Magnetron sputtering NbSe₂ film as lubricant for space current-carrying sliding contact

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Abstract: This study demonstrates that magnetron-sputtered NbSe₂ film can be used as a lubricant for space current-carrying sliding contact, which accommodates both metal-like conductivity and MoS₂-like lubricity. Deposition at low pressure and low energy is performed to avoid the generation of the interference phase of NbSe₃. The composition, microstructure, and properties of the NbSe₂ films are further tailored by controlling the sputtering current. At an appropriate current, the film changed from amorphous to crystalline, maintained a dense structure, and exhibited excellent comprehensive properties. Compared to the currently available electrical contact lubricating materials, the NbSe₂ film exhibits a significant advantage under the combined vacuum and current-carrying conditions. The friction coefficient decreases from 0.25 to 0.02, the wear life increases more than seven times, and the electric noise reduces approximately 50%.

Keywords: NbSe₂ films; magnetron sputtering; vacuum; current-carrying tribological properties

1 Introduction

Sliding electrical contact is a mechanical design used to transmit electric energy and signals in the sliding contact state [1–3]. It is widely used in aerospace applications. For example, electrically conductive slip rings are used to transfer energy collected by a solar panel to a spacecraft system. The lubrication and wear resistance of the electrical contact materials directly determine the reliability of the entire system. The extreme service conditions, such as high vacuum and carrying-current, bring huge challenges for electrical contact materials. Compared with traditional friction, the one with current-carrying is affected also by the electric field accompanied by arc discharge and other phenomena [4]. During electrical contact sliding, frictional heating and Joule heat generated by the current can lead to high temperature near the electrical contact interface and thus the severe adhesive wear [5]. Therefore, electrical contact materials must have good electrical conductivity and lubricity [6–8]. At present, the research in this field is still not sufficient. The major reason is that vacuum environment and current-carrying evaluation conditions are difficult to achieve simultaneously, and the guidance for material design is insufficient.

Gold, platinum, and other precious metals are widely used as lubricating materials for space current-carrying sliding contact owing to the excellent electrical conductivity and chemical stability [9, 10]. However,
precious metal materials exhibit high friction coefficient (usually above 0.3) and serious adhesion wear, which seriously limits the wear life. The specific characteristic of space sliding electrical contact limits the applicability of a lot of traditional solid lubricants. For example, although graphite is a solid lubricant with good electrical conductivity, it has high friction and wear in vacuum [11, 12]. MoS2 exhibit low friction in vacuum, but has high resistivity and poor transport characteristic. Therefore, it is meaningful to develop new space lubrication material systems for sliding electrical contact.

NbSe2 is a transition metal compound with a unique layered structure, which accommodates both metal-like conductivity (even superconductivity at low temperature) and MoS2-like lubricity [13–15]. For example, the electrical resistivity of NbSe2 is 3.5 × 10⁻⁴ Ω·cm, whereas that of MoS2 is 8.5 × 10² Ω·cm. NbSe2 has been used in motor brushes and specific bearings in atmospheric environments. However, the application of NbSe2 in a specific space environment is yet to be investigated [16]. Liu et al. [17] used the radio-frequency (RF) magnetron sputtering method to prepare NbSe2 films. The as-prepared films exhibited the excellent dual-functional characteristics of lubrication and electrical conductivity in air. But the existence of the interfering substances such as NbSe3 and Nb2O5 in the films could adversely affect the lubrication and conductivity performances. Thus it is of great significance to further optimize the magnetron sputtering NbSe2 film and explore its application potential for space current-carrying system.

In this study, NbSe2 films were prepared using the DC closed-field magnetron sputtering method. Deposition at low pressure and low energy was performed to keep the purity of NbSe2 sputtering products and avoid the generation of interference phase such as NbSe3. The composition, microstructure, and properties of the NbSe2 films were further tailored by controlling the sputtering current. The tribological properties under real service conditions with current-carrying in vacuum were systematically investigated and compared with those of traditional electrical contact materials, such as the electroplated Au coating and the MoS2 film.

2 Experimental

2.1 Film deposition

NbSe2 films were deposited using a Teer PlasMag CF-800 closed unbalanced field magnetron sputtering system (a single NbSe2 sputtering target and two Ti sputtering targets). The bias voltage was supplied by a pulsed DC power source, while the target current was supplied by a DC supply. Deposition at low pressure of 0.11 Pa (benefit from high ionization closed magnetic field tech.) and low sputtering energy (DC power) was specially designed to keep the purity of NbSe2 sputtering products and avoid the generation of interference phase. The substrates were stainless steel disk (1Cr18Ni9Ti, with surface roughness below 30 nm), Si (100) wafer (with surface roughness below 10 nm), and copper disk (with surface roughness below 30 nm). The stainless steel samples were used for the mechanical properties test, the Si (100) wafer samples were used for the composition, structure, and morphology analyses, and the copper samples were used for X-ray diffraction (XRD) analysis and tribological properties test. Before being placed into the deposition system, the substrates were washed by ultrasonic waves sequentially in deionised water and acetone for 20 min respectively and then dried in nitrogen. The distance between the substrates and targets was maintained at 150 mm. There was no additional heat source to heat the substrate during the deposition. The substrates were rotated in front of each target at a speed of 5 rpm. The Ar gas was introduced when the base pressure was lower than 1×10⁻⁵ mbar, the native oxides on the surface of the substrates were removed by Ar⁺ ions etching for 20 min. A thin interlayer of Ti (250 nm) was deposited to improve the adhesion. The sputtering current of the NbSe2 target was 0.4, 0.5, 0.6, 0.7, and 0.8 A, and the corresponding samples were denoted as C0.4, C0.5, C0.6, C0.7, and C0.8, respectively. The process parameters for film deposition were listed in Table 1. Traditional materials, such as electroplated Au coating [18] and MoS2 film [19], were selected to compare the current-carrying tribological properties to those of the NbSe2 film.

2.2 General characterization

The chemical compositions of the NbSe2 films were
characterized using energy-dispersive spectroscopy (EDS)–scanning electron microscopy (SEM, JSM-5600LV, Japan) and X-ray photoelectron spectroscopy (XPS, ESCALAB 250Xi). XRD patterns were obtained using an X-ray diffractometer (GIXRD, EMPYREAN, PANalytical) in the grazing mode (2°). The morphologies of the NbSe$_2$ films were observed using field-emission scanning electron microscopy (FESEM, JSM-6701F, JEOL). Cross-sectional images of the films were obtained by breaking the silicon substrate after the film deposition. Focused ion beam (FIB, FEI scios) was used to fabricated the nanoscale-thick lamellar specimens of NbSe$_2$ film for HRTEM (FEI Talos-F200S) observation. The nanohardnesses and elastic modulus of the NbSe$_2$ films were measured using a nanoindenter (Nano Hardness Tester, CSM) equipped with a Berkovich diamond probe tip. The indentation depth was controlled to below 10% of the film thickness to minimise the effect of the substrate. The adhesive strength of the films was evaluated using a CSM Revetest Scratch tester. The normal load range was 1 to 10 N (loading rate: 4.5 N/min). The square resistance of the film was assessed using a four-probe measuring instrument (McP-y610, Loresta GP, Japan). The resistivity was calculated by $R = \rho/W$, where $W$ is the film thickness and $\rho$ is the square resistance.

### 2.3 Tribological test

The tribological properties were evaluated under the combined vacuum and current-carrying conditions. The ball-on-disk tester (CSM) was configured with the current loading system and voltage testing device (Fig. 1). The counterpart was a GCr15 bearing steel ball ($\Phi = 6$ mm, $Ra = 20$ nm). The current applied during the friction test (1 A) was supplied by the power supply (KXN-10050D), and the current density was similar to the actual working value. All tests were carried out in vacuum (pressure lower than $2 \times 10^{-5}$ mbar) with a load of 1 N, sliding frequency of 3 Hz, reciprocating amplitude of 5 mm, and duration of 6,000 cycles. The tribological tests were repeated four times.

### 3 Results and discussion

#### 3.1 Composition and structure

XPS was used to characterize the chemical composition of the NbSe$_2$ films with different sputtering currents. Figure 2(a) showed the typical Nb 3d peak of the film at a sputtering current of 0.6 A (C0.6). By fitting, it could be decomposed into four peaks at 203.2, 205.9, 207.3, and 210.0 eV, corresponding to Nb 3d$_{5/2}$ and Nb 3d$_{3/2}$ peaks of Nb$^{4+}$ in NbSe$_2$ and Nb 3d$_{3/2}$ and Nb 3d$_{3/2}$ peaks of Nb$^{5+}$ in Nb$_2$O$_5$, respectively [20, 21]. The surface of the NbSe$_2$ film was consisted of the majority of Nb$_2$O$_5$. Previous studies had demonstrated oxidation pollution on the surface of sulphur (selenide) sputtered films due to surface oxygen adsorption or residual oxygen during deposition [17, 22]. To characterize actual chemical composition of films, The Ar ion was employed to etch the surfaces of the NbSe$_2$ films for 2 min to eliminate the external influence on chemical composition. Figure 2(b) showed the typical XPS results for the C0.6 film after the etching process. The Nb$_2$O$_5$ content of the C0.6 film was significantly reduced after the etching process. NbSe$_2$ became the major component of the film. It can be attributed to the existence of the Nb$_2$O$_5$ oxidized surface, partly due to surface oxidation during the transportation process and inevitable residual oxygen-containing substances in the membrane chamber during the preparation process. Figure 2(c) showed the XPS results

| Table 1 Film deposition conditions and thickness. |
|-----------------|-----------------|-----------------|
| No.  | Sputtering time (h) | Sputtering current (A) | Thickness (μm) |
| C0.4 | 5               | 0.4             | 0.70            |
| C0.5 | 5               | 0.5             | 0.82            |
| C0.6 | 5               | 0.6             | 1.15            |
| C0.7 | 5               | 0.7             | 1.35            |
| C0.8 | 5               | 0.8             | 1.25            |

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Fig. 1 Schematic of the ball-on-disk tribometer equipped with the current loading system and voltage testing device.
of NbSe_2 films with different sputtering currents after etching for the same time. With the increase of the sputtering current, the content proportion of NbSe_2 increased and the oxidation decreased. This is because that the low sputtering current results an inadequate sputtering deposition, and the residual O in the membrane chamber can be more easily combined with Nb.

The detection depth of XPS was only tens of nanometres, while EDS can analyze the composition in the range of micrometres, and can more comprehensively characterize the composition of the films. The EDS elemental analysis results for the five different samples were shown in Table 2. With the increase of the sputtering current from 0.4 to 0.8 A, the Se/Nb ratio increased from 1.78 to 2.03. The stoichiometric ratio of the films prepared at high sputtering current was very close to that of NbSe_2. The EDS and XPS results showed that the film prepared under high sputtering current has a lower oxidation content [23]. Compared to the interior of the film, there was obvious oxidation on its surface. In this study, deposition at low pressure and low sputtering energy was specially designed to maintain the purity of NbSe_2 sputtering products and avoided the generation of interference phase such as NbSe_3, which provided favourable conditions for obtaining excellent conductivity and lubrication properties.

The XRD results of the NbSe_2 films with different sputtering currents were shown in Fig. 3. To eliminate the interference of the Cu substrate, the XRD pattern of the Cu substrate was referenced. In addition to the diffraction peak of the Cu substrate, the C0.6, C0.7, and C0.8 films exhibited hcp-NbSe_2 (002), (101), (103), (112) diffraction peaks with a preferred orientation of (002) plane. The absence of Nb_2O_5 crystal phases indicated that it should be amorphous structure. The C0.4 and C0.5 films did not exhibit distinct NbSe_2 diffraction peaks, which indicated that they are mainly

Table 2 Element content of the NbSe_2 films with different sputtering currents.

| No.  | Se/Nb | O (wt%) |
|------|-------|---------|
| C0.4 | 1.78  | 5.1     |
| C0.5 | 1.83  | 4.3     |
| C0.6 | 1.88  | 3.8     |
| C0.7 | 1.96  | 3.6     |
| C0.8 | 2.03  | 3.6     |
amorphous structure. When the sputtering current reached 0.6 A, there exhibited a distinct NbSe$_2$ (002) diffraction peak. With the further increase of the sputtering current, the diffraction peak became stronger. It showed that a high sputtering current was conducive to the crystallization of the NbSe$_2$ films. When the sputtering current was less than 0.6 A, the film existed in an amorphous structure. When the sputtering current reached 0.6 A, the films existed in the form of hcp-NbSe$_2$ (002) preferentially oriented crystals. The low sputtering current can significantly reduce the diffusion ability of the free particles, inhibit the formation and development of crystal nuclei, and eventually lead to a decrease of crystallization.

Figure 4 showed the cross-sectional and surface morphologies of NbSe$_2$ films with different sputtering currents. The structure of the C0.4 film was very dense, which corresponded to the amorphous structure detected by XRD. With the increasing sputtering current, a columnar crystal structure was evident observed from the cross-sectional morphology of the C0.7 and C0.8 films, and the films became loose. Particularly, when the sputtering current was 0.8 A, columnar crystal with distinct gaps penetrated the cross-section of the film. For surface morphologies, with the increasing sputtering current, the cracks at the surface of the film gradually increased, corresponding to the columnar crystal structure. The sputtering current of 0.6 A was the change tipping point, that it exhibited crystalline characteristic while avoiding a several columnar crystals and maintaining a relatively dense structure. According to the XPS results, the Nb$_2$O$_5$ content in the C0.6 film was significantly reduced, maintaining the excellent NbSe$_2$ chemical composition and crystallization. These observations were consistent with the XRD results. The apparent structural difference was related to the migration ability of the deposited particles at the substrate surface. The low sputtering current inhibited the nucleation and growth of base-plane-oriented grains, and thus the film structure tended to be amorphous [24–27]. The formation of the crystal structure (Fig. 4(i)) was mainly caused by sufficient surface diffusion and
insufficient bulky diffusion of adatoms [13, 28, 29], which benefited from the high sputtering current.

Figure 5 showed the HRTEM cross-section images and selected-area diffraction result of the C0.6 film. The films exhibited a special nanocrystalline/amorphous composite superlattice structure. The spacings of the nanocrystal grains were approximately 0.63 and 0.23 nm, corresponding to the (002) and (102) crystal planes of NbSe2, respectively. The continuous rings of the NbSe2 (002) and (102) planes can be observed in the selected area electron diffraction pattern (Fig. 5(b)). Many grain boundaries existed in the nanocrystalline/amorphous composite superlattice, which can effectively impede the movement of dislocations. The internal stress can be released by grain boundary sliding. Such a special nanocrystalline/amorphous composite superlattice structure can effectively impede the movement of dislocations by grain boundaries, and release internal stress by the sliding of the grain boundary [30].

3.2 Mechanical properties

The hardness and elastic modulus of the NbSe2 films with different sputtering currents were shown in Fig. 6. As the increased sputtering current, the hardness decreased gradually. However, the film has the highest elastic modulus when the sputtering current is 0.6 A. The C0.6 film had excellent mechanical properties with the highest elastic modulus and relatively high hardness. It can be understood from two aspects. On one hand, the film denseness was one of the key factors affecting the hardness. According to the structural characterization results, with the increased sputtering current, the films changed from amorphous structure to nanocrystalline/amorphous composite structure. The films prepared under the low sputtering current had a denser microstructure and thus the higher hardness. On the other hand, for the nanocrystalline/amorphous composite structure, the amorphous boundary can limit the crack diffusion in the amorphous matrix, which yields the high hardness and elastic modulus of the C0.6 film [31, 32].

Figure 7 showed scratch morphologies of the NbSe2 films with different sputtering currents. As the sputtering current increased from 0.4 to 0.6 A, the adhesive strength (corresponds to the critical load for the film peeling) increased significantly from 4 to 8 N. While as it further increased to 0.6 A, the adhesive strength decreased to 2 N. The C0.6 film had the best adhesive strength. It was worth noted that brittle cracks appeared at the edge of the scratch track for C0.4, C0.7, and C0.8 films, while none for C0.5 and C0.6 films, which also indicated the better mechanical strength and elasticity for the nanocrystalline/amorphous composite structure [33].

3.3 Electrical conductivity

Figure 8 showed the electrical resistivity results of the NbSe2 films with different sputtering currents. The as-prepared NbSe2 films had excellent conductivity with resistivity in the magnitude of 10^-5 Ω·m. The resistivity of the C0.8 sample was highest, whereas that of the C0.6 sample was lowest. The conductivity of the film was determined by the carrier concentration and Hall mobility. The periodic potential field and scattering process of free electrons inside the film hinder electron transmission. In contrast, the NbSe2 films with high crystallinities can effectively weaken...
this effect and promote electron transmission. Thus, the resistivity of the C0.6 film was lower than those of the C0.4 and C0.5 films. C0.7 and C0.8 films had a loose structure, which was considered to lead to structural defects and dislocations with an increased scattering of a large number of columnar platelet boundaries, and weakening the carrier concentration of the conductive material, resulting in high resistivities [34, 35]. Compared with the NbSe2 film prepared by the RF magnetron sputtering method, the conductivity was reduced from $1 \times 10^{-3}$ to $6 \times 10^{-5}$ $\Omega \cdot m$ for that by DC magnetron sputtering, owing to the low-energy-sputtering induce denser structure and purer composition [17].

3.4 Tribological property under the combined current-carrying and vacuum conditions

Figures 9(a) and 9(b) showed typical tribological properties of the NbSe2 films under the combined current-carrying and vacuum conditions. The friction coefficient and wear rate of the NbSe2 films initially decreased, and then decreased with the increase of the sputtering current. The C0.6 film had the best tribological properties. The friction coefficient was approximately 0.025, while the wear rate was $3.8 \times 10^{-6}$ mm$^3$/((N·m)). Figure 9(c) showed the online contact voltage curves of the NbSe2 films. The C0.5 and C0.7 samples can maintain a low contact voltage of 0.3 V before 1,000 cycles, and then slowly increased during friction process. The contact voltage of the C0.8 film reached approximately 0.5 V and fluctuated violently. The contact voltage of the C0.6 film was lowest and stable, owing to the excellent electrical conductivity and mechanical and tribological properties. The stable stability of electrical signal transmission capability was due to its excellent tribological properties. In contrast, C0.4 and C0.8 exhibited high contact voltages and fluctuation. As known, transition metal dichalcogenides (TMDs) materials usually have good lubricity owing to the lamellar structure, which had weak interlayer interaction and easy sliding nature between neighboring atomic layers [36, 37]. As for the sputtering TMDs films, the original (002) crystal orientation was also conducive to the formation of the ordered lamellar structure at the sliding surface, thus leading to low friction. This can explain the high friction coefficient and wear rate for the films prepared by the low sputtering current. The films prepared by the high sputtering current had poor mechanical strength, and were easily damaged during the friction process [38–40].
3.5 Comparison with the available electrical contact lubricating materials

In a series of NbSe₂ films prepared by us, the C0.6 film exhibited the most excellent tribological properties under the combined current-carrying and vacuum conditions. To evaluate its practical application value, electroplated Au coating and traditional MoS₂ space lubricating film materials were selected to compare with C0.6. Their tribological performances and failure mechanisms under real service conditions were systematically studied. Figure 10(a) showed the friction curves of the C0.6 film, MoS₂ film, and electroplated Au coating under the combined current-carrying and vacuum conditions. For the electroplated Au coating, the friction coefficient is approximately 0.22, while the wear life is 1.6 × 10⁴ cycles (the tests were stopped as the friction coefficient exceeded 0.4). For the traditional MoS₂ space lubricating film, the friction coefficient was approximately 0.05 and the wear life is 2 × 10⁴ cycles (the tests were stopped as the friction coefficient exceeded 0.2). The characterization of the wear scar depth also showed that the Au coating and MoS₂ film had been worn out. For the NbSe₂ film, the friction coefficient is only approximately 0.02, which was 10 times lower than that of the electroplated Au coating. The NbSe₂ film was still not worn after 1 × 10⁵ cycles, and the wear life was at least 6.25 and 5 times of the electroplated Au coating and MoS₂ film, respectively. Regarding the electrical contact performance (Figs. 10(a) and 10(b)), the contact voltages of the electroplated Au coating and NbSe₂ film were at the same level of magnitude, which indicated that the NbSe₂ films had excellent electrical conductivity similar to that of the electroplated Au coating. The MoS₂ film exhibited a very high contact voltage (0.6 V) owing to its poor conductivity. Regarding the electrical contact stability, the contact voltage and current fluctuations showed that the NbSe₂ film exhibited a lower and optimal electrical noise. It showed the distinct advantages and potential applications of the NbSe₂ films as a new lubricant for space current-carrying sliding contact.

To explore the effect mechanism of the current-carrying on tribological properties, we also carried out the comparative tests without current-carrying in vacuum. Figure 11 showed SEM images of the wear surfaces of the tribo-pairs (NbSe₂ film, MoS₂ film, ...
and electroplated Au coating VS steel ball) without current-carrying in vacuum. The wear tracks were flat and smooth, and a dense transfer film was observed on the counterpart ball surface for all three materials. As shown in Fig. 12(c), obvious traces of erosion were observed (dashed line) on the wear track of the MoS₂ film. The Joule heat generated by the high contact voltage damaged tribo-pairs severely. This was inconsistent with the tribological comparison results, which the wear life of the MoS₂ film in vacuum is reduced from $1.3 \times 10^5$ cycles (see the Electronic Supplementary Material (ESM)) without current-carrying to $2 \times 10^4$ cycles with current-carrying. Upon sliding against the electroplated Au coating, the contact areas of the wear scars on the counterpart ball become largest. The film was severely damaged and a large amount of gold was transferred to the counterpart ball surface. This also showed that the NbSe₂ film exhibits excellent tribological properties under the vacuum and current-carrying conditions. The current and voltage applied to the friction pair have arc erosion effects. When the film had poor electrical conductivity, an arc is generated between the friction pairs, ablating and destroying the film structure (e.g., the MoS₂ film). On the other hand, the Joule heat generated by the current softened the film and caused
Fig. 12  SEM images of the wear surfaces of the tribo-pairs with current-carrying in vacuum.

very large wear (e.g., electroplated Au and MoS₂ film). The combination of the excellent electrical conductivity and lubricity of the NbSe₂ film enables it to form a stable electrical contact state, weakening the current erosion and Joule heating [41, 42].

4 Conclusions

A series of NbSe₂ films were prepared using the DC closed-field magnetron sputtering method. The relationship between the sputtering current and composition, microstructure, and mechanical and tribological properties under the combined vacuum and current-carrying conditions were studied. The feasibility of the NbSe₂ film as a new type of space electrical contact lubricant was investigated. The main conclusions are as follows.

1) Deposition at low pressure and low energy was performed to keep the purity of NbSe₂ sputtering products and avoid the generation of interference phase such as NbSe₃, which endowed it excellent electrical conductivity and lubricity integrated performance.

2) The low sputtering current resulted in the amorphization and densification trend of NbSe₂ film, and the high sputtering current leaded to the loose structure of NbSe₂ film with significant columnar crystals. While at an appropriate sputtering current, the NbSe₂ film not only changed from amorphous to crystalline, but also maintained a dense structure with excellent comprehensive properties.

3) As compared with electroplated Au coating now in service, NbSe₂ film showed significant advantage at real service conditions with vacuum and current-carrying, where the friction coefficient decreased from 0.25 to 0.02, the wear life increased from 14,000 cycles to more than 100,000 cycles, and the electric noise fluctuation was reduced by about 50%. It developed a new material system, and provided a new idea for solving the lubrication problem of space sliding electrical contact.

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