Conductive capacity and tribological properties of several carbon materials in conductive greases

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Abstract

Purpose – The purpose of this paper is to synthesize a new kind of conductive grease which possesses a prominent conductive capacity and good tribological properties.

Design/methodology/approach – A two-step method was used to prepare complex lithium-based grease. Ketjen black (KB), acetylene black (AB) and carbon black (CB) were characterized by transmission electron microscope and used as lubricant additives to prepare conductive greases. Conductive capacity was evaluated by a conductivity meter, a surface volume resistivity meter and a circuit resistance meter. Tribological properties were investigated by a reciprocating friction and wear tester (MFT-R4000). The worn surfaces were analyzed by a scanning electron microscope, Raman spectroscopy, energy-dispersive X-ray spectroscopy and X-ray photoelectron spectroscope.

Findings – The conductive grease prepared with KB has a prominent conductive capacity at room temperature, 100°C and 150°C. Further, this conductive grease also possesses better tribological properties than AB and KB greases. When the concentration of KB is 1.8 Wt.%, the coefficient of friction and wear width reduced by 11 and 14 per cent, respectively.

Originality/value – This work is a new application of nanometer KB as a lubricant additive in grease, which provides a direction for preparing conductive grease. The conductivity and tribology experiments have been carried out though the variation of experiment conductions.

Keywords Tribology, Conductivity, Friction and wear, Ketjen black

Paper type Research paper

1. Introduction

Machinery and automotive components need lubricating greases to protect friction parts from friction and wear losses. However, when the conductive parts of power equipment need to be lubricated, such as switches, parallel groove clamps and so on, the conductive capacity of lubricating greases should be given more attention. Lubricating greases possess high conductivity, not only reducing friction and wear losses but also saving energy by ameliorating the quality of electrical connection, enhancing the lubrication function and extending the working life (Fan *et al.*, 2014; Wang *et al.*, 2014, 2012; Zhao, 2010; Wu *et al.*, 2014). Thus, it is significantly important to develop greases which have high conductive capacity and excellent tribological performance. The conventional conductive grease is prepared by adding additives which possess good electrical conductivity such as gold, copper and silver powders including ionic liquids (ILs) into base grease (Ge *et al.*, 2015a, 2015b). However, these conductive additives, such as gold and silver powder are too expensive for widespread application. Although copper powder has the advantage of low price, it is easily oxidized. ILs can corrode electrical connection. Scholars are looking for new grease conductive additives.

Carbon is a multifunctional material, and there are many allotropes of carbon. They are widely used in various fields such as medication, power industry, lubrication and material fields (Barnes *et al.*, 2009; Sohi *et al.*, 2011; Kang *et al.*, 2010; Ler *et al.*, 2007; Fawcett *et al.*, 2010; Lin *et al.*, 2011). Ketjen black (KB), prepared through a special process, is a type of a nanometer carbon material. It is used frequently as a conductive agent in batteries and electrochemical capacitors. It has a unique chain structure and has an extremely high specific surface area (SSA) (Chen *et al.*, 2013). Wang *et al.* (2010) found that KB was more beneficial to enlarge the electrochemical surface area and direct cell performance of the electrode catalyst layer. Chen *et al.* (2015) researched that ZnO/ketjen nanocomposite exhibited superior electrochemical performances and enhanced the conductivity of active materials in lithium ion batteries. Bakenov and Taniguchi (2010) discovered that the composite $LiMnPO_4$/C electrode prepared with KB which possesses the largest SSA, showed the largest discharge capacity. In previous studies, it was indicated that carbon with large SSA could easily form electrical contact points and provide stable and quick routes for current. Therefore, KB with large SSA and high conductivity could be a superior option when choosing a better conductive material to

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prepare conductive grease. However, little research about KB on lubrication has been done.

In this paper, the conductive complex lithium-based grease was synthesized using oil-soluble polyether (OSP320) as the base oil; lithium hydroxide, sebacic acid and 12-hydroxystearic acid as the thickening agent; nanometer particles: KB, acetylene black (AB) and carbon black (CB) as additives. Their physicochemical and tribological properties were investigated in detail, and the worn surfaces were examined by scanning electron microscope (SEM), Raman spectroscopy, energy-dispersive X-ray spectroscopy (EDX) and X-ray photoelectron spectroscope (XPS) to analyze the friction and wear mechanism.

2. Experimental details

2.1 Materials

The base oil (OSP320) used in this paper was obtained from Nanjing Golden Chemical Co., Ltd. Lithium hydroxide, sebacic acid and 12-hydroxystearic acid were purchased from Sinopharm Chemical Reagent Co., Ltd. All the chemical reagents were analytically pure and were used without further purification. Figure 1 are the transmission electron microscope (TEM) images of KB, AB and CB particles. The typical properties of OPS320 are shown in Table I, and the typical properties of KB (Lion Ltd.), AB (Tianyishiji Chemical) and CB (Kejun Chemical) are shown in Table II.

2.2 Preparation and characterization

2.2.1 Preparation of conductive complex lithium-based grease

The complex lithium-based grease was prepared by a two-step method. First, pure OSP320 whose content is 40 Wt.% and 12-hydroxystearic acid were added into the reaction vessel and stirred. The mixture was heated up to 80°C to dissolve 12-hydroxystearic acid. Second, the reaction temperature was raised to 100°C, and equivalent aqueous solution of lithium hydroxide monohydrate was added into the vessel, then the temperature was maintained at 100°C for 1 h. Third, the rest of the base oil and equivalent aqueous solution of lithium hydroxide monohydrate was slowly added into the vessel. After that, the reaction temperature was raised to 210°C and maintained for 10 min. Fourth, when the mixture was cooled to 80°C, a certain amount of the conductive additive was slowly added into the vessel and the mixture was stirring for another 30 min. Finally, the mixture was cooled to room temperature (RT) and the conductive complex lithium-based grease was obtained after three steps of fine grinding/homogenization with a three-roller mill.

2.2.2 Characteristics of conductive greases

The conductivities of the prepared greases were measured using a DDSJ-308A conductivity meter. Because of the measuring range of the meter, the order of magnitude of conductivity below 10^{-8} cannot be tested. A GEST-121 surface volume resistance tester [Figure 2(a)] was used to test the volume resistivity of the grease. For the reciprocal relationship between conductivity and volume resistivity, the conductivity that cannot be tested by the DDSJ-308A conductivity meter can be calculated. The penetration, the dropping point and the copper-strip corrosion of the prepared conductive greases were characterized under national standards GB/T 269, GB/T 3498 and GB/T 7326-87. The initial and final contact resistances were measured at different torques by HLY-200A circuit resistance tester

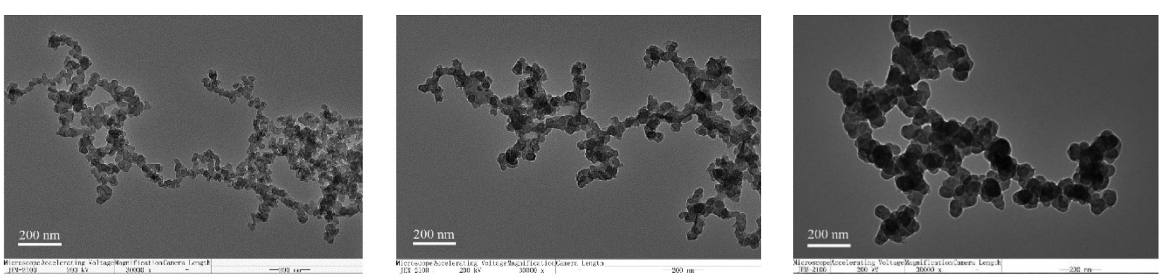
Table I Typical properties of the oil soluble polyether (OSP320)

Item	OSP320	Standard
Kinematic viscosity (cSt) 40°C	320	ASTM D445
Kinematic viscosity (cSt) 100°C	36	ASTM D445
Viscosity index	163	ASTM D5293
Pour point (°C)	−37	ASTM D97
Flash point (°C)	230	ASTM D92
Fire point (°C)	260	ASTM D92
Aniline point (°C)	<−30	ASTM D611-01

Table II Typical properties of nanometer particles: KB, AB and CB

Additives	KB	AB	CB
Grain size	35 nm	45 nm	50 nm
Electric resistivity (s/cm)	5	0.007	0.004
Purity (Wt.%)	>98	>98	>98
SSA ($m^{2/g}$)	>1,300	>800	1,031
Moisture content (Wt.%)	0.5	1	1
Ash content	<0.5	<0.5	<0.5
Pore volume ($cm^{3/g}$)	>5.1	>3.8	>4.0

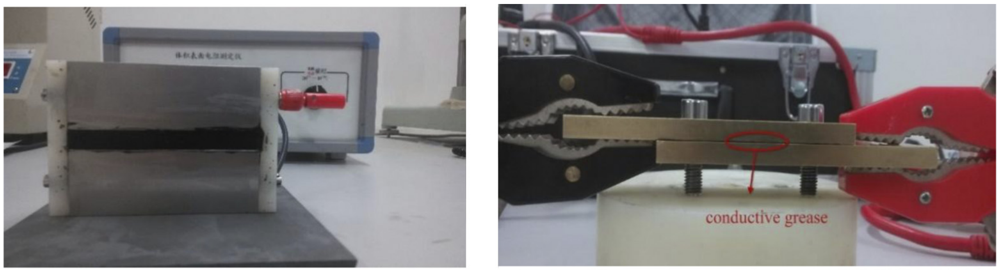
Figure 1 TEM images of nanoparticles



(a) (b) (c)

Notes: (a) KB; (b) AB; (c) CB

Figure 2 (a) GEST-121 surface volume resistivity tester and (b) HLY-200A circuit resistance tester



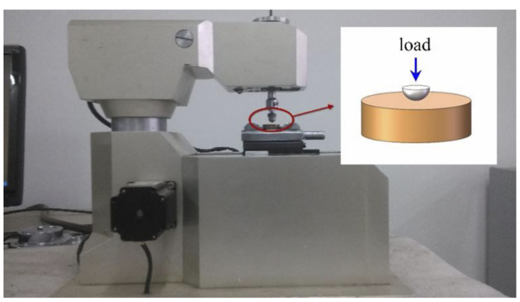
(a) (b)

[Figure 2(b)], and the testing current and time were 100 A and 10 s, respectively.

2.3 Friction and wear tests

The ball-on-discs (MFT-R4000) reciprocating friction and wear tester [Figure 3] was used to evaluate the tribological properties of the prepared greases. The upper ball of 5-mm diameter (AISI 52,100 steel, surface roughness Ra = 0.06 μm, hardness 7.05-7.57 GPa) slides reciprocatingly at an amplitude of 5 mm against the stationary lower discs (ø 24 × 7.9 mm, 0.03-0.35P, 5.5-7.0Sn, surface roughness Ra = 0.05 μm, hardness 2.65-2.75 GPa) to achieve contact between friction pairs. All the tests were conducted at RT and air, and each test lasted for 30 min. All the steel balls and discs specimens were cleaned in petroleum ether using an ultrasonic cleaner for 10 min. Prior to the friction and wear test, about 1 g grease was introduced to the ball-discs' contact area. A computer connected to the MFT-R4000 meter could automatically record the coefficient of friction (COF). The optical microscope was used to determine the wear width of the lower discs. Three repeated measurements were performed, and the average values with error bars were also reported in this paper. The EVO-18 SEM (Zeiss, Germany), Raman spectroscopy with 514 nm laser excitation (Renishaw, UK), EDX (Bruker, Germany) and XPS (Escalab 250, USA) were used to obtain the morphologies of the worn surfaces and analyze the elements in the tribofilm.

Figure 3 MFT-R4000 reciprocating friction and wear tester



3. Results and discussion

3.1 Properties of the greases

3.1.1 Physicochemical characteristics of conductive greases

Table III records the typical properties of the prepared greases. As can be observed, all the prepared conductive greases have a good corrosion resistivity property (copper corrosion 1a), the dropping points of KB greases are higher than others and their penetration are the lowest. The reason of

Table III Typical properties of the prepared conductive greases

Sample	Dropping point (°C)	1/4 Penetration (0.1mm)	Copper corrosion (T2copper, 100°C, 24h)
Base grease	213	97.9	1a
Lithium + 1.6 Wt.% KB grease	265	85.4	1a
Lithium + 1.8 Wt.% KB grease	297	81.3	1a
Lithium+2.0 Wt.% KB grease	334	78.8	1a
Lithium + 2.2 Wt.% KB grease	–	76.4	1a
Lithium + 2.4 Wt.% KB grease	–	74.5	1a
Lithium + 1.6 Wt.% AB grease	220	95.1	1a
Lithium + 1.8 Wt.% AB grease	231	94.1	1a
Lithium + 2.0 Wt.% AB grease	239	93.8	1a
Lithium + 2.2 Wt.% AB grease	241	91.9	1a
Lithium + 2.4 Wt.% AB grease	246	89.8	1a
Lithium + 1.6 Wt.% CB grease	234	95.8	1a
Lithium + 1.8 Wt.% CB grease	236	93.7	1a
Lithium + 2.0 Wt.% CB grease	241	90.6	1a
Lithium + 2.2 Wt.% CB grease	248	89.2	1a
Lithium + 2.4 Wt.% CB grease	251	88.4	1a

this physical property is that the KB has relative high SSA and pore volume (Choi *et al.*, 2010; Shen *et al.*, 2012). High SSA can cause obstacles to the movement of the liquid molecules attributing to the surface force (which is similar to the intermolecular force). Thus, the KB has relative high adsorption which would be responsible for the high dropping point and low penetration (Wang *et al.*, 2004).

3.1.2 Effect of temperature on conductivities

The conductivities of the prepared greases at different temperature are shown in Figure 4. As is seen from Figure 4(a), with additive concentration increasing, there is a nonlinear relationship between conductivity and additive concentration, and the evolution trend of conductivity matches the percolation theory (Ruschau *et al.*, 1992). The data in Figure 4(b) and (c) also have the same change trend. The KB grease possesses much higher conductivities than other greases at different temperatures, and with the concentration increasing, all the greases reaches the highest conductivities at concentration of 2.4Wt.%:

- the conductivity of KB grease is about 4.5E7 times higher than AB and CB greases at RT;
- the conductivity of KB grease is about 1.7E7 times higher than AB and CB greases at 100°C; and
- the conductivity of KB grease is about 8.9E4 times higher than AB and CB greases at 150°C.

Figure 5 is the distribution of additives of nanoparticles in greases which can explain the improvement of the conductive capacity. Attributable to the rich branch structure of KB nanoparticles, there is always a part of KB gathering together or only separated by very thin oil membrane layers (Yang *et al.*, 2013; Zhang *et al.*, 2011). Then the electrons activated by thermal vibration or strong internal electric field can cross the barrier formed by the oil membrane layer and jump to the adjacent nanoparticles, forming a larger tunnel current (Simmons, 1963; Li *et al.*, 1997; Beek and Pul, 2003; David, 2000). At 100°C, the rising temperature activates more electrons to jump, and leads to increasing conductivity. In the case of CB and AB, because of the spherical shape, there are less contact points. CB and AB nanoparticles are covered by

Figure 5 Distribution and mechanism of KB, AB and CB nanoparticles in conductive grease

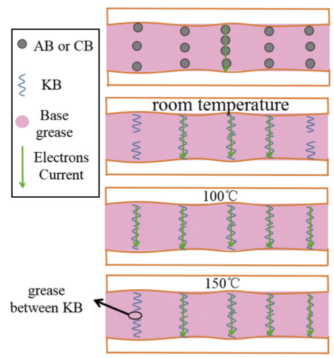
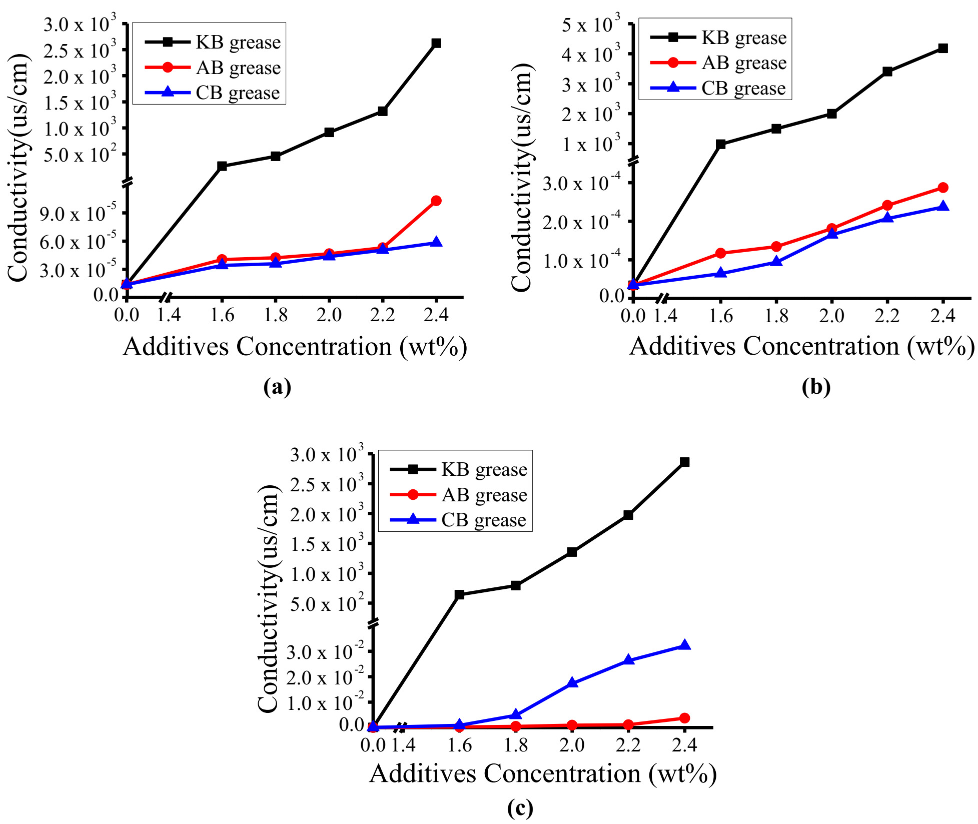


Figure 4 Conductivities of the conductive greases at different temperatures



Notes: (a) RT; (b) 100°C; (c) 150°C

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oil, isolatedly distributed in oil and cannot link each other to complete the conductive path, leading to low conductivity. However, when the temperature is 150°C, the thermal expansion of grease is so serious that the distances among KB particles become large. Thus, compared with 100°C, KB cannot form a good enough conductive network and the conductivity of KB grease at 150°C is 32 per cent lower than that of KB grease at 100°C. The conductivities of AB and CB greases gradually increase when the temperature is raised from 25°C to 150°C. This is because when the AB and CB concentration are between 1.6 and 2.4 per cent, the number of contact points is relative little. At this condition, the thermal expansion of grease has little influence on conductivity of AB and CB greases. Only because of the increase in temperature, the electronic thermal motion is more intense, leading to a little higher conductivity (Liu *et al.*, 2013).

3.1.3 Contact resistance results

Table IV enumerates the electrical conductivities of the conductive greases (additives content is 1.8Wt.%) at different torques (5, 10, 15 N·m). The stabilizing coefficient is the specific value of final contact resistance (F-R) and initial contact resistance (I-R). The KB grease has the lowest contact resistance and stabilizing coefficient among the three kinds of prepared greases. It indicates that KB can decrease heat production and energy loss, and promote the current steadier.

3.2 Tribological test results

To evaluate the tribological performances of the additives, this article studied three predominant factors (additive content, load and frequency).

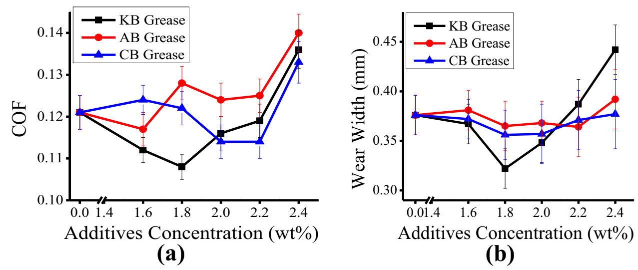
3.2.1 Effect of additive concentration

Figure 6 shows COFs and wear widths of the conductive greases at 15 N, 5 Hz and RT. When the concentration of KB is between 1.6 and 2.2Wt.%, the COFs of KB greases (0.108-0.119) are lower than that of base grease (0.121). When the concentration of KB is 1.8Wt.%, the COF of KB grease is reduced by about 11 per cent and reaches the lowest value. With increase in the KB concentration, the wear width decreases first and then increases. When the concentration of KB is 1.8Wt.%, the wear width (0.322 mm) is about 14 per cent lower than base grease (0.376 mm) and the wear width is the lowest. The results indicate that when the concentration of KB is 1.8Wt.%, the friction-reducing and wear resistance properties are the best among all the greases. Therefore, in the following experiments, the concentration of additives in grease is 1.8Wt.%.

3.2.2 Effect of load

Figure 7 lists the COFs and wear widths of the conductive greases at different loads, 5 Hz and RT. At low load (15 N),

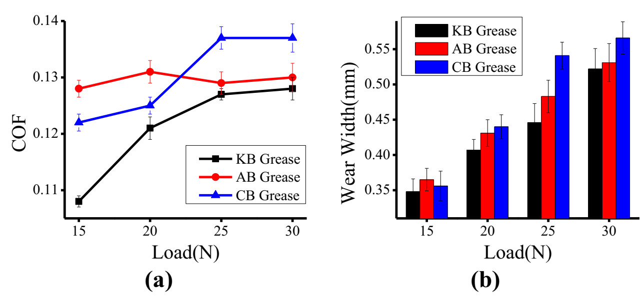
Figure 6 The evolution of average COFs (a) and average wear widths (b) for the prepared greases at different additives contents at RT



Notes: Stroke = 5 mm, duration = 30 min; load: 15 N; frequency= 5 Hz

the COF of KB grease (0.108) is obviously smaller than that of AB (0.128) and CB greases (0.122). With the load increasing, the COF of KB grease increases, and that of AB and CB greases, change little. When the loads are 25 and 30 N, the COFs of KB grease (about 0.127) are close to those of AB grease (about 0.130). The results demonstrate that the KB particle shows better friction-reducing property than AB and CB at low load; however, at high load, the friction-reducing property of AB and KB is similar. When load is 15 N, the wear width of KB grease (0.348 mm) is close to that of AB (0.365 mm) and CB greases (0.356 mm). However, at 20, 25 and 30 N, compared with CB grease, the wear widths of KB grease reduced by 7.5, 17.6 and 7.8 per cent, respectively. The results indicate that KB grease shows better wear resistance than AB and CB greases.

Figure 7 The evolution of average COFs (a) and average wear widths (b) at 15, 20, 25 and 30N for the conductive greases at RT



Notes: Additives content = 1.8Wt.%; stroke = 5 mm; duration = 30min; frequency = 5 Hz

Table IV Contact resistance of conductive greases ($\mu\Omega$)

Sample torque	KB grease I-R	KB grease F-R	KB grease Stabilizing coefficient	AB grease I-R	AB grease F-R	AB grease Stabilizing coefficient	CB grease I-R	CB grease F-R	CB grease Stabilizing coefficient
5	23.5	23.1	1.02	33.2	31.8	1.04	37.3	36.2	1.03
10	19.8	19.7	1.01	29.2	28.4	1.03	29.2	28.3	1.03
15	17.9	17.4	1.03	25.4	24.7	1.03	28.6	27.9	1.03

3.2.3 Effect of frequency
Figure 8 shows the COFs and wear widths of the conductive greases at different frequencies, 20 N and RT. It is evident from Figure 8(a) that the COFs of the three kinds of greases reduce with frequency increasing. At different frequencies, the COFs of KB grease (about 0.121-0.124) are lower than those of AB and CB greases (all above 0.125). The results demonstrate that KB grease has better friction-reducing property among all the greases. From Figure 8(b), it is obviously seen that the wear widths of KB grease are close to those of CB grease at 2 and 5 Hz. At 3 and 4 Hz, the wear widths of KB grease are about 12 per cent lower than those of CB grease. The results demonstrate that KB grease has a little better wear resistance than AB and CB greases at all frequencies.

3.3 Surface analysis
To explore the friction and wear mechanisms, SEM, Raman spectroscopy, EDX and XPS were used to analyze the worn surfaces. The morphologies (SEM) of the worn surfaces lubricated by prepared greases at 20 N, 5 Hz and RT are shown in Figure 9. It can be observed clearly that the worn surface lubricated by KB grease [Figure 9(a)] is smoother and there are less wear scars. In sharp contrast, the worn surfaces lubricated by AB [Figure 9(b)] and CB greases [Figure 9(c)] have deep scuffing and dense holes, which are dominated by abrasive wear and adhesion wear. A smoother surface is obtained by the KB greases, as the KB possesses better anti-wear property than AB and CB.

The Raman spectrum of the nanometer KB is shown in Figure 10(a). KB is confirmed by the Raman spectra analysis of the two most intense features of the D band (about 1,335 cm^{-1}) and G band (about 1,580 cm^{-1}). The most prominent D band and G band originate from the breathing modes of sp^2 atoms in rings and the bond stretching of all pairs of sp^2 atoms in both rings and chains, respectively (Ferrari, 2007). After the tribological test, the discs were cleaned ultrasonically in petroleum ether for 10 min. We examined the wear scar with the Raman microscope. Figure 10(b) shows the Raman

Figure 10 Raman spectrum of the nanometer KB (a) and worn surface (b)

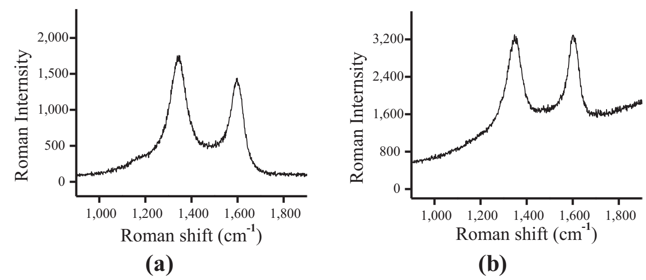
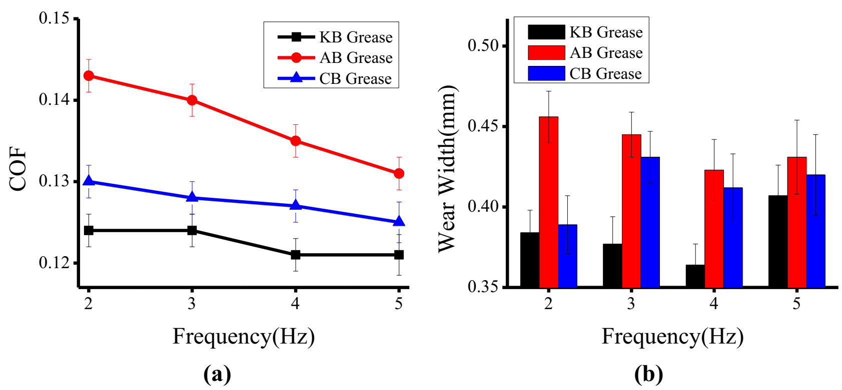
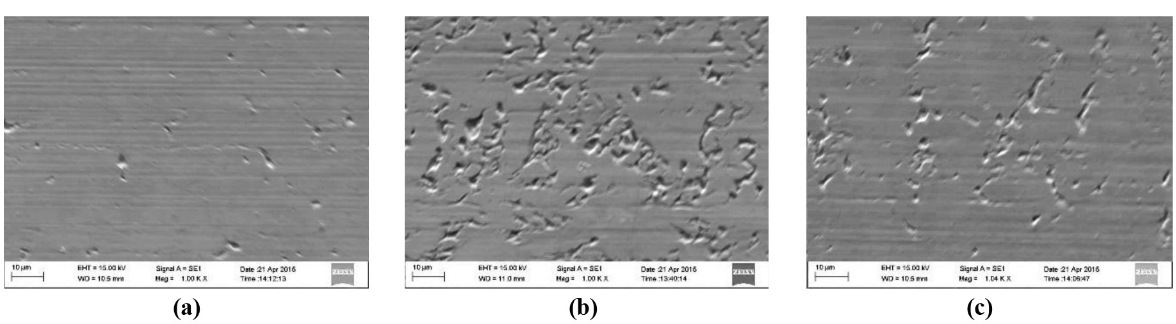


Figure 8 The evolution of average COFs (a) and average wear widths (b) at 2, 3, 4 and 5 Hz for the prepared greases at RT



Notes: Additives content = 1.8Wt.%; stroke = 5 mm; duration = 30 min; load = 20 N

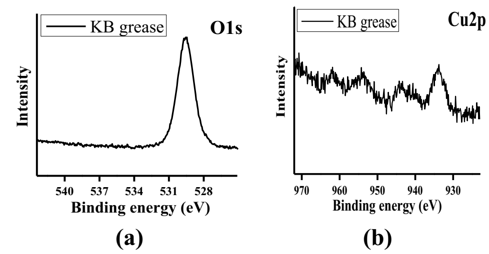
Figure 9 SEM morphologies of the worn surfaces lubricated by the greases



Notes: (a) KB grease; (b) AB grease; (c) CB grease (magnification is 1,000x, load = 20 N, stroke = 5 mm, frequency = 5 Hz and duration = 30 min) spectrum of wear scar. The D band and G band of the worn surface confirmed the presence of KB as a readily sliding tribolayer on the friction pairs. The Raman test results proves that nanometer KB is indeed present and has formed a deposited film on the worn surface. Raman spectroscopy was carried out to further confirm that the better tribological behavior is attributed to the presence of KB on the rubbing surface.

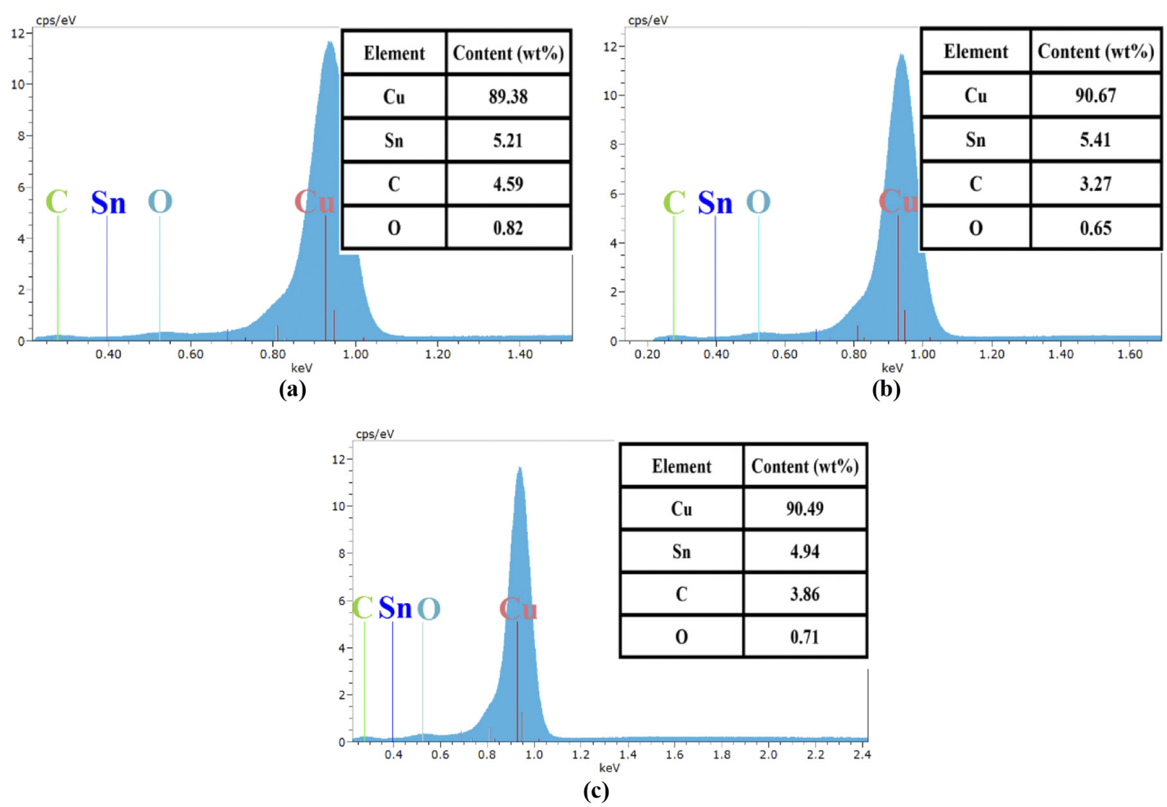
Figure 11 (EDX of worn surfaces) shows the main elements and their contents on the worn surfaces lubricated by the prepared greases. The peaks confirm the existence of C and O elements and their contents on the worn surface lubricated by KB grease are more than those of AB and CB greases. Combining the Raman spectra analysis, we could infer that there is more carbon deposited on the worn surface lubricated by KB grease and it might form a better protective film than AB and CB greases. Figure 12 shows the XPS spectra of O1s and Cu2p on the worn surface lubricated by KB grease. The peak of O1s shows that oxide species occur on the worn surface. The peak of O1s (about 529.6 eV) and the peak of Cu2p (about 933.9 eV) might be assigned to CuO (Mcintyre *et al.*, 1975; Li *et al.*, 2013). The XPS analysis implies that there might be a protective film formed by metallic oxide on worn surface. And because the most of the contents of the O element on worn surface is lubricated by KB grease, we could surmise a little better protective film was formed by metallic oxide on KB lubricating surface.

Figure 12 XPS of the worn surfaces lubricated by KB grease at 20 N



From the above results, the better friction-reducing and wear resistance of KB grease could be explained by the following factors. First, during the course of friction and wear, nanometer KB could act as spacers, preventing the close touch between the contact surfaces (Chen *et al.*, 2002; Jiang *et al.*, 2008). Second, during the sliding process, the nanometer KB could deposit on the worn surface to form a better tribofilm. Third, the oxide species also occur on worn surface to form a metallic oxide protecting film. Because of the above reasons, nanometer KB has better friction reducing and anti-wear properties than AB and CB. However, with the KB content increasing, the COFs and wear widths of KB greases become higher. The reason might be that nanometer KB has large SSA and rich branch structure. Its high content may lead to nanometer KB not dispersing uniformly in the base grease.

Figure 11 EDX of the worn surfaces lubricated by conductive greases at 20 N



(a)

Element	Content (wt%)
Cu	89.38
Sn	5.21
C	4.59
O	0.82

(b)

Element	Content (wt%)
Cu	90.67
Sn	5.41
C	3.27
O	0.65

(c)

Element	Content (wt%)
Cu	90.49
Sn	4.94
C	3.86
O	0.71

Notes: (a) KB grease; (b) AB grease; (c) CB grease (load = 20 N, stroke = 5 mm, frequency = 5 Hz; duration = 30 min)

Therefore, when the KB content is beyond 2.0 per cent, the COFs and wear widths of KB greases become higher.

4. Conclusions

The conclusions drawn from the above experimental work are:

- The conductive grease prepared with nanometer KB has a prominent conductive capacity at RT, 100°C and 150°C. When the concentration is 2.4 per cent, the conductivity of KB grease is about 4.5E7 times of AB and CB grease at RT. The contact resistance lubricated by KB grease is also lower than AB and CB greases. This indicates that nanometer KB has a better application in conductive grease.
- The Raman spectra, EDX and XPS analyses indicate that a protective film is formed by the C element deposited and the metallic oxide on worn surface in the course of friction and wear, and the higher C concentration leads to the formation of a better protective film; thus, nanometer KB has better friction-reducing and wear resistance than AB and CB. The optimal concentration for nanometer KB is recommended as 1.8Wt.%.

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