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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 24×7.9 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

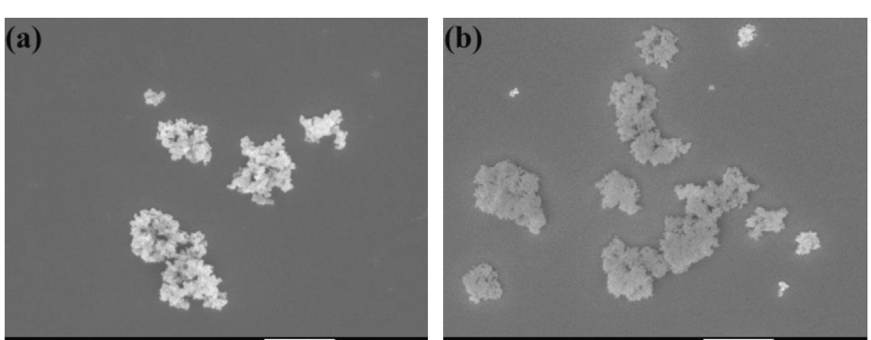

Fig. 1. Scanning electron microscope images of the (a) nano-TiO_2 and (b) nano-SiO_2.