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Tribological properties and insulation effect of nanometer TiO_2 and nanometer SiO_2 as additives in grease

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ABSTRACT

New greases were synthesized using oleophilic nanometer-TiO_2 and nanometer-SiO_2 as additives. When the additives in naphthenic oil is 0.1 wt%, the alternating current (AC) breakdown strength is enhanced by 10.4% and 8.2% at power frequency, respectively. Also the grease volume resistivities are improved by 23% and 30% compared with base grease, which use naphthenic oil as base oil. The greases tribological behaviors were explored. Scanning electron microscope linked with energy dispersive X-ray spectroscope was utilized in order to analyze these scratches. The good tribological characteristics of nanometer-TiO_2 greases and the good friction-reducing characteristic of nanometer-SiO_2 greases are ascribed to the nanoparticles mechanical effect, and are also ascribed to the protect film generated by Ti and Si deposited or metallic oxide.

1. Introduction

When grease is harnessed in electrical apparatus, such as high voltage cable connectors, electrical connection components, battery connector, etc., the electric insulating characteristics of the grease are very essential to guarantee the electrical apparatus operating safety. Besides friction-reducing and anti-wear, the grease takes on an important part in insulation in electrical apparatus, such as preventing corona discharge and eliminating arc, thus the insulating and tribological characteristics of the insulation grease are especially momentous [1–5].

The nanometer TiO_2 (nano-TiO_2) and nanometer SiO_2 (nano-SiO_2) particles possess particularly physical, chemical and electrical performance; hence they have been put into use in many realms, including functional materials, catalyst, plastics, rubbers paints, biomedicine and semi-insulation materials. Several researches are about the influences of nanoparticles on enhancing tribological characteristics [6–8]. Many articles have been carried out on TiO_2 and SiO_2 as coating materials [9–14] or reinforcement in composite materials [15,16] for achieving better tribological performance. Some papers focus on the insulation improvement of TiO_2 in transformer oil [17,18]. However, few articles related to nanometer TiO_2 and nanometer SiO_2 serviced in grease to improve the insulating and tribological characteristics have been reported.

In this paper, nano-TiO_2, nano-SiO_2, nanometer Sb doped SnO_2 (ATO), micrometer TiO_2 (micro-TiO_2) and micrometer SiO_2 (micro-SiO_2) particles were added into naphthenic oil to achieve nanofluids (NFs) and microfluids (MFs). And then, insulating greases were prepared by using pure naphthenic oil, NFs or MFs as base oil, respectively. The thickener was polytetrafluoroethylene (PTFE) (Dyneon™ TF9207), and the polarity dispersant was acetone (Sinopharm). The physicochemical, insulating and lubricating characteristics of the insulating greases were emphatically studied. The scratches were witnessed through a scanning electron microscope (SEM) (JSM-6700F, Japan), the lubricating mechanisms were probed by energy dispersive X-ray spectroscope (EDS).

2. Experiment details

2.1. Materials

According to Lv's work [17,18], the naphthenic oil used as base oil in this paper was a kind of transformer oil (25# Karamay, China), and its typical characteristics are recorded in Table 1. The density of PTFE is 2.2 g/cm^3, and the grain size is about 4 μm. The grain size of micro-TiO_2, micro-SiO_2, nano-TiO_2, nano-SiO_2, and ATO (DK nano technology, Beijing, China) are about 1 μm, 1 μm, 35 nm, 30 nm and 20 nm, respectively. The SiO_2 and TiO_2 are modified using γ-methacryloxy propyl trimethoxyl silane, thus they are oleophilic. All the chemical reagents employed in this test were analytical grade and without additional refinement. Figs. 1 and 2 are the SEM

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images and the X-ray diffraction patterns of nano-SiO_2 and nano-TiO_2, respectively.

2.2. Preparation of the modified oil and greases

The transformer oil-based MFs and NFs were prepared also according to the literatures [17,18] by dispersing the additives into transformer oil with various contents. Due to the low dispersion of the additives in transformer oil, only 0.05 wt%, 0.07 wt%, and 0.1 wt% additives contained MFs and NFs were prepared.

The greases were synthesized following the procedures below. Firstly, the pure transformer oil (MFs or NFs) was injected into the reaction vessel and agitate at once. Secondly, the PTFE powder was gently poured into the reaction vessel with fiercely agitating. As the base oil was blended homogenously with the PTFE powder, acetone whose mass was approximately half of the PTFE was injected drop by drop and agitated for about 30 min in order to confirm the PTFE powder was of entirely homo-disperse in transformer oil. Thirdly, the compound was warmed to 80 °C and conserved for 30 min to eliminate acetone. Lastly, the compound was cooled down to ambient temperature, and then the base grease (MFs grease or NFs grease) was attained after three times isolated refined grinding/homogenization periods by a three-roller mill.

2.3. Characterization of the modified oil and greases

The water content of the MFs, NFs and pure oil sample is between 8–9 μL/L. Also to measure the AC breakdown voltages according to ASTMD1816, a Jiantong 6801 automatic 50 Hz electrical breakdown tester, which had brass spherical electrodes (Fig. 3) was employed, and the gap between the electrodes was set at 1.5 mm. The voltage ascending ratio was 2 kV/s. The beginning stand-by phase was 5 min. The time interval between each breakdown was 1 min, and during the interval, the oil was agitated. All tests were executed at ambient temperature. 60 times breakdown was acquired for each sample. The copper strip tests, the penetration, and the dropping point of the insulating greases were surveyed on the basis of national standards, including GB/T 7326, GB/T 269, and GB/T 3498, respectively. A GEST-121 volume surface resistivity tester was introduced to assess the grease volume resistivity.

Table 1
Properties of the naphthenic oil.

Item	25# Karamay
Density (20 °C) (Kg/m^3)	883
Kinematic viscosity (40 °C) (mm^2/s)	9.936
AC breakdown voltage (2.5 mm gap) (kV)	60
Pour point (°C)	−35
Flash point (°C)	145
Acid value (mgKOH/g)	0.02
Moisture (mg/Kg)	<30
Interfacial tension (mN/m)	45

2.4. Tribological tests

To probe tribological characteristics of synthetic insulating greases, an MFT-R4000 reciprocating friction and wear tester as shown in Fig. 4 was utilized. Throughout the test, the upper ball (hardness 710 Hv, diameter 5 mm, AISI 52100 steel) was pressed down to contact the lower fixed disks (hardness 590–610 Hv, ø $24 \times 7.9\ mm^2$, AISI 52100 steel). The ball slides at a stroke of 5 mm back and forth. All the experiments were operated at ambient temperature and the duration was 30 min. Before and after every tribological test, all the balls and disks were cleansed in petroleum ether for 10 min utilizing an ultrasonic cleaner. Before each tribological test, approximately 1 g grease was applied to the contact interface. The friction coefficient (COF) was noted down automatically by a computer attached to the frictional tester. An optical microscope was occupied to measure the wear width on the disks. Three reduplicative tests were executed, and the mean values with an error bar are provided in the results. The scratches features were dissected utilizing an SEM, and an EDS was utilized to probe the elements on the scratches.

3. Results

3.1. Characteristics of the modified oil and greases

Fig. 5 sums up the evolution of AC breakdown voltages of the pure oil and modified oils with different additives content. It is clearly viewed that AC breakdown voltages of nano-TiO_2 NFs and nano-SiO_2 NFs increased by 10.4% and 8.2% compared with pure oil, respectively. These experimental results validate that nano-TiO_2 and nano-SiO_2 particles can enhance the AC breakdown strength of the transformer oil.

Table 2 affords the fundamental characteristics of the insulating greases. All the greases exhibit high dropping point (approximately 330 °C) and good corrosion resistivity (copper corrosion 1a). The additives have slightly influence on the grease dropping point.

Fig. 6 shows the volume resistivity of the prepared greases. The volume resistivity escalates as the additives content growing. The insulating mechanism of the additives in base matrix is displayed in Fig. 7 [19]. Nano-TiO_2 and nano-SiO_2 particles increasing the volume resistivity are corresponding to electron capture theory [20]. Existing studies have proven that electron can cooperate with oil molecules by polarizing them and together developing to a group, which float at the effect of local electric field for a short period. Then the electron and the group detaching and reforming

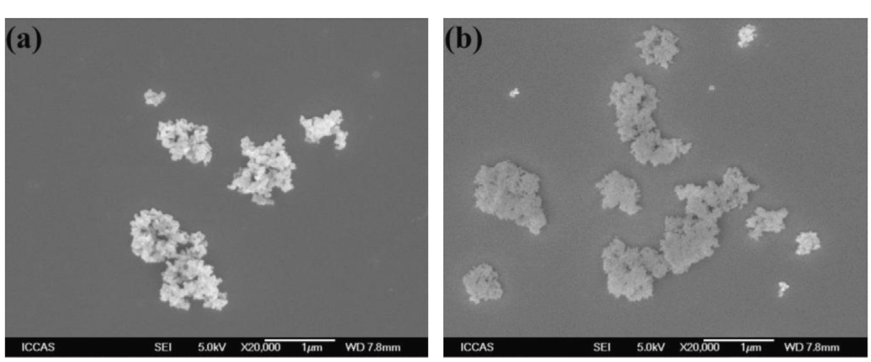

Fig. 1. Scanning electron microscope images of the (a) nano-TiO_2 and (b) nano-SiO_2.

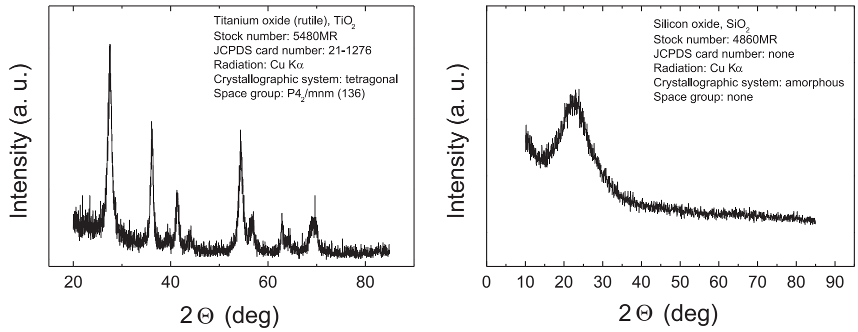

Fig. 2. The X-ray diffraction pattern of the nano-TiO_2 and nano-SiO_2.

Fig. 3. Brass spherical electrodes of the automatic 50 Hz electrical breakdown tester.

Fig. 4. The MFT-R4000 reciprocating friction and wear tester.

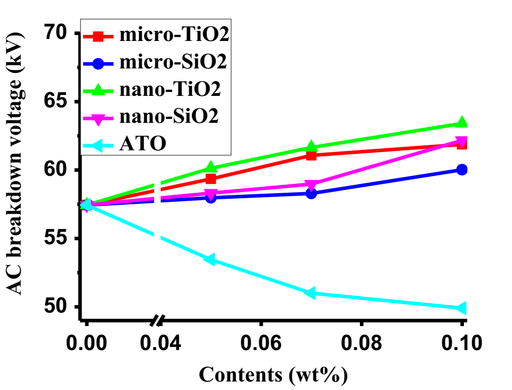

Fig. 5. AC breakdown voltage of the oil modified by five kinds of additives at different contents.

Table 2
Typical properties of the synthesized greases.

Sample	Dropping point (°C)	Penetration (1/4 mm)	Copper corrosion (T2copper, 100 °C, 24 h)
Base grease	333	76.6	1a
0.05 wt% micro-TiO_2	335	76.6	1a
0.07 wt% micro-TiO_2	333	75.9	1a
0.1 wt% micro-TiO_2	332	76	1a
0.05 wt% micro-SiO_2	330	76.8	1a
0.07 wt% micro-SiO_2	329	76.5	1a
0.1 wt% micro-SiO_2	331	76.3	1a
0.05 wt% nano-TiO_2	334	76.5	1a
0.07 wt% nano-TiO_2	333	76.5	1a
0.1 wt% nano-TiO_2	334	76.4	1a
0.05 wt% nano-SiO_2	331	76.2	1a
0.07 wt% nano-SiO_2	329	76.4	1a
0.1 wt% nano-SiO_2	329	76.1	1a
0.05 wt% ATO	332	78.2	1a
0.07 wt% ATO	333	79.6	1a
0.1 wt% ATO	332	80.9	1a

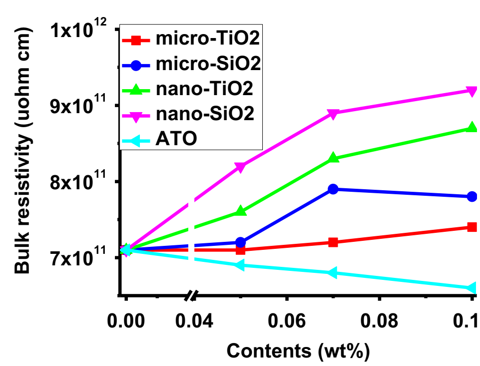

Fig. 6. Volume resistivity of the greases at different additives contents.

again results in the jumping transport process in oil. The group can be considered as an electron trap [21], and nanoparticles with high specific surface area are easier to generate high density of traps [22]. While floating from high electric field to low electric field,

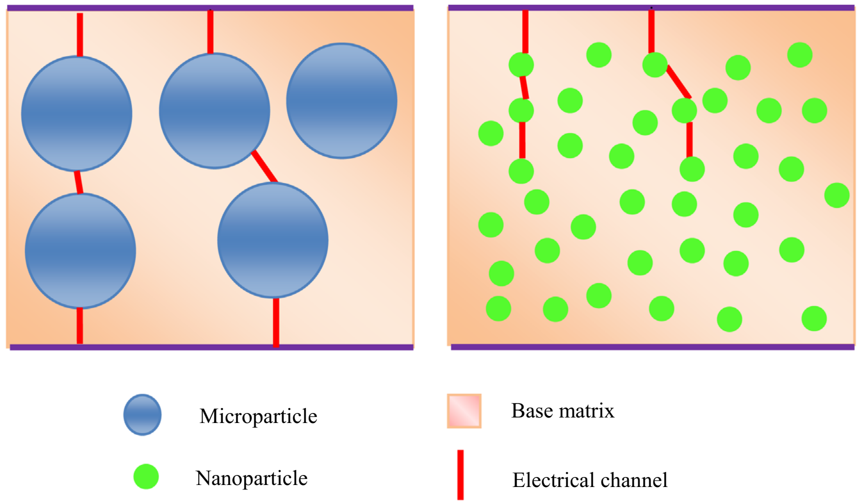

Fig. 7. Insulating mechanism of the nanoparticle and microparticles in base matrix.

fast electrons could be seized and liberated by the traps, consequently translated to slow electrons in base matrix; and finally lead to the electrical channel developing slowly. The process of seizing and liberating occurred on electron could be one of the major electron transport methods in base matrix [23]. High trap density formed by nano-TiO_2 and nano-SiO_2 particles is anticipated to greatly contribution to the enhanced dielectric strength of base matrix [24,25].

In the case of micrometer particles (micro-TiO_2 and micro-SiO_2), because of the larger grain size and the lower specific surface area than nanoparticles, the density of electron traps generated by microparticles would be also lower. Fast electron could float rapidly from high electric field to low electric field, and finally lead to the electrical channel develops faster. Also microparticles can introduce many defects which help electrons float through easily and lead to the reduction in breakdown strength and volume resistivity of the base matrix [26,27].

3.2. Tribological characteristics of the greases

The tribological characteristics of the additives were explored by three aspects including additive content, load, and frequency.

3.2.1. Influences of additive content

Fig. 8 reveals the variation of COF and the wear widths on the disk lubricated by greases at 50 N, 5 Hz and ambient temperature. The nano-TiO_2 and nano-SiO_2 greases demonstrate better friction-reducing performance compared with other greases; which can be judged from the COF reducing from 0.108 to about 0.093 and 0.094 as the additives content increasing. When the additives content is 0.1 wt%, the nano-TiO_2 grease and nano-SiO_2 grease exhibit the lowest COF, which is about 14% lower than that of base grease. The slightly higher COFs of ATO grease than other greases might be resulting from degradation of mechanical characteristics occurred in insulating grease [28]. The wear widths of nano-TiO_2 greases reduce from 0.323 mm to 0.313 mm as the additives content increasing from 0 to 0.07 wt%, and rise slightly at 0.1 wt%. The smallest wear width at 0.07 wt% indicates that nano-TiO_2 can mostly upgrade the anti-wear characteristic of the base grease at this content. In the case of micro-TiO_2, nano-SiO_2 and ATO, the wear widths increase slightly at 0.05 wt% content compared with base grease, and increase much more at 0.07 wt% content. In terms of micro-SiO_2, though the wear widths reduce at 0.07 wt% content compared with 0.05 wt% content, the wear width is still larger than base grease. This might be caused by the agglomeration of additives especially at 0.07 wt% content. The agglomerated additives act as abrasive particles, and lead to the growing of wear widths.

In the case of microparticles, due to their large size, some oil would be expelled out of the lubrication area. And for their larger size (1 μm) than boundary film thickness (<50 nm [29]), they would act as abrasive particles throughout the sliding course and lead to the higher COFs.

3.2.2. Influences of load

Fig. 9 presents mean COF and wear width of the insulating greases, which contain 0.1 wt% additive, at multiple loads, 5 Hz and ambient temperature. The results illustrated that the mean COFs of all additive contained greases are lower than those of base grease, and reduce as the load growing, to some extent. And the mean COFs of all nano-SiO_2 greases (approximately 0.093–0.091) are much smaller than those of other greases, and reduce with increasing of the load. It is indicated that nano-SiO_2 particles are capable of performing better friction-reducing performance than the other four additives at all loads. The wear width of nano-TiO_2 grease is still relatively lower than those of other greases under the examination condition, implying the best anti-wear capacity among the greases.

3.2.3. Influences of frequency

Fig. 10 exposed the mean COF and the wear width of the 0.1 wt % additive contained greases at multiple frequencies. The mean COFs of the nano-SiO_2 grease decline with frequency increasing, and are smaller than those of other greases. The COF of nano-SiO_2 grease can be nearly reduced by 15% compared with the base grease at the condition of 100 N and 5 Hz; hence nanometer SiO_2 particles performed well friction-reducing characteristic. The wear widths results visibly demonstrated that the nano-TiO_2 grease possesses much better anti-wear capacity than others at whole frequencies throughout the whole testing time.

3.3. Surface analysis

For exploring the lubricating mechanism throughout the frictional process, SEM and EDS were applied. Fig. 11 exposes high magnification images of the scratches features on disks lubricated at 100 N, 5 Hz and ambient temperature. The images clearly disclose that the scratches of base grease and ATO grease (Fig. 11a and b) have many

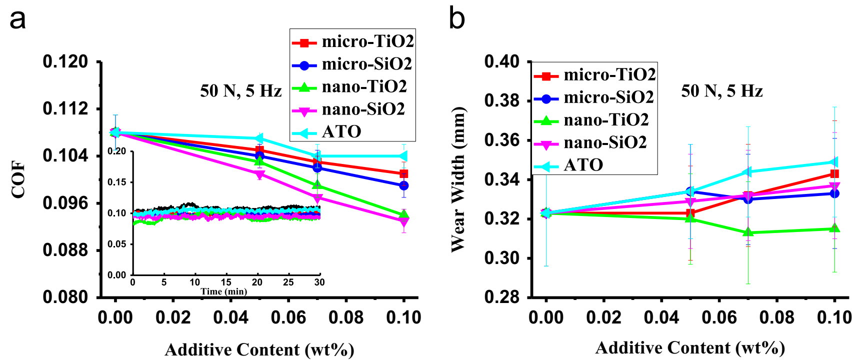

Fig. 8. (a) Average COFs and (b) wear widths for the greases at different additives contents at ambient temperature (load = 50 N; frequency = 5 Hz; stroke = 5 mm; duration = 30 min).

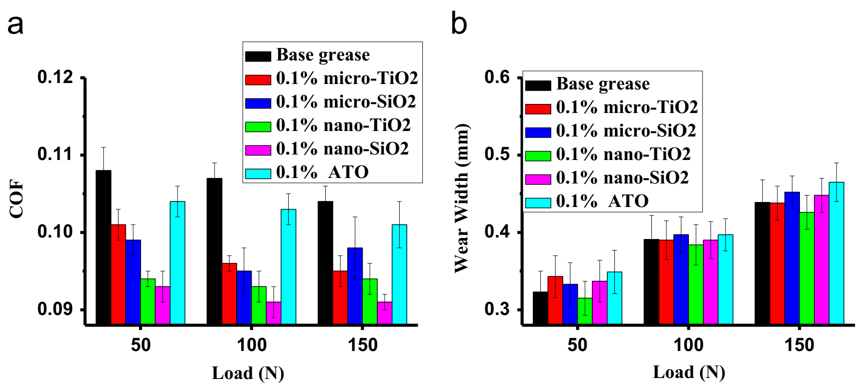

Fig. 9. (a) Average COFs and (b) wear widths at 50, 100 and 150 N for the greases at ambient temperature (frequency = 5 Hz; stroke = 5 mm; duration = 30 min).

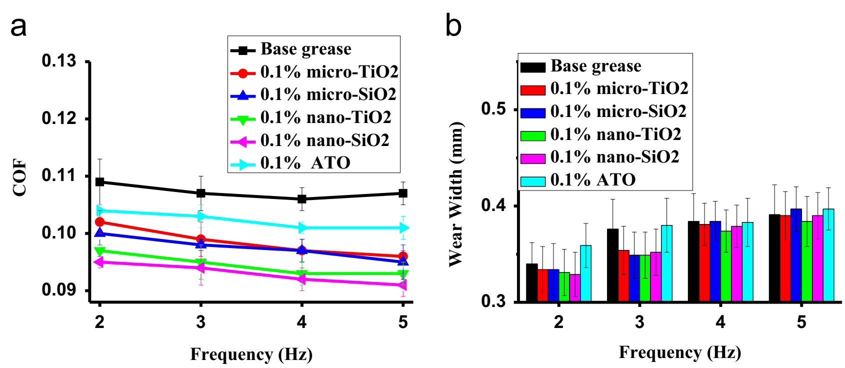

Fig. 10. (a) Average COFs and (b) wear widths at 2, 3, 4 and 5 Hz for the greases at ambient temperature (load = 100 N; stroke = 5 mm; duration = 30 min).

pits, indicating that many materials transferred and adhesive wear occurred in this instance. The adhesive wear appears when cracks developed below the surface and spread to the surface under incessant loading situations. These cracks extend to the surface and cause surface mutilation. Throughout the frictional process, the misplacements accumulate at a short distance below the surface. When the frictional process keep on going, vacuums are developed at the accumulate section. Vacuums gather together and finally turn into a crack, which would spread to the surface parallel and damage the surface eventually [30].

The worn steel surface lubricated by 0.1 wt% nano-TiO_2 grease (Fig. 11c) shows much smoother, and only slight adhesive wear; also the nano-TiO_2 particles have some polishing effect [30], which denotes to better tribological characteristics. The scratch lubricated by 0.1 wt% nano-SiO_2 grease (Fig. 11d) shows little furrows, which contributes to the better friction-reducing characteristics.

EDS examination was engaged to further illuminate the chemical component on the scratches. Fig. 12 provides EDS spectrums of some typical chemical elements on the scratches, which were lubricated by prepared insulating grease containing 0.1 wt% additives at the condition of 100 N and 5 Hz.

Crests of Si and Ti are low as shown in Fig. 12c and d, and these are ascribed to the small additives content in insulating grease (which is only 0.1 wt%). Still, the element content validates the existence of Si (0.67 wt%) and Ti (0.43 wt%), respectively. It is presumed that the Ti and Si deposited on the surface or metallic oxidized to form a protect film throughout frictional process. The protect film considerably donates to the tribological characteristics of the insulting grease. Consequently, the nano-TiO_2 exhibits outstanding tribological characteristics; and the nano-SiO_2 exhibits good friction-reducing characteristic.

Fig. 13 is the schematic of friction and wear mechanisms, and the dispersal of nanoparticles in insulating grease. The enhancement in tribological characteristics of nano-TiO_2 and nano-SiO_2 can be explicated by following aspects. Firstly, grease can uniformly settled down between the contact surfaces. Nanoparticles

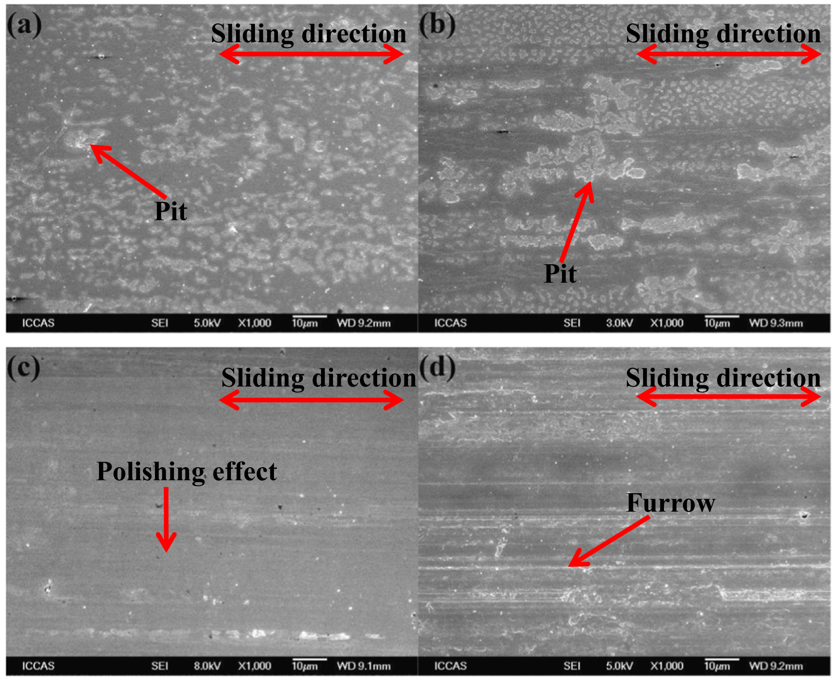

Fig. 11. High magnification SEM morphologies of the worn surfaces lubricated by the greases at 100 N and 5 Hz. (a) Base grease, (b) ATO grease, (c) nano-TiO_2 grease, (d) nano-SiO_2 grease.

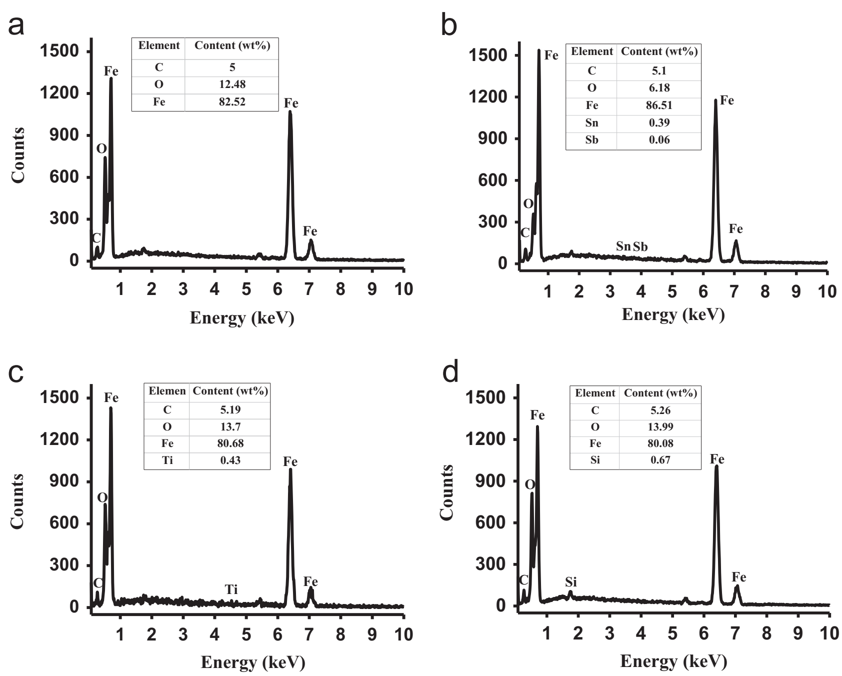

Fig. 12. EDS of the worn surfaces lubricated by the greases at 100 N and 5 Hz. (a) Base grease, (b) ATO grease, (c) nano-TiO_2 grease, (d) nano-SiO_2 grease.

in grease can fill in the valley of surface, increasing contact area and performing like spacers throughout frictional process, avoiding direct contact between the contact interfaces [31,32]. Secondly, nanoparticles also have rolling effect, which means nanoparticles would roll instead of slide between the contact interfaces [33–36]. In this way nanoparticles reduce the shear stress, and lead to the reducing in COFs. Thirdly, throughout the sliding process, the additives would sedimentation at the contact area due to gravity effect. On account of oleophilic property and high specific surface area for their small size, nanoparticles can act as centers, adsorbing oil to generate a tightly-structured network [37,38], in which way makes more oil participate in the lubrication process, and contributes to improved anti-wear property. However, in the case of microparticles, due to their smaller specific surface for the larger size than nanoparticles, some oil would be expelled out of the lubrication area, lead to less oil participating in lubrication. And also for their larger size (1 μm) than boundary film thickness (<50 nm [29]), they would act as abrasive particle throughout the sliding course and lead to the higher COFs. Furthermore, Ti and Si ions incorporated into the surface film throughout sliding process and enhanced the film. Consequently, the COF and the wear width kept steady and slim at ambient temperature.

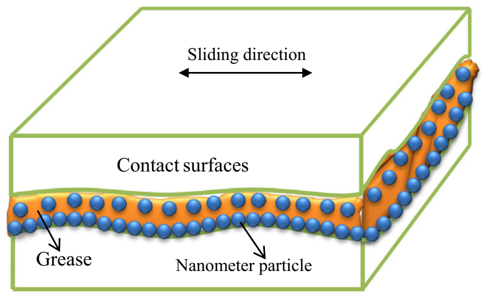

Fig. 13. Schematic of friction mechanism of the nanometer particles in grease.

Additionally, stray current exists in many electrical apparatus, it can cause electrochemical corrosion and hence increase the friction and wear between the contact surfaces [39,40]. Methods to protect electrical apparatus from stray current corrosion is increasing the insulating capacity of the contact surfaces [41–43]. Insulating grease is a material that applied between the contact surfaces; it can increase the insulating capacity between the interfaces and reduce the corrosion caused by stray current. To sum up, while applied to some electrical apparatus, the grease insulating capacity can improve the friction-reducing and anti-wear properties of the grease to some extent.

4. Conclusions

Nano-TiO_2 and nano-SiO_2, two new kinds of additives, possess a superior insulating characteristic (high volume resistivity) in grease. These insulating greases can be applied to the electrical apparatus. The greases prepared using nano-TiO_2 exhibit good tribological characteristics; and the greases prepared using nano-SiO_2 display good friction-reducing characteristic. EDS analysis reveals that Ti and Si deposited or metallic oxidized on the surface throughout the frictional process forming a protect film. The protect film considerably donates to the tribological characteristics of the greases prepared using nano-TiO_2 and nano-SiO_2.

Acknowledgments

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