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Influence of Carbon Nanotubes on Conductive Capacity and Tribological Characteristics of Poly(ethylene Glycol-Ran-Propylene Glycol) Monobutyl Ether as Base Oil of Grease

Carbon black (CB) and three kinds of carbon nanotubes (CNTs) including multiwalled CNTs (MWCNTs), carboxyl multiwalled CNTs (CMWCNTs), and single-walled CNTs (SWCNTs) were doped as conductive additives in poly(ethylene glycol-ran-propylene glycol) monobutyl ether (denoted as PAG) to afford conductive greases in the presence of polytetrafluoroethylene (PTFE) as the thickener and acetone as the polar dispersant. The effects of the conductive additives on the conductive capacity and tribological characteristics of the PAG grease were investigated, and the tribological action mechanisms of the conductive additives were analyzed in relation to worn surface analyses by scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS). Results indicate that the SWCNTs can reduce the volume resistivity of the base grease by over 10,000 times. In the meantime, the CB and the three kinds of CNTs as conductive additives can improve the tribological characteristics of the base grease to some extent, and the CNTs are advantageous over the CB in improving the friction-reducing and antiwear abilities of the base grease. The reason lies in that CNTs with a small size and a large specific surface area can be easily adsorbed on sliding steel surfaces to form a surface protective film. [DOI: 10.1115/1.4031232]

1 Introduction

Grease is harnessed in electrical apparatus such as microelectronic mechanical system, electrical switch, integrated circuit, and power machine [1–3]. Besides reducing friction and wear, the grease plays an important role in enhancing lubrication efficiency, decreasing contact resistance, and extending service life, thereby saving energy [4]. In this sense, it is imperative to introduce conductive additives so as to improve the conductive ability and tribological characteristics of grease. However, traditional conductive additives such as gold, silver, and nickel powders are too expensive for massive application, copper powder is easily oxidized, and ionic liquids are usually corrosive to metal [5].

The above mentioned disadvantages of the traditional conductive additives, fortunately, could be overcome by replacing with CNTs, because the CNTs possess outstanding mechanical characteristics (high tensile strength, thermal conductivity, and electrical conductivity) as well as desired electrical and tribological characteristics [6–16]. Liu et al. investigated the influence of temperature and velocity on the tribological characteristics of the CNTs-thickened grease, but they did not correlate the tribological characteristics of the grease with the functional group like COOH in the grease molecule [17,18]. Mohamed et al. investigated the effect of one kind of CNT on the tribological characteristics of lithium grease, but they did not provide details about whether single-walled or multiwalled CNTs were used [19].

In the present research, we select three kinds of CNTs, i.e., MWCNTs, CMWCNTs, and SWCNTs as conductive additives of a synthetic grease and investigate their effect on the tribological characteristics of the base grease. This paper reports the influence of various conductive additives on the physicochemical properties and tribological characteristics of the base grease in relation to worn surface analyses by SEM and EDS.

2 Experiment Details

2.1 Materials. CNTs were provided by Chengdu Organic Chemicals (Chengdu, China). CB provided by Kejun Chemical Company (Quanzhou, China) was introduced as a conductive additive for comparative study. PAG, commercially obtained from Dow Chemical, was used as the base oil for preparing conductive greases. PTFE (provided by Dyneon TF 9207, density: 2.2 g/cm^3, and grain size: $\sim$4 μm) was used as the thickener to thicken the base oil into grease. Acetone (provided by Sinopharm, Beijing, China) was used as the polar dispersant. All the chemical reagents employed are analytical grade and used as-received. Table 1 lists the physicochemical characteristics of the CB and CNTs, and Fig. 1 shows their SEM images.

2.2 Preparation of Conductive Greases. Variable contents of the CB and CNTs were dispersed in the PAG base oil under ultrasonic vibration to obtain the PAG-based nanofluids (NFs). Based on the fluidity of the NFs, the content of the conductive additives in the as-prepared conductive greases is adjusted as 0.1%, 0.3%, 0.5%, 0.7%, 1.0%, and 1.5% (mass fraction, the same hereafter). Briefly, pure PAG or NFs (70%) were injected into the reaction vessel and agitated vigorously. Then the PTFE (30%) was gently poured into the reaction vessel under vigorous agitation, followed by dropwise injection of acetone (mass: about 50% of that of the PTFE) under 30 mins of agitation to achieve

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Table 1 Typical physicochemical properties of CB and three kinds of CNTs

Sample	CB	MWCNTs	CMWCNTs	SWCNTs
Outer diameter (nm)	35	50	50	1–2
Inner diameter (nm)	—	5–15	5–15	0.8–1.6
Length (μm)	—	10–20	10–20	5–30
Electric conductivity (s/cm)	0.04	>100	>100	>100
Purity (wt.%)	98	>95	>95	>90
Special surface area (m^2/g)	1031	>40	>40	>380
–COOH content (wt.%)	—	—	0.49	—

homogeneous dispersion of the PTFE in PAG (or NFs). Resultant mixture was heated to 80 °C and held there for 30 mins to eliminate acetone. Upon completion of acetone evaporation, the resultant mixture was cooled to ambient temperature and rolled on a three-roller mill to afford the target products, i.e., the base grease or conductive greases with a different content of conductive fillers.

2.3 Characterization. The conductivity of a series of the NFs and conductive greases was measured with a DDSJ-308A conductivity tester. The anticorrosion behavior, the penetration capacity, and the dropping point of the greases were examined by Chinese National Standards GB/T 7326 (copper strip test), GB/T 269, and GB/T 3498, respectively. A GEST-121 volume-surface resistivity apparatus and a DDSJ-308A conductivity tester were utilized to assess the volume resistivity and conductivity of the greases.

2.4 Friction and Wear Test. An MFT-R4000 reciprocal friction and wear apparatus was performed to evaluate the tribological characteristics of as-synthesized conductive greases in a ball-on-disk configuration. The upper ball (AISI 52100 steel; hardness: 710 HV, ø 5 mm) was driven to reciprocally slide against the lower stationary disk (AISI 52100 steel; hardness: 500–520 HV, ø 24 × 7.9 mm) at a stroke of 5 mm and an ambient temperature of ~20 °C for a duration of 30 mins. Before and after each sliding test, the steel ball and disk were ultrasonically washed with petroleum ether for 10 mins. Prior to sliding, approximately 1 g of the to-be-tested grease was introduced to the contact zone of the sliding pair. The coefficient of friction (COF) was recorded by a computer attached to the friction and wear tester. An OLS4000 three-dimensional topography device was employed to measure the wear volume loss. The sliding test under each preset condition was repeated three times to minimize data scattering. The mean values of the COF and wear volume are provided in association with error bars in this paper. The morphology and chemical composition of the wear scars were analyzed with a JSM-6700F scanning electron microscope (SEM; JEOL, Japan) equipped with an EDS attachment.

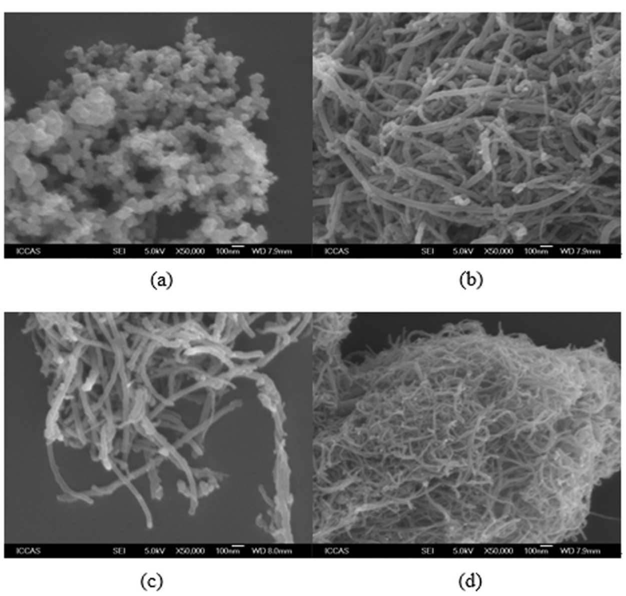

Fig. 1 SEM images of (*a*) CB, (*b*) MWCNTs, (*c*) CMWCNTs, and (*d*) SWCNTs

Table 2 Physicochemical properties of as-prepared conductive greases containing 1.0% conductive additives

Sample	Dropping point (°C)	Penetration (1/4 mm)	Copper corrosion (T2 copper, 100 °C, 24 hrs)	Volume resistivity (Ω cm)
Base grease	283	70.6	1a	2.8×10^8
CB grease	332	58.1	1a	2.1×10^7
MWCNTs grease	332	58.3	1a	1.9×10^5
CMWCNTs grease	330	57.7	1a	4.4×10^5
SWCNTs grease	331	57.6	1a	2.3×10^4

3 Results and Discussion

3.1 Physicochemical Characteristics. Table 2 lists some physicochemical characteristics of the as-synthesized greases. All the conductive greases exhibit higher dropping point (approximately 330 °C) than base grease (approximately 280 °C), and they also exhibit good anticorrosion behavior. This indicates that the conductive additives have great influences on the dropping point and anticorrosion behavior of the base grease. The reason might lie in that CB and various kinds of CNTs have a high specific surface area and can retard the transfer of liquid molecules, thereby leading to increased dropping point and anticorrosion ability [20–24].

Figure 2 shows the conductivity of the as-prepared NFs and conductive greases. It can be seen that the conductivity of various NFs is ranked in the order of CBs < CMWCNTs < MWCNTs < SWCNTs, and their highest conductivity is reached at a conductive additive content of 2.1% (the maximum conductivity of SWCNTs, MWCNTs, and CMWCNTs NFs is about 7.8, 4.2, and 1.5 times that of CB NF). Similarly, the greases containing 1.5% of conductive additives exhibit the highest conductivity, and the maximum conductivity of the greases doped by 1.5% of SWCNTs or MWCNTs or CMWCNTs is about 491, 104, and 61 times that of the grease doped by 1.5% of the CB. This demonstrates that the CNTs exhibit better conductive capacity than the CB. Moreover, as shown in Table 2, SWCNTs, MWCNTs, and CMWCNTs can reduce the volume resistivity of the base grease by about 12,000, 1500, and 600 times, but the CB can only reduce the volume resistivity of the base grease by ten times, which is in accordance with percolation theory [25]. Figure 3 shows the distribution of the conductive additives in the greases. It can be seen that the nanoscale conductive additives are well separated by a grease layer which acts as a barrier. Tubular MWCNTs and SWCNTs tend to gather together, and hence they are separated by a thin grease layer (low barrier). As a result, the electrons activated by the

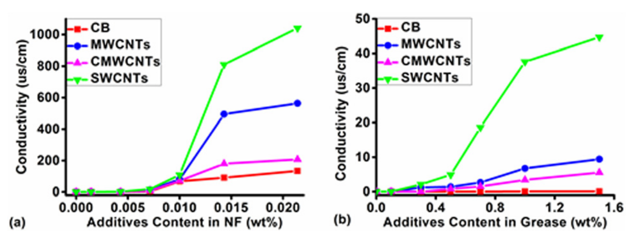

Fig. 2 Conductivities of (*a*) NFs and (*b*) conductive greases

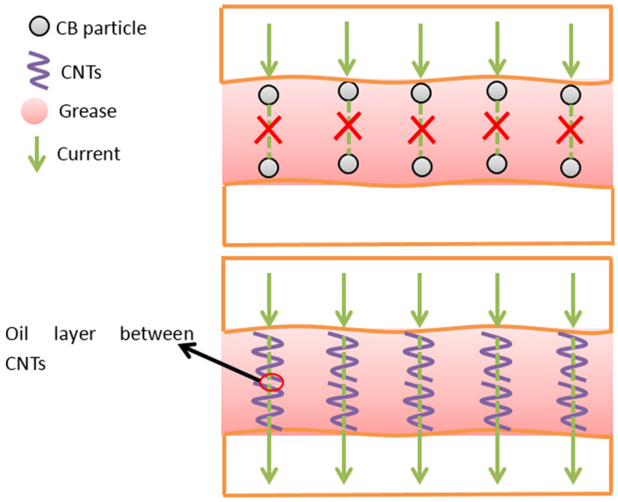

Fig. 3 Distribution and action mechanism of CB nanoparticles and CNTs in conductive grease

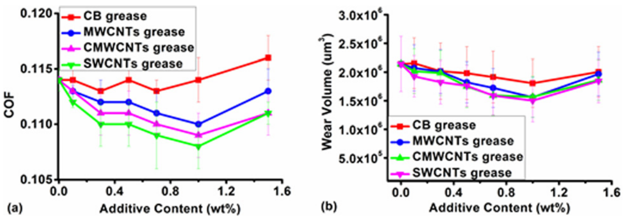

Fig. 4 (*a*) Mean COFs and (*b*) mean wear volumes of lower steel disks lubricated by as-prepared greases with a different additive content (load: 50 N, frequency: 5 Hz, stroke: 5 mm, and duration: 30 mins)

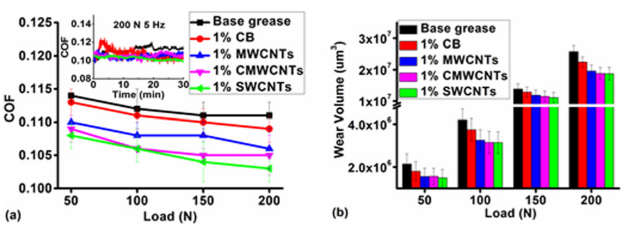

Fig. 5 (*a*) Mean COFs and (*b*) mean wear volumes of lower steel disks lubricated by as-prepared greases at various loads (frequency: 5 Hz, stroke: 5 mm, and duration: 30 mins)

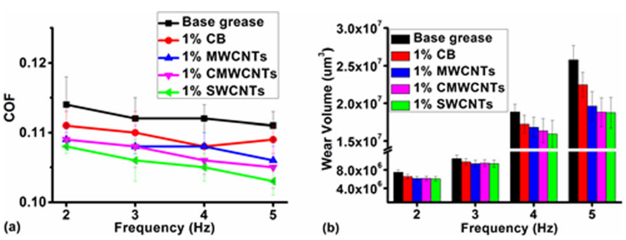

Fig. 6 (*a*) Mean COFs and (*b*) mean wear volumes of lower steel disks lubricated by as-prepared greases at various frequencies (load: 200 N, stroke: 5 mm, and duration: 30 mins)

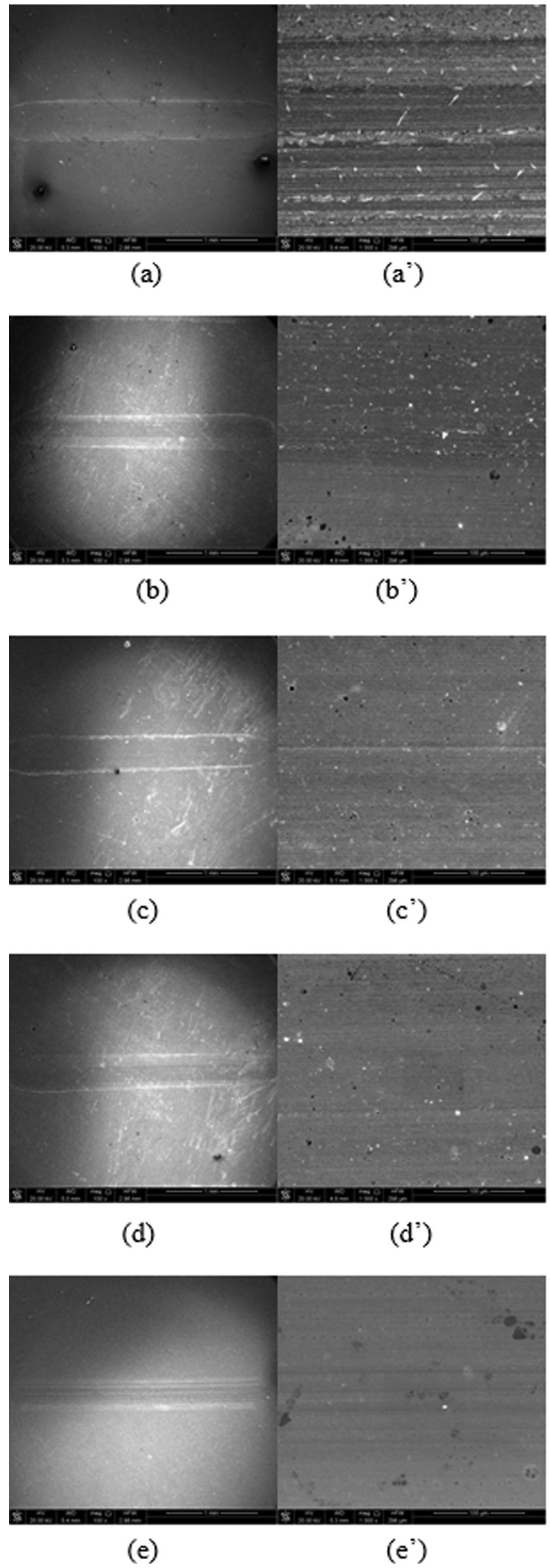

Fig. 7 SEM images of the wear scars of lower steel disks lubricated by ((*a*) and (*a*′)) base grease, ((*b*) and (*b*′)) CB-doped grease, ((*c*) and (*c*′)) MWCNTs-doped grease, ((*d*) and (*d*′)) CMWCNTs-doped grease, and ((*e*) and (*e*′)) SWCNTs-doped grease (load: 200 N, stroke: 5 mm, and duration: 30 mins)

thermal vibration or strong internal electric field can easily cross the barrier and jump to the adjacent particles to form a larger tunnel current, thereby significantly increasing the conductivity [26–29]. In the case of CMWCNTs which has the similar tubular structure, its carboxyl allows it to be easily dispersed in the base grease. As a result, the surface of CMWCNT particles is well covered by a thick grease layer (high barrier), thereby leading to increased volume resistivity of the conductive grease. Differing

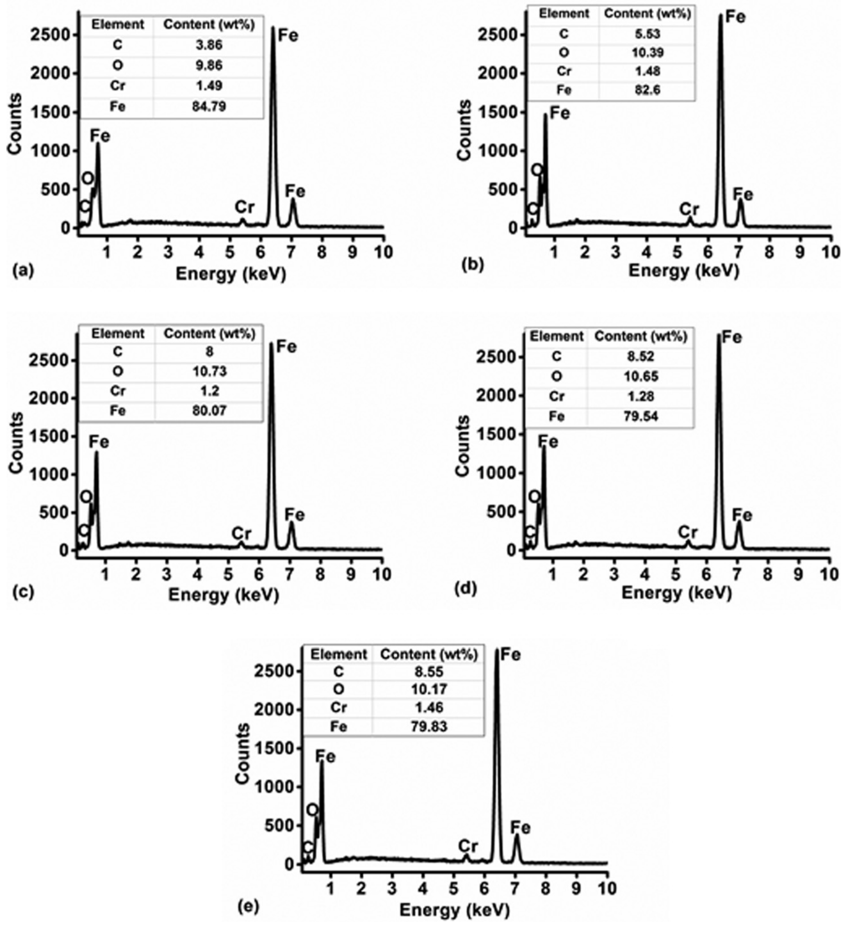

Fig. 8 EDS patterns of the wear scars of lower steel disks lubricated by (*a*) and base grease, (*b*) and CB-doped grease, (*c*) and MWCNTs-doped grease, (*d*) and CMWCNTs-doped grease and (*e*) and SWCNTs-doped grease (load: 200 N, stroke: 5 mm, and duration: 30 mins)

from various CNTs, the CB particles without tubular structure cannot link each other to form desired conductive path in the base grease, thus CB-doped grease exhibits high volume resistivity. Therefore, MWCNTs and SWCNTs are recommended as the candidate conductive fillers to improve the conductivity of the base grease and reduce the heat generation and energy consumption as well.

3.2 Tribological Characteristics

3.2.1 Influence of Additive Content. Figure 4 reveals the variation of the COF and wear volume of the steel disk under the lubrication of greases doped with various conductive additives (frequency: 5 Hz, stroke: 5 mm, load: 50 N, and duration: 30 mins, ambient temperature). It can be seen that all the tested CNTs can effectively improve the tribological characteristics of the base grease. Namely, the base grease exhibits a COF of 0.114; the CB-doped grease (CB content: 0.1%–1.0%) exhibits a COF of 0.113–0.114; and the CNTs-doped greases (additive content: 1.0%) exhibit the COFs of 0.108 (SWCNTs), 0.110 (MWCNTs), and 0.109 (CMWCNTs), respectively. Besides, the wear volumes of the steel disks lubricated by the base grease and 1.0% CB-doped grease are about $2.1 \times 10^6\,\mu m^3$ and $1.8 \times 10^6\,\mu m^3$; all the wear volumes of the steel disks lubricated by CNTs-doped greases (additive content: 1.0%) are about $1.5 \times 10^6\,\mu m^3$. Moreover, when the content of CNTs is higher than 1.0%, the COFs and wear volumes increase with increasing additive content, possibly because the CNTs with higher contents tend to agglomerate and act as abrasive particles.

3.2.2 Influence of Load. Figure 5 presents the mean COFs and wear volumes of the steel–steel sliding pair under grease lubrication at various loads (5 Hz, 5 mm, and 30 mins). It is seen that all the additives are favorable for slightly reducing the COFs, and the COFs tend to decline with increasing load. The mean COFs of CNTs-doped greases are between 0.108 and 0.103, and they are smaller than those of the base grease (0.114–0.111) or CB-doped grease (0.113–0.109). This reveals that all kinds of CNTs have better friction-reducing ability than the CB in the selected range of load. In the meantime, the wear volume of the steel disk under the lubrication of CNTs-doped greases is relatively lower than that under the lubrication of CB-doped grease, which indicates the CNTs have better antiwear ability than the CB particles.

3.2.3 Influence of Frequency. Figure 6 gives the mean COFs and mean wear volumes of the steel disks lubricated by conductive greases at various frequencies (200 N and 30 mins). The COFs under the lubrication of CNTs-doped greases are slightly smaller than those under the lubrication of base grease and CB-doped grease, and they tend to decrease with increasing frequency. Besides, the mean wear volume of the steel disks lubricated by CNTs-doped greases is smaller than that lubricated by the base grease or CB-doped grease, which well corresponds to the better antiwear ability of various CNTs than CB.

3.3 Worn Surface Analysis. SEM and EDS analyses of worn steel disks were conducted to explore the lubricating mechanism of the conductive greases. Figure 7 shows the SEM images of the wear scar of lower steel disks lubricated by the grease doped with various conductive additives (200 N, 5 Hz, and 30 mins). It can be seen that the wear scar of the disk lubricated by base grease alone is relatively rough and contains some scratches and furrows (Figs. 7(*a*) and 7(*a*')), and the wear scar of the steel disk lubricated by CB-doped grease seems to be a little bit smoother and contains fewer scratches (Figs. 7(*b*) and 7(*b*')). Different from the above-mentioned, the wear scars lubricated by CNTs-doped greases are much smoother and smaller (Figs. 7(*c*)–7(*e*) and 7(*c*')–7(*e*')), which corresponds well with the better antiwear abilities of various CNTs than the CB.

Figure 8 provides the EDS spectra of some typical elements on the wear scars (200 N, 5 Hz, and 30 mins). The content of C element on the wear scars lubricated by CNTs-doped greases is obviously higher than that of C element on the wear scars lubricated by base grease and CB-doped grease. Besides, the content of Fe element on the wear scars lubricated by CNTs-doped greases is obviously lower than that of Fe element on the wear scars lubricated by base grease and CB-doped grease. Therefore, it can be inferred that various CNTs conductive additives can be well deposited on steel surfaces to form protective films, thereby avoiding the direct contact of the sliding interface and effectively reducing the friction and wear of the steel–steel contact.

The good tribological characteristics of the CNTs as conductive additives of grease can be explained as follows. First, the CNTs can improve the mechanical strength of the grease, thereby improving the antiwear ability of the base grease [30,31]. Second, the CNTs conductive additives in the grease can fill up the voids on sliding steel surfaces and increase the contact area of the steel–steel pair, thereby performing like spacers to avoid direct contact between the contact interfaces [32]. Third, the CNT nanoparticles can act as rollers to transform sliding mode into rolling mode in some sense, thereby reducing the shear stress and COFs [33–36]. Fourth, CNT nanoparticles with small size and large specific surface area can be well adsorbed on sliding surfaces to participate in the lubrication process, thereby improving antiwear ability [37]; and in the meantime, the CMWCNTs with carboxyl can easily adsorb base oil to participate in lubrication process so as to improve the antiwear ability of the base oil [38].

4 Conclusions

The three kinds of CNTs as conductive additives are able to greatly improve the conductive capacity of the base grease, and in particular, the SWCNTs can reduce the volume resistivity of the base grease by over 10,000 times. Besides, the CNTs as conductive additives can also improve the friction-reducing and antiwear abilities of the base grease. This is because CNT nanoparticles with small size and large specific surface area can be easily adsorbed on sliding steel surfaces to form a surface protective films, thereby effectively reducing the friction and wear of the steel–steel sliding pair.

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