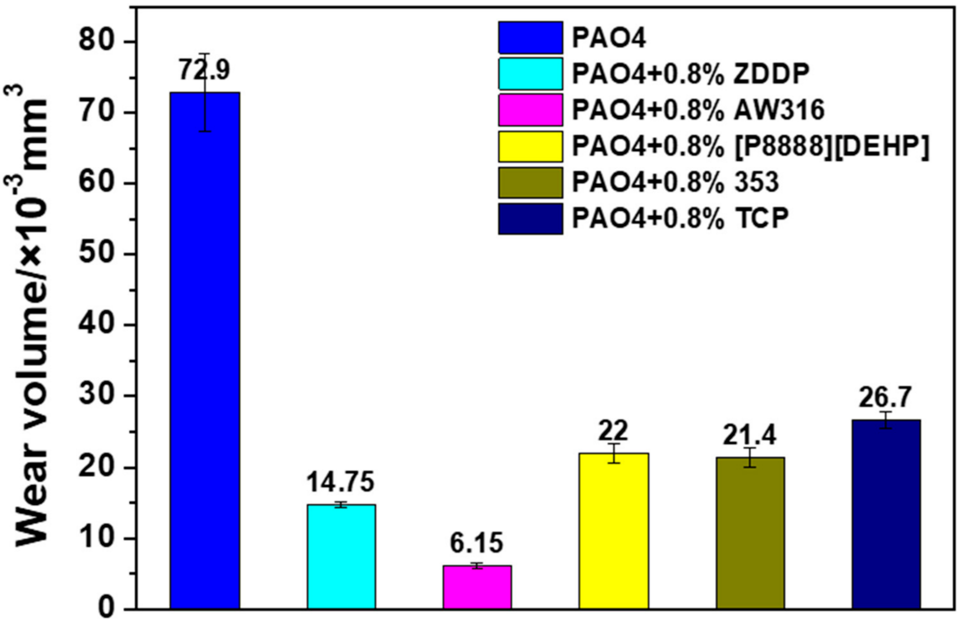

Figure 4. Wear volumes of copper plates after tribological tests.

3.3. Worn Surfaces Analysis

Worn surfaces of copper plates were ultrasonically cleaned with petroleum ether after the tribological tests. Both the steel ball and copper plate were examined with SEM. Figure 5 shows the top-view images and 3D morphologies of the worn surfaces. As observed, PAO4 (a_1, a_2, a_3) showed the largest and deepest wear scar among these lubricants. Deep and large grooves and large bumps were detected on the wear scar of PAO4, indicating both abrasive wear and adhesive wear occurred on the rubbing surfaces. The addition of anti-wear additives greatly alleviated the wear performance of base oil. As clearly shown, among all the lubricants, the wear scar of PAO4 with AW316 was the narrowest and shallowest, followed by PAO4 with ZDDP. Overall, the results displayed by the SEM analysis of the worn surface correspond well to the worn volume of the lubricants.

Element compositions on the copper-worn surfaces were also examined using EDS and are listed in Table 3. It can be seen in the table, that both phosphorus and sulfur were detected on the worn surface of ZDDP. Moreover, ZDDP also contains the highest zinc content among these additives, which is probably due to the tribochemical reaction between ZDDP and the copper surface. All the signature elements of AW316, such as oxygen, phosphorus, and nitrogen, were found on the worn surfaces, indicating the worn surface was covered by a boundary lubrication film. Due to the high reactivity between sulfur and copper, 353 showed a high sulfur content of 2.12 wt.%, indicating sulfur plays a critical role in the formation of boundary film. For TCP, only a few phosphorus (around 0.22 wt.%) were detected on the worn surfaces, suggesting that a limited or inadequate tribochemical reaction took place on the interfaces, which explain its poor tribological performances.

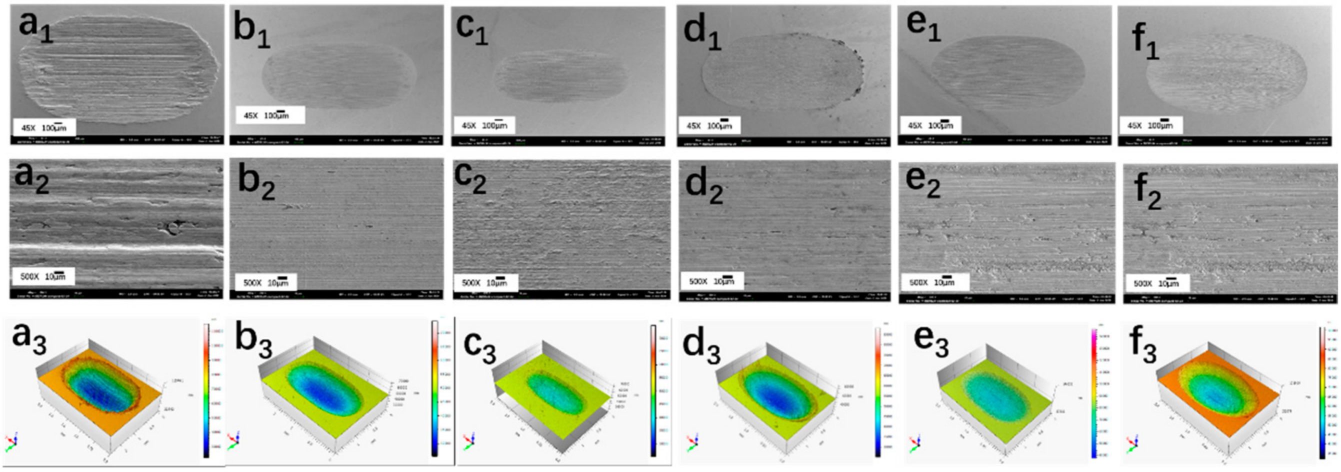

Figure 5. SEM images and 3D morphologies of copper-worn surfaces. (PAO4: ($\mathbf{a_1}$–$\mathbf{a_3}$); PAO4 + 0.8% ZDDP: ($\mathbf{b_1}$–$\mathbf{b_3}$); PAO4 + 0.8% AW316: ($\mathbf{c_1}$–$\mathbf{c_3}$); PAO4 + 0.8% [P_{8888}][DEHP]: ($\mathbf{d_1}$–$\mathbf{d_3}$); PAO4 + 0.8% 353: ($\mathbf{e_1}$–$\mathbf{e_3}$); PAO4 + 0.8% TCP: ($\mathbf{f_1}$–$\mathbf{f_3}$).

Table 3. Elements composition of worn surfaces of copper plates obtained from EDS analysis.

Elements (wt.%)	PAO4	PAO4 + 0.8%ZDDP	PAO4 + 0.8%AW316	PAO4 + 0.8%[P_{8888}][DEHP]	PAO4 + 0.8%353	PAO4 + 0.8%TCP
O	4.23	3.69	4.51	5.04	5.69	3.67
Fe	1.00	1.63	1.40	1.97	1.86	0.78
Cu	54.80	54.68	63.76	60.78	53.77	67.71
P	-	0.76	0.75	0.99	0.56	0.22
S	-	1.63	-	-	2.12	-
N	-	-	2.52	-	-	-
Zn	38.86	35.76	25.56	28.59	34.60	24.61
Pb	1.11	1.85	1.50	2.63	1.40	3.01

3.4. FIB-TEM Analysis

To further understand the tribological mechanism of AW316, the FIB-TEM technique was applied to analyze the tribofilm generated with the lubrication of PAO4 + AW316. It is identified that a white tribofilm with a thickness of 10–15 nm is formed on the worn surface, as shown in Figure 6. Underneath the tribofilm is a subsurface plastic deformation zone with a thickness of about 250 nm. It is observed that the tribofilm is even and flat, which is consistent with the SEM results (c2 in Figure 5). It was this tribofilm that protected the rubbing pair by separating the direct contact of the friction pairs. As demonstrated in Table 3, phosphorus is one of the most significant elements of the boundary lubrication film. Therefore, the variation of the phosphorus composition with the depth of film could provide possible evidence of the thickness of the boundary film. Figure 6f depicts the phosphorus content, along with the surface depth. It is clearly shown that the phosphorus content decreases with the depth of the surface; when the depth is higher than about 12 nm, the phosphorus becomes almost 0, indicating that the boundary lubrication film has reached its end, and beyond that is the substrate material, which further demonstrates the film thickness of the boundary lubrication film [18].

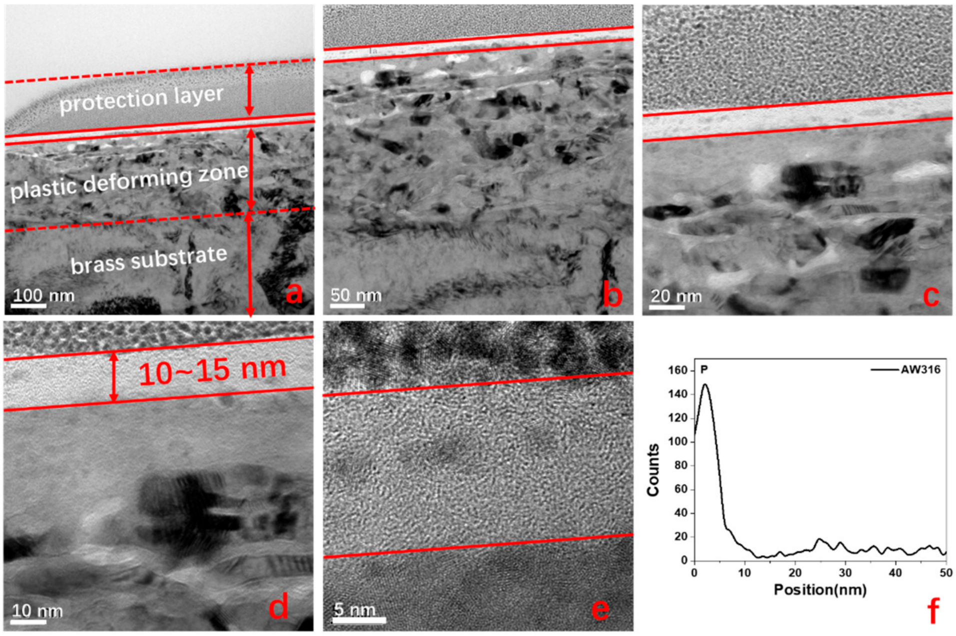

Figure 6. (**a**–**e**) Overview of TEM graph of FIB-cut cross-section of the tribofilm formed on the brass disc lubricated by PAO4 + AW316, (**f**) EDS line (phosphorus) analysis along the brass disc.

3.5. XPS Analysis

To get further insight into the chemical composition of tribofilm on copper disc, XPS spectra of the key elements of PAO + AW316 were analyzed and the results are shown in Figure 7. The Cu_{2p} peaks at 932.6 can be assigned to Cu_2O for PAO + AW316 [19,20]. A typical peak near 132.8 eV, in the high-resolution spectra of P_{2p} of PAO + AW316, can be assigned to the P–C bond [21–23]. N_{1s} of PAO + AW316 give a strong peak, appearing at 399.8 eV and 398.5 eV, which is most possibly identified as Cu–N [24,25]. The O1s peak of PAO4 + AW316, at approximately 531.6 eV, can be assigned to C–O bonding. The above XPS results suggest that tribochemical reactions occurred between anti-wear additives and copper during the tests. These processes generate a boundary lubrication film mainly composed of oxides, phosphates, and cuprous oxide, which can effectively protect against the direct contact of the rubbing surface and reduce the friction coefficient of the tribology system, resulting in better tribological properties.

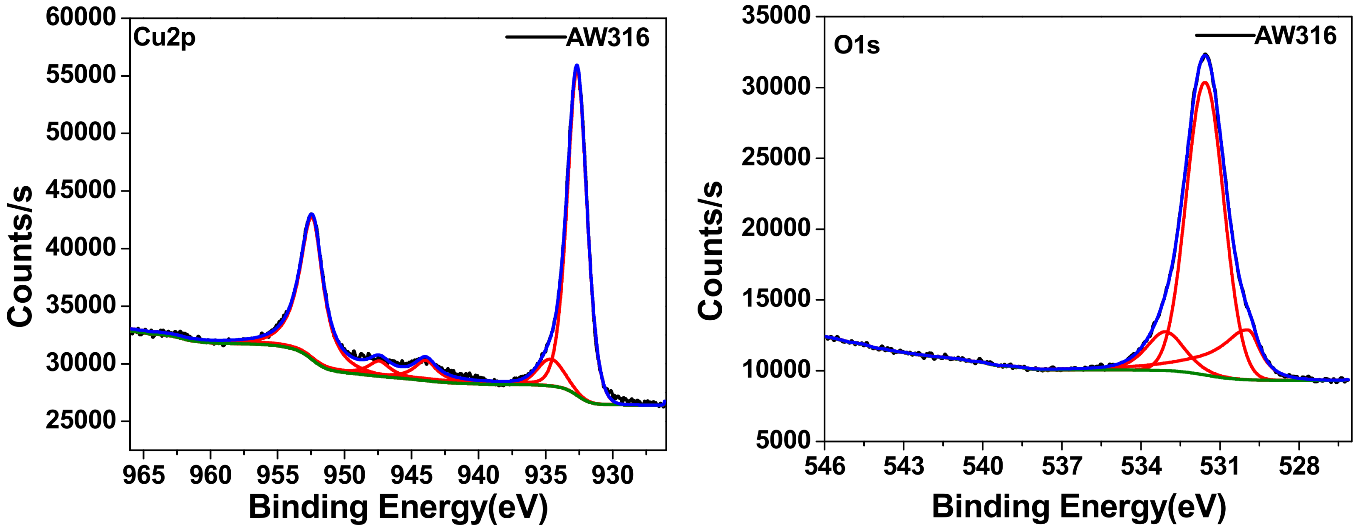

Figure 7. *Cont.*

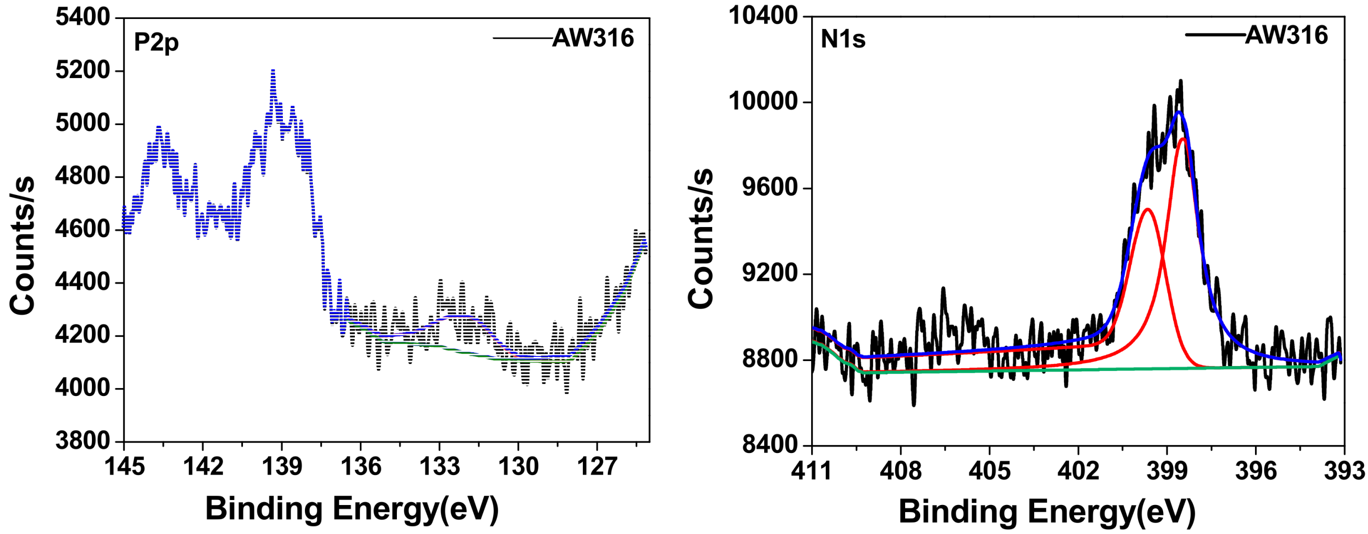

Figure 7. XPS spectra of Cu2p, O1s, P2p, and S2p on the brass disk lubricated with PAO4 + AW316.

4. Conclusions

Five typical anti-wear additives were studied as lubricant additives for steel–copper friction pairs. The tribological performances were investigated and their tribological mechanisms were discussed. The main findings are summarized below:

(1) Tribological behavior was highly determined by the chemical structure of anti-wear additives. Higher chemical activity (high polarity and high acid value) is a favorite to achieve better tribological performance.
(2) A protic ionic liquid (amine phosphate, AW316) was found to possess the best anti-wear additive for steel–copper sliding, which achieves the lowest mean friction coefficient (0.083) and smallest wear volume (6.15×10^{-3} mm^3).
(3) The superior tribological performance of AW316 can be attributed to the rapid formation of an even boundary lubrication film with a thickness of 10–15 nm and is composed of oxides, phosphates, and cuprous oxide.

In general, a careful screen of additives is essential for steel–copper friction pairs since the tribological are highly influenced by additive type and chemistry. This study may provide some guidance in the selection of suitable anti-wear additives when developing lubricants for steel–copper friction pairs.

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