Investigation on the electrical conductivity and tribological properties of NbSe$_2$-doped lubricating grease

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Abstract
Developing lubricants with good electrical conductivity and good tribological properties is necessary for the power equipment. Here, niobium selenide (NbSe$_2$) and boron nitride (BN) were employed to act as additives to fabricate the lubricating greases with superior electrical conductivity and tribological properties. The lubricating grease containing different concentrations of additives were synthesized and their conductivities were measured by a volume resistance meter at the room temperature of about 25 °C. The tribological properties of the lubricating greases were also investigated and the worn surfaces were characterized in detail by scanning electron microscopy (SEM), x-ray photoelectron spectroscopy (XPS) and Raman spectroscopy to analyze the lubrication mechanism after friction test. The results showed that NbSe$_2$ could effectively reduce the volume resistivity by ten times as compared with the base grease. Tribological tests showed that when the concentration of NbSe$_2$ was 2 wt%, it could reduce the COF and wear scar width by 23.5% and 12.8% under 150 N and 5 Hz, indicating 2 wt% NbSe$_2$ doped lubricating grease exhibited the outstanding tribological properties. In addition, based on the analysis of the wear surfaces, the superior tribological properties of NbSe$_2$ grease were attributed to the effective lubricating film generated on the friction surface, which played a key role in reducing friction and anti-wear.

1. Introduction

Friction usually takes place at the contact interfaces when two physical objects move relative to each other and wear is the result of friction [1–3]. Wear could lead to deterioration of the contact surface such as vibration, noise, and even failure of mechanical equipment. Therefore, controlling friction behavior and reducing wear have been paid close attention by academia and industry [4–7].

Lubricant is a kind of material which can effectively enhance the friction reducing and anti-wear properties of friction pair. In ancient times, lubricant started modestly with fluids like animal oil, vegetable oil, water, and so forth. As different lubricant has been developed over the years, petroleum-based lubricant has been widely employed because of the superior lubricating properties. Lubricant could promote a protective lubricating film generated on the contact surface, which can separates the friction surface and reduces wear and energy loss [8–10].

However, with the rapid development of mechanical instruments, the lubricant used in the special fields should possess some special properties to meet the requirement. For example, aerospace equipment requires lubricant have good radiation resistance and thermal stability [11–13]. The lubricants used on ship should have good non-toxic and biodegradable properties [14–16]. Lubricants used for high-temperature equipment need to exhibit good tribological properties under high-temperature conditions [17, 18].

In this paper, our attention is mainly focused on the electrical devices such as switch, parallel slot clamp and so on. When these electrical devices need to be lubricated, not only the lubricant is required to have good friction reducing and wear resistance properties, but also the lubricant should possess good electrical conductivity.
Table 1. Typical base parameters of silicone oil.

| Appearance          | Viscosity (25 °C)/(m·s⁻¹) | Index (25 °C) | Flash point/°C | Density (25 °C)/(g·cm⁻³) |
|---------------------|-----------------------------|---------------|----------------|--------------------------|
| Colorless and       | 100                         | 1.403         | 326            | 0.964                    |
| transparent liquid  |                             |               |                |                          |

[19–22]. Although traditional lubricants have good lubricating properties, their conductivity is poor. These lubricants usually are dependent on the physical adsorption and chemical reaction protective films on the wear surface to achieve friction reducing and anti-wear effects. However, these lubricating protective films usually have poor electrical conductivity, which increases the contact resistance [23, 24]. Therefore, developing lubricants with good electrical conductivity and good tribological properties is necessary for the power equipment.

Niobium selenide (NbSe₂) is a typical layered crystal, which belongs to the close-packed hexagonal structure and has a layered structure similar to that of graphite. The interlayer non-metallic atoms are combined by the van der Waals force. The metal atoms and non-metallic atoms in the layer are connected by covalent bonds. The interlayer has weak shear stress, and this structure makes it easy to slide between layers [25, 26]. Meanwhile, it has been reported that NbSe₂ has excellent electrical conductivity [27, 28]. Therefore, given the consideration of the unique microstructure, mechanical and electrical properties, NbSe₂ has become a research hotspot in recent years. However, there is few literature about the influence of NbSe₂ on the electrical conductivity and tribological performances of lubricating grease.

In this study, the NbSe₂ is employed as the additive to regulate the electrical conductivity and tribological properties of the lubricating grease and the boron nitride (BN) is used as the contrastive additive. A series of lubricating greases were prepared and their electrical conductivity and tribological behaviors were investigated in detail. After test, the scanning electron microscopy (SEM), x-ray photoelectron spectroscopy (XPS) and Raman spectroscopy were also employed to analyze the related mechanisms.

2. Experimental details

2.1. Materials

Boron nitride (BN) and niobium selenide (NbSe₂) are used as additives and they are both purchased from Sinopharm Group Reagent Co., Ltd. Silicone oil is used as base oil and Poly tetrafluoroethylene (PTFE) is used as thickener. Table 1 lists the typical basic parameters of silicone oil. All the reagents used in the experiment are all analytically pure without secondary treatment.

2.2. Preparation of lubricating grease

According to the previous report [29], the lubricating grease is prepared as following procedures: (1) Some amount of additive was added to the silicone oil and stir mechanically for 15–30 min (2) PTFE (30 wt%) as thickener and acetone as dispersant were added to the mixture of additives and silicone oil. (3) The mixture was continuously stirred for 30 min, and then heated to 85 °C for 30 min to remove acetone. (4) After that, the mixture was cooled to room temperature, and grinded three times on a three-roller mill to obtain the lubricating grease. The concentration of additives is a key factor affecting the performance of lubricants. Therefore, in order to obtain the optimal additive concentration, the concentration of additives was adjusted by 0.5 wt%, 1.0 wt%, 2.0 wt%, 3.0 wt% and 4.0 wt%, respectively. Figure 1 gives the actual photograph of the (a) base grease, (b) BN grease (2.0 wt%) and (c) NbSe₂ grease (2.0 wt%).

2.3. Test experiment

The volume resistivity of the prepared lubricating grease was characterized by a GEST-121 (Beijing Guanshi Jingdian Instrument Co., Ltd) tester based on the standard DL/T 373–2010. The test was conducted at room temperature and the current is 100 A. The volume resistivity is calculated as follows:

$$\rho = R \cdot \frac{A}{h}$$

Here, $\rho$ is the volume resistance; $h$ is the average thickness of the sample; $A$ is the contact area; $\rho$ is the volume resistivity of the grease.

The microstructure and crystal structure of NbSe₂ and BN were characterized by scanning electron microscope (SEM, Thermo Fisher Scientific) and x-ray diffraction (XRD, Bruker, Germany) with a copper tube (Ko, $\lambda = 1.54$ Å), respectively.
The tribological behaviors of lubricants were performed on an MFT-R4000 tribometer with a ball-on-disk configuration. Figure 2 gives the picture and diagrammatic sketch of the MFT-R4000 tribometer. The lower disc (Φ24 mm × 7.5 mm) and the upper ball (diameter: 5 mm) were all made of AISI 52100 steel and the hardness was about 750 Hv. Lubricating grease possessed different tribological properties under different loads and frequencies. Due to the limitation of the performance of the MFT-R4000 tribometer, the upper ball moved relatively to the lower disc under applied loads of 50, 100 and 150 N and a stroke of 5 mm. The frequency ranged from 2 to 5 Hz. The surface roughness of the polished disc was about 0.05 μm. Every tribological test was continued for 30 min. About 0.3 g of lubricating grease was introduced into the friction zone for every tribological test. The tests were conducted at room temperature and the relative humidity was about 30%. In order to get more reliable data, each test was performed 3 times under the same experimental conditions. After the tribological test, the disc was ultrasonically cleaned with acetone for 10 min. An optical microscopy (CX43, Olympus) was employed to obtain the wear scar width. A scanning electron microscopy (SEM, Thermo Fisher Scientific) was employed to get the pictures of the worn surfaces. Raman spectrometer (Renishaw, UK) a 514 nm laser excitation and an x-ray photoelectron spectroscopy (XPS, Thermo Fisher Scientific) with Al-κα radiation and a carbon reference (284.6 eV) were used to characterize the chemical states of the characteristic elements.

3. Results and analysis

3.1. Characteristics of additives and lubricating greases
Figure 3 gives the microstructure of NbSe2 and BN. It can be seen that NbSe2 and BN all have a typical hexagonal layered crystal structure. Figure 4 gives the XRD pattern of the NbSe2 and BN. It can be observed that the peak shape of the NbSe2 and BN were sharp, which indicates a high degree of crystallinity. Meanwhile, the half-peak width were small, which indicates a large grain size [30, 31]. The analysis result of the XRD were consistent with the microstructure shown in figure 2.
3.2. Conductive behaviors of the lubricants

Figure 5 shows the effect of additive concentration on the electrical conductivity of lubricating grease. It can be seen that since BN and NbSe\textsubscript{2} have different electrical conductivities. Therefore, the effect on the electrical conductivity of lubricating grease also exhibited different trends. With the increase of additive content, the volume resistivity of BN lubricating grease gradually increased, while the volume resistivity of NbSe\textsubscript{2} lubricating grease gradually decreased. When the addition amount was 2 wt\%, the volume resistivity of NbSe\textsubscript{2} lubricating grease dropped sharply. Especially when the addition was 4 wt\%, NbSe\textsubscript{2} could effectively reduce the volume resistivity by ten times as compared with the base grease. However, when the addition of boron nitride was 4 wt\%, the volume resistivity was 1.6 times of the base grease. The electrical conductivity of lubricating grease was closely related to the conductivity and concentration of the additive.

The electrical conductive properties of lubricating greases can often be explained by permeation theory \[32, 33\]. When additive particles with good electrical conductivity are added into the grease, they can contact with each other to form a conductive network. Moreover, with the increase of the additive concentration, a denser conductive network will be formed, thereby reducing the volume resistivity of the grease \[34\]. NbSe\textsubscript{2} has an excellent electrical conductivity, thus with the concentration increased, a dense conductive network could be formed and the volume resistivity of the grease gradually decreased.

The increase in the volume resistivity of lubricating grease is generally related to ‘electronic traps’. Electrons can polarize surrounding molecules in response to a local electric field, producing charged particles and forming directional motions. When the charged particle has moved a certain distance, the electrons will detach and reform new charged particles again and then continue moving. However, as the electrons move and develop new charged particles, the energy of the electrons gradually diminishes. Therefore, these charged particles can be called electron traps \[10, 35\]. BN has a poor conductivity. Therefore, the addition of BN could increase the amount of the electron traps, resulting in an increase in the volume resistivity of the grease.

**Figure 3.** Microstructure of (a) niobium diselenide (NbSe\textsubscript{2}) and (b) boron nitride (BN).

**Figure 4.** XRD pattern of (a) niobium diselenide (NbSe\textsubscript{2}) and (b) boron nitride (BN).
3.3. Tribological behaviors of the lubricants

Figure 6 shows the effect of the additive content on the COFs and wear scar width of lubricating grease when the additive content ranged from 0 to 4 wt%. It can be seen that with the increasing additive content, the COFs and wear scar widths of the lubricating greases decreased first and then increased. However, the COFs and wear scar width of NbSe2 lubricating grease were always the lowest. Meanwhile, when the addition content was 2 wt%, the tribological performances were optimal. Among these two kinds of additives, 2 wt% NbSe2 reduced the COFs by 28.2% (0.234 to 0.168) and the wear scar width by 18.7% (0.375 to 0.305 mm) as compared to the base grease.

The concentration of additives is a key factor which significantly affects the performances of lubricants. Additive concentrations that are too low will result in insufficient lubricating protective film formation during friction process. However, high additive concentration can make it difficult to disperse the additive uniformly, thereby negatively impacting the lubricating performances. The results shown in figure 6 indicated that when the concentration was 2 wt%, BN and NbSe2 all could exhibit the best tribological properties.

Figure 7 shows the COFs and wear scar width of the lubricating greases (both with a content of 2 wt%) under different loads. It can be seen that with the increase of load, the COFs and wear scar widths of the lubricating greases increased slowly. In general, high load results in an aggressive friction behavior and a large amount of heat in the contact area. The heat could reduce the lubricating effect of the lubricating film. Thus, all COFs and wear scar widths increased slowly with a raising load [36]. However, under different loads, the COFs and wear scar width of NbSe2 were always the smallest, which indicated that NbSe2 could form an effective lubricating protective film under both high and low applied loads.

Figure 8 gives the COFs and wear scar widths of the lubricating greases (both at 2 wt%) at different frequencies. It can be seen that with the increasing frequency, the COFs and wear scar widths of the lubricating
greases gradually increased. The tribological properties of the greases at low frequency was better than that at high frequency. This was mainly attributed to that high frequency leaded to a severe the friction behavior, resulting in a decrease in the tribological properties of the lubricating greases. However, under the conditions of the four tested frequencies, the COFs and wear scar width of NbSe2 were still the smallest, indicating that the friction reducing and anti-wear abilities were the best among the tested samples.

3.4. Analysis of the scratch surface

Figure 9 depicts the SEM images of the wear tracks lubricated by the different lubricating greases under 150 N and 5 Hz. It can be seen that the wear surface under lubrication of the base grease was relatively rough. Deep grooves and sticking areas appeared on the worn surface, which indicated that the vigorous friction behavior occurred during the process of friction. The wear surfaces lubricated by BN and NbSe2 greases were relatively smooth. There was only a few shallow scratches, which implied both BN and NbSe2 exhibited good anti-wear properties during the friction.

XPS is very sensitive to the chemical state of element, which can provide information about the main chemical compositions of the worn surface to analyze the lubrication mechanisms. Figure 10 gives the XPS spectrums of the typical chemical elements on the worn surfaces. The XPS spectrum of Fe2p (figure 10(a)) had two distinct peaks at 707.8 eV and 720.3 eV, which may belong to Fe2O3 and/or Fe3O4 [37, 38]. This indicated that severe wear occurred during the friction process, resulting in the formation of iron oxides on the worn surface. This layer of iron oxides could act as a lubricating protective film to improve the tribological properties.

Figure 10(b) gives the XPS spectrum of Nb3d which also had two sharp peaks at 204.3 and 207.2 eV. These peaks belong to the Nb2O5, which indicated that NbSe2 decomposed to generate Nb2O5 during friction process [39]. Figure 10(c) gives the XPS spectrum of O1s which possesses the peaks at 532.2, 531.3 and 530.1 eV. Combining the XPS results shown in figures 10(a) and (b), these oxides may consist of Fe2O3, Fe3O4, Nb2O5 and oxy-hydroxide [37, 38, 40]. Figure 10(d) gives the XPS spectrum of Se3d which exhibited two peaks at 54.5 and 53.4
These peaks belong to the Se element of NbSe$_2$, which indicates that NbSe$_2$ may deposit on the friction surface.

Figure 11 further gives the Raman spectrum of the worn surface lubricated by NbSe$_2$ grease. The typical peaks at 203.4 and 237.2 cm$^{-1}$ could be observed on the Raman spectrum of the worn surface, which belong to Se-Se and Nb-Se of NbSe$_2$ [43, 44]. This result confirmed that NbSe$_2$ was deposited on the worn surface during the friction process, which can act as a lubricating protective film on the friction surface, effectively separating the friction interfaces to improve the tribological properties.

The tribological properties of NbSe$_2$ grease under different concentrations, loads and frequencies were tested and the worn surface was also characterized in detail by SEM, XPS and Raman. Experiments show that NbSe$_2$ has better tribological properties than base grease and BN grease, and the reasons can be summarized in these aspects as shown in Figure 12. (1) Figure 3 indicates that the NbSe$_2$ has a layered structure. Therefore, layered NbSe$_2$ particles could be transferred to the exfoliated phases with fewer layers because of the weak Vander Waals interaction between Se-Se layers. These exfoliated phases could strongly adhere to worn surface to act as a lubricating film to avoid direct contact and reduce the shear stress between the friction interfaces, thereby reducing the friction coefficient and improving the wear resistance. (2) During the friction process, NbSe$_2$ can be deposited on the friction surface under the action of applied load, which can repair and polish the worn surface. This behavior can reduce the roughness of the friction surface to a certain extent, thereby reducing the wear of the friction pair. (3) The XPS and Raman analysis suggests that sever chemical reactions occurred on the worn surface to promote the generation of products including Fe$_2$O$_3$, Fe$_3$O$_4$, Nb$_2$O$_5$, oxy-hydroxide and so on. These reaction products could also act as an effective lubricating protective film to enhance the reducing friction and anti-wear of the friction pair [30, 45, 46].

4. Conclusions

In this study, the electrical conductivity and tribological behaviors of the NbSe$_2$ doped lubricating grease were investigated in detail. The conductive and lubricating mechanisms are also analyzed in depth. The main conclusions are as follows:
1) NbSe₂ has a typical layered structure and it could greatly reduce the volume resistivity of the lubricating grease. When the concentration is 4 wt%, NbSe₂ could effectively reduce the volume resistivity by ten times as compared with the base grease.

2) Compared with the base grease and BN doped grease, when the concentration is 2 wt%, NbSe₂ doped grease exhibited better tribological performance under different applied load and frequencies. Especially, NbSe₂ grease could reduce the COF and wear scar width by 23.5% and 12.8% under 150 N and 5 Hz, respectively.

Figure 10. XPS curves of the typical elements including Fe2p, Nb3d, O1s and Se3d on the worn surfaces lubricated by NbSe₂ grease.

Figure 11. Raman spectrum of the worn surface lubricated by NbSe₂ grease.
(3) NbSe2 has a layered structure. Therefore, NbSe2 particles could be transferred to the exfoliated phases with fewer layers, which can strongly adhere to worn surface and act as a lubricating film to avoid direct contact and reduce the shear stress between the friction interfaces, thereby enhance the tribological performance of the friction pair.

(4) XPS analysis suggests that complex chemical reaction products were formed on the worn surface and Raman analysis indicates NbSe2 deposited on the worn surface. These complex substances could act as an effective lubricating protective film on the worn surface to improve the tribological performances.

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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