Effect of ionic liquids modified nano-TiO$_2$ as additive on tribological properties of silicone grease

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Abstract

A series of silicon greases were fabricated by using polytetrafluoroethylene as thickening agent and polydimethylsiloxane as base oil. Four kinds of ionic liquids modified nano-titanium dioxide were employed as solid additives with different concentrations of 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 wt%. The chemical composition and thermal stability of the additives were explored by a Fourier transform infrared spectroscopy and a thermal gravimetric analyzer at a heating rate of 10 °C min$^{-1}$ under a flow of nitrogen, respectively. The volume resistivity of greases was measured by a volume surface resistance tester in the room temperature of 25 °C. The tribological properties were investigated using a ball-on-disc tribometer under a stroke of 5 mm, a frequency of 5 Hz and different applied loads of 50, 100, 150 and 200 N. After tribological tests, a scanning electron microscopy and x-ray photoelectron spectroscopy were used to probe the lubrication mechanisms. Results showed that nano-titanium dioxide were successfully modified by ionic liquids and their decomposition temperatures were all higher than 300 °C. Meanwhile, the addition of additives had a significant influence on the conductivity and tribological properties of the greases. Specially, the grease containing 1.5 wt% additives exhibited good friction reduction and anti-wear abilities. The analysis of the worn surfaces indicated that the reasons for raising the anti-friction and anti-wear abilities were attributed to the synergistic effects of ionic liquids and solid additives which promoted the formation of lubricating films on the worn surfaces.

1. Introduction

Conductive grease has been widely used in contact joints of electrical connectors under current carrying conditions, which was responsible for reducing power loss, transmitting power and electrical signal. Electromechanical equipment was different from the traditional application conditions, in addition to the good friction reduction and anti-wear abilities, it requires lubricants possess good conductivity [1–3]. Traditional lubricants could from lubricating protective films on the worn surface to exhibit good anti-friction and anti-wear abilities by complex physical interactions and chemical reactions. However, most of the lubricating films generally possess poor conductivity, which undoubtedly impede the flow of electricity for electromechanical equipment [4, 5]. Therefore, it is necessary to explore new lubricants with good conductivity and tribological performances to meet the special lubricating requirements of the electromechanical equipment.

Silicone oil is an ideal base oil of commercial electric grease with strong insulation [6, 7]. However, it has a low conductivity which restricts the applications. In order to increase the conductivity of silicon grease, many approaches have been investigated, especially using of nano-sized inorganic particles as addictive [8]. Xia et al prepared a series of heat-conducting silicone by using CNTs/BN and AlN as solid additives [9, 10]. Zhang et al evaluated the thixotropy effects of nano-SiO$_2$ composite grease fluids [11]. Nano-TiO$_2$ is also an ideal addictive because of the low price, non toxicity, good chemical stability, light transmittance and semiconductor properties.
Colorless and transparent liquid

Table 1. Main parameters of polydimethylsiloxane (PTFE).

| Appearance       | Viscosity (25 °C)/(mm²·s⁻¹) | Refractive index (25 °C) | Flash point (opening)/ °C | Density (25 °C)/g cm⁻³ | freez- ing point/°C |
|------------------|------------------------------|--------------------------|---------------------------|-------------------------|---------------------|
| Colorless        | 100                          | 1.403                    | 326                       | 0.964                   | −55                 |

Table 2. Main parameters of nano-TiO₂.

| External diameter (nm) | Length (nm) | purity   | Specific surface area (m² g⁻¹) | Conductivity (Ω cm⁻¹) |
|------------------------|-------------|----------|--------------------------------|-----------------------|
| 20–30                  | 10–30       | >98%     | >100                           | >100                  |

[12, 13]. Ge et al has added the nano-TiO₂ into the lubricating grease and found the grease exhibited good anti-friction and anti-wear properties. However, the conductivity of the grease was reduced at the same time [14].

Ionic liquids (ILs) is a kind of the most promising materials, which possess good conductivity, outstanding lubricating ability and good thermal stability and so forth [5]. ILs has been widely used in many fields, such as catalysis, capacitor, battery, green solvent as organic reaction and nano material preparation [15]. Imidazole ILs has excellent anti-pressure ability, anti-wear and anti-friction properties. However, there is few literatures about the introduction of nano-TiO₂ modified by imidazole ILs into lubricating grease, and there is little knowledge about the influence on the conductivity and tribological performances of silicone oil.

In this work, four kinds of imidazole ILs modified nano-TiO₂ with different concentrations of 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 wt% were employed as solid additives to fabricate lubricating grease. The chemical composition and thermal stability of the additives were explored by a Fourier transform infrared spectroscopy and a thermal gravimetric analyzer at a heating rate of 10 °C min⁻¹ under a flow of nitrogen, respectively. The volume resistivity of greases was measured by a volume surface resistance tester in the room temperature of 25 °C. The anti-friction and anti-wear performances of grease were investigated using a ball-on-disc tribometer under a stroke of 5 mm, a frequency of 5 Hz and different applied loads of 50, 100, 150 and 200 N. Due to the most of moving parts of electromechanical equipment were made of AISI 52100 steel. Therefore, this kind of ball-on-disc configuration which was made of AISI 52100 steel was chosen. After friction test, the mechanism was analyzed by a scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS).

2. Experimental details

2.1. Materials

The basic parameters of silicone oil are listed in table 1. Polytetrafluoroethylene (PTFE) is used as thickener. Nano-TiO₂ is purchased from Maclin Reagent company, and its basic parameters are listed in table 2.

Four kinds of ILs were purchased from the Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, and their chemical molecular structures are shown in table 3.

2.2. Preparation of additive and lubricating grease

The preparation of four kinds of additives including [PnMIm][PF₆]-TiO₂, [C₁₂mim][PF₆]-TiO₂, [PnMIm][NTf₂]-TiO₂ and [C₁₂mim][NTf₂]-TiO₂ were as following procedures. (1) A certain amount of nano-TiO₂ and ILs were dispersed in anhydrous acetone by ultrasonic wave for 5 min. (2) The mixtures was grinded for 4 h at room temperature. (3) The mixture was dried at 80 °C in an oven for 24 h to obtain. The ratio of nano-TiO₂ to ILs is 9:1 [16].

The preparation process of lubricating grease is as following procedures: (1) A certain amount of additive was added into the silicone oil and the mixture was mechanically stirred for 15–30 min (2) PTFE (30 wt%) as thickener and n-hexane as dispersant was added into the mixture of the additive and silicone oil. (3) The mixture was continuing stirred for 30–60 min, and then the temperature was raised to 80 °C for 30 min to remove n-hexane. (4) The temperature of the mixture was cooled to room temperature and was grinded on a three roll grinder for three times to obtain the greases. The mass fraction of additives was 0.5%, 1.0%, 1.5%, 2.0%, 2.5%, 3.0% respectively.

2.3. Test experiment

According to the national electric power industry standard DL/T 373-2010, the volume resistivity of the conductive grease was measured by a GEST-121 volume resistivity tester which produced by Beijing Guanshi Jingdian instrument and Equipment Co., Ltd. The test current is 100 A and the test time is 10 s in room.
The volume resistivity is calculated as follows:

\[
\rho = R \cdot \frac{A}{h}
\]

In the above formula: \(\rho (\Omega \cdot \text{cm})\) is the volume resistivity of the grease; \(R (\Omega)\) is the volume resistance; \(A (\text{cm}^2)\) is the contact area; \(h (\text{cm})\) is the average thickness of the sample.

The chemical composition of the additives was explored by Fourier transform infrared spectroscopy (FT-IR, Thermo Fisher Scientific). The tested wavenumber ranged from 400–3200 cm\(^{-1}\). The thermal stability of the additives were explored by a thermal gravimetric analyzer (TGA, TA Instrument) at a heating rate of 10 °C min\(^{-1}\) under a flow of nitrogen.

The tribological behaviors of lubricants were conducted on an MFT-R4000 reciprocal tribometer with a ball-on-disc configuration. Figure 1 gives a ball-on-disc contact model. The steel ball (diameter: 5 mm) and the lower disc (Φ24 mm × 7.5 mm) were all made of AISI 52100 steel and the hardness were all about 750 Hv. Due to the limited ability of reciprocal tribometer, the upper ball moved against the fixed disc under an applied normal load of 50, 100, 150, 200 N and at a frequency of 5 Hz and a stroke of 5 mm. The disc has a surface roughness of about 0.05 um after polishing treatment. All the tests were conducted under a temperature

![Figure 1. Ball-on-disc contact model.](image)
of 25 °C and a relative humidity of 30%. To get a more reliable test data, every test was repeated for three times under the same experiment condition and the average value with the standard deviation were given.

After anti-friction and anti-wear test, the discs were ultrasonically cleansed for 10 min by acetone. The pictures of the worn surfaces were taken by a scanning electron microscopy (SEM, Thermo Fisher Scientific). The chemical states of the characteristic elements were probed by an x-ray photoelectron spectroscopy (XPS, Thermo Fisher Scientific) with an Al-Kα radiation and the reference of carbon (284.6 eV).

3. Results

3.1. Characteristics of additives

The FTIR spectra of ILs modified nano-TiO2 was shown in figure 2a. The PF6–sharp peaks of [PnMIm][PF6]+TiO2 and [C12mim][PF6]+TiO2 are located at 839 cm\(^{-1}\) [5]. The C-N bands of imidazole rings exhibited obvious peaks at 1565 and 1473 cm\(^{-1}\) [17–19]. The C=N stretch vibrations exhibited peaks at 2960 cm\(^{-1}\) [20]. These FT-IR results indicated a successful modification of TiO2 with functional groups from imidazole ILs. Figure 2b lists the TGA of the ILs modified nano-TiO2 which showed that all the decomposition temperatures of ILs modified nano-TiO2 are higher than 300 °C, indicating all the additives exhibited good thermal stability. In some extreme conditions, the highest temperature of electrical contact could reach 150 °C [21, 22]. Herein, all the additives have decomposition temperatures than 300 °C, which could meet the special requirement.

3.2. Conductive behaviors of the lubricants

The volume resistivity of grease is given in table 4, it can be found that the volume resistivity of the grease increased gradually with the increasing concentration of nano-TiO2. The maximum volume resistivity was reached at the mass fraction of nano-TiO2 in the base grease of 1.5 wt\%, which was about 2.2 times than that of
the base grease. Nano-TiO₂ could provide a high concentration of electron trap to improve the insulating ability of insulating grease \[23, 24\]. The results showed that nano-TiO₂ is a kind of insulating agent, which can reduce the electrical conductivity of grease.

As shown in table 4, the volume resistivity of greases was greatly reduced by using nano-TiO₂ modified by ILs as additives, illustrating that the conductive ionic liquids can greatly improve the conductivity of nano-TiO₂. In addition, the volume resistivity of nano-TiO₂ modified by [NTf₂] was lower than that of nano-TiO₂ modified by [PF₆] when the cation is the same. Meanwhile, when the anions are the same, the volume resistivity of grease increased with the increasing length of carbon chain of cationic alkyl. The reason may be lie in that, when the anion is the same, the van der waals force and the association decreased, the conductivity increased with the increasing length of the cation chain. However, for ILs with the same cation, the smaller anion has more dispersed charge, leading to a weaker hydrogen bond and association with cation, and then resulting in a higher conductivity \[15\].

**Figure 3.** Coefficient of friction (a) and scratch widths (b) of grease under 50 N at a stroke of 5 mm, a frequency of 5 Hz and 25 °C.

**Figure 4.** Coefficient of friction (a) and scratch widths (b) of grease containing 1.5 wt% additive under 50, 100, 150 and 200 N at a stroke of 5 mm, a frequency of 5 Hz and 25 °C.

| Additives              | Volume resistivity (×10¹¹Ω·cm) |
|------------------------|---------------------------------|
| TiO₂                   | 3.1 4.33 6.25 6.69 5.97 5.77 6.15 |
| [PhMIm][PF₆] + TiO₂    | 3.1 0.89 0.92 0.93 0.99 1.11 1.11 |
| [C₁₂mim][PF₆] + TiO₂   | 3.1 1.07 1.11 1.23 1.27 1.35 1.71 |
| [PhMIm][NTf₂] + TiO₂   | 3.1 0.61 0.70 0.99 0.97 1.12 1.61 |
| [C₁₂mim][NTf₂] + TiO₂  | 3.1 1.11 1.21 1.22 1.23 1.21 1.77 |
Figure 5. SEM morphologies of the worn surfaces on the steel discs under a normal load of 200 N and a mass of 1.5% at 5 Hz. (a) nano-TiO$_2$, (b) [PnMIm][PF$_6$]+TiO$_2$, (c) [C$_{12}$mim][PF$_6$]+TiO$_2$, (d) [PnMIm][NTf$_2$]+TiO$_2$, (e) [C$_{12}$mim][NTf$_2$]+TiO$_2$. 
3.3. Tribological behaviors of the lubricants

In general, high concentration of solid additive may lead to abrasive wear while low concentration of solid additive could not form effective enough lubricating film. Therefore, the tribological properties of the greases with different additive concentrations were investigated under 50 N at a stroke of 5 mm, a frequency of 5 Hz and 25 °C, and figure 3 shows the coefficient of friction (COF) and scratch widths. It can be seen that the five kinds of additives can reduce the COF and scratch widths. With the increase of additive concentrations, the COF and scratch widths of the greases all first decreases and then increases. The optimum additive concentration is about 1.5 wt%, and the COF and scratch widths of the grease reaches the minimum. Figure 4 shows the COF and scratch widths of the test under different loads of 50, 100, 150 and 200 N at a stroke of 5 mm, a frequency of 5 Hz and 25 °C when the additive concentration is 1.5 wt%. It can be seen from the figure 4 that with the increase of load, the COF and widths of wear scar increases gradually. When the cations are the same, [NTf₂] shows better
anti-friction and anti-wear performances. Besides, when the anions are the same, with the cationic carbon chain growing, the tribological properties of the additives became better. This is due to the good compatibility of base oil which make it easy to enter the friction surface [25].

3.4. Analysis of the scratch surface
To further compare the tribological properties, the SEM morphologies of the scratch surfaces was showed in figure 5. The scratch surface (figure 5(a)) lubricated by silicone grease containing nano-TiO₂ was relatively wide and there was a lot of spalling and deep grooves, implying a severe wear appearing. When the friction pair was lubricated by silicone grease containing nano-TiO₂ modified by ILs (figures 5(b)–(e)), not only the scratch width was reduced, but also the scratch surface became smoother, implying nano-TiO₂ modified by ILs improved the anti-wear ability of friction pair. As shown in figures 5(b)–(e), the scratch surface appeared polishing phenomenon under the lubrication of [C₁₂mim][PF₆] and [C₁₂mim][NTf₂], indicating that the ILs with long carbon chain had a better anti-wear performance. The peeling and furrow of the scratch surface under the lubrication of [NTf₂] are lower.

![Figure 8. Elemental energy spectrum of wear scar surface under 200 N.](image)

Table 5. High-resolution peak separation results of Fe 2p on the worn surface.

| Peak Binding energy(eV) | Half peak width(eV) | Peak intensity |
|-------------------------|---------------------|----------------|
| [PnMim][PF₆] + TiO₂     |                     |                |
| Fe0                     | 706.9               | 563.9271       |
| Fe₂⁺                   | 711                 | 4922.709       |
| Fe₃⁺                   | 712.8               | 12 250.57      |
| [C₁₂mim][PF₆] + TiO₂    |                     |                |
| Fe0                     | 907                 | 1274.848       |
| Fe₂⁺                   | 710.8               | 6122.973       |
| Fe₃⁺                   | 712.9               | 4257.686       |
| [PnMim][NTf₂] + TiO₂    |                     |                |
| Fe0                     | 706.8               | 1770.839       |
| Fe₂⁺                   | 710.7               | 14 483.83      |
| Fe₃⁺                   | 713                 | 11 855.83      |
| [C₁₂mim][NTf₂] + TiO₂   |                     |                |
| Fe0                     | 706.9               | 908.9329       |
| Fe₂⁺                   | 710.8               | 9923.794       |
| Fe₃⁺                   | 713.4               | 14 312.45      |
than that of [PF₆] (figures 5(b), (d) and (c), (e)), indicating that [NTf₂] had better anti-wear performance than [PF₆], and the reason for this phenomenon was lie in that PF₆ anion could cause serious corrosion, which may lead to a higher corrosion wear [26–28].

To understand the lubrication mechanisms, the chemical states of the elements including O, C, Si, Fe, Ti, F and N were detected by an XPS. Figure 6 shows the XPS spectrum of O 1s (528 – 536 eV), C 1s (282 – 290 eV), Si 2p (99 – 105 eV), Fe 2p (703 – 734 eV), Ti 2p (457 – 459 eV), F 1s (684 – 686 eV), N 1s (398 – 402 eV). The elements of N, Ti and F in the sample come from the additives, which indicates that the additives were attached to the scratch surfaces. Besides, a strong peak of Fe appeared on the scratch surfaces in the process of friction.

Figure 7 and table 5 showed the results of Fe 2p peaks on the scratch surfaces. It can be seen that there was a certain amount of Fe(0), Fe(II) and Fe(III) on the worn surfaces lubricated different greases. However, the composition of three kinds of Fe was slightly different under different grease lubrication. Fe₂p exhibited obvious peak at about 710.5 – 713.4 eV, which implied that complex iron oxides were generated on the scratch surfaces to raise the anti-wear ability of the friction pair.

Figure 8 shows high-resolution spectrum of the O 1s, N 1s, F 1s, Si 2p. The peak of O 1s at 530 – 532 eV indicates that complex oxides are formed on the scratch surfaces [1, 29]. The XPS spectra of O 1s has the peak at 530 eV and 532 eV, which were assigned to Fe-O. Combining the XPS spectra shown in figure 7, it could be inferred that the lubricating films composed of complex iron oxides were indeed formed on the scratch surfaces to raise the anti-wear ability of the friction pair [30–32]. The binding energy of F 1s coming from ILs is at 689.1 eV, corresponding to organic fluorine oxides. Combined with the spectrum of Fe, it shows that FeF₃ or FeF₂ are formed [7]. Si is the constituent elements of silicone oil and the peaks is located at about 102.5 eV belonging to Si-C, indicating the existence of silicone oil on the friction surface. The N 1s coming from ILs has an obvious peak of C-N bond at 402.2 eV, which suggests the oxide of organic amines and nitrogen were formed on the scratch surfaces [33]. The XPS analysis implies that complex physical and chemical reactions appeared on the scratch surfaces to form a lubricating film composed of such as Fe₂O₃, Fe₂O₄, FeF₃, FeF₂ and so on to raise the tribological performances of the friction pair.

Figure 9 shows the XPS spectrum of Ti 2p on the scratch surfaces. The peaks of Ti 2p₃/₂ and Ti 2p₁/₂ are located at 458.4 eV and 464.5 eV, respectively. The binding energy between 457.0 and 459.1 eV was belonged to the Ti 2p₃/₂ peak of TiO₂ [34, 35]. Meanwhile, when the cations are the same, the peaks of Ti 2p₁/₂ and Ti 2p₃/₂ on the scratch surface are stronger under the lubrication of [NTf₂] grease than that of [PF₆] grease, indicating that more TiO₂ is deposited on the scratch surface.

The above conducted tribological tests shows that TiO₂ and TiO₂ modified by ILs could exhibit good anti-friction and anti-wear abilities in silicone grease. Combining the SEM and XPS analysis, the reason could be concluded as following three aspects. (1) It has been reported that nanomaterial could fill the valley of the contact friction pair to reduce the surface roughness and act as a spacer to reduce the direct contact between friction interfaces. Meanwhile, when the contact interfaces move against each other, the nanomaterial could roll instead of slide. These lubrication mechanisms could promote TiO₂ to reduce the shear force of the friction pair, thereby raising the anti-friction and anti-wear abilities of the friction pairs [2, 14]. (2) The XPS analysis shown in figure 9 suggests that TiO₂ could deposit on the scratch surfaces to form an effective lubricating film to enhance the anti-wear performances. (3) During the friction process, the scratch surface was easily to be oxidized to form a protective lubricating films which was proved by figures 7 and 8(a) [31, 32, 36].
The tribological test showed that TiO$_2$ modified by ILs could exhibit better anti-friction and anti-wear abilities in silicone grease than unmodified TiO$_2$. The reason may be attributed to another two aspects. (1) Dispersive ability has an important influence on the tribological properties of lubricants. The related research showed that nanomaterials could be modified by ILs to improve the dispersive ability in lubricants, thereby improving the tribological properties [15, 19, 20]. The FT-IR analysis suggests the TiO$_2$ has been successfully modified by ILs, therefore, they exhibited better dispersive ability and friction reduction and anti-wear abilities in lubricating grease than unmodified TiO$_2$. (2) It was known that ILs exhibit excellent tribological properties [15, 25]. The TiO$_2$ was successfully modified by ILs through physical and chemical interactions, which also implies that ILs and/or functional group of ILs existed on the TiO$_2$ modified by ILs. When it was used as additive, the ILs and/or functional group of ILs could also form lubricating films on the scratch surface. This lubrication mechanism was also proved by the XPS analysis of F1s and N1s coming from ILs shown in figures 8(b) and (d).

As for the difference on tribological properties among different ILs modified TiO$_2$, the reason was mainly related to the different cations and anions. When the anion was same, the ILs containing [C$_{12}$mim] cation could form a better protective lubricating film because it has a longer carbon chain than [PnMIm] cation. When the cation was same, the ILs containing [NTf$_2$] anion exhibited better anti-wear performance than ILs containing [PF$_6$] anion because of the serious corrosion caused by [PF$_6$] which has been proved by some related research [26, 28].

4. Conclusions

In this study, four kinds of ILs including [PnMIm][PF$_6$], [C$_{12}$mim][PF$_6$], [PnMIm][NTf$_2$], [C$_{12}$mim][NTf$_2$] modified nano-TiO$_2$ were used as additives in the silicone grease. The conductive and tribological behaviors of the four grease additives were investigated in detail. The lubrication mechanisms were probed by SEM and XPS. The main conclusions are as follows:

1. The nano-TiO$_2$ modified by ILs can reduce the volume resistivity of silicone grease, and the conductive effect is related with the length of substituent carbon chain in ILs. The volume resistivity of nano-TiO$_2$ modified by ILs containing [NTf$_2$] anion is lower than that of nano-TiO$_2$ modified by ILs containing [PF$_6$] anion.

2. All the additives have the decomposition temperatures were all higher than 300 °C, indicating a good thermal stability.

3. Nano-TiO$_2$ modified by ILs can improve the anti-friction and anti-wear abilities of silicone grease. The good tribological properties are attributed to the synergistic effects of ionic liquids and solid additives.

4. Nano-TiO$_2$ modified by ILs containing [NTf$_2$] anion has better friction reduction and wear resistance, and the optimal mass fraction is 1.5 wt%.

5. Complex physical and chemical interactions occurred on the scratch surface to form a protective film composed of Fe$_2$O$_3$, Fe$_3$O$_4$, FeF$_3$, FeF$_2$ and so on, which enhanced the tribological properties of the friction pair.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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