



## Featured Letter

Response of MoS<sub>2</sub>-Sb<sub>2</sub>O<sub>3</sub> film to low-earth-orbit space environment

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## ABSTRACT

Many failures and anomalies of spacecrafts originated from space environmental factors. Meanwhile, MoS<sub>2</sub> based films have been widely used in space technology as solid lubricants. In this paper, the compact MoS<sub>2</sub>-Sb<sub>2</sub>O<sub>3</sub> composite film was rf-sputtered and exposed in low earth orbit (LEO) environment, which was aboard the exterior of Shenzhou-7 Manned Spaceship (SZMS-7). The results revealed that the space exposure effect on MoS<sub>2</sub>-Sb<sub>2</sub>O<sub>3</sub> composite film mainly exhibited the oxidation at the film surface layer less than 3.5 nm. No severe oxidation and cracking phenomena were observed from the space-exposed MoS<sub>2</sub>-Sb<sub>2</sub>O<sub>3</sub> composite film, which was normal for many space materials. Namely, the MoS<sub>2</sub>-Sb<sub>2</sub>O<sub>3</sub> composite film exhibited a better capability resistant to LEO space environment.

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## 1. Introduction

In space environment, the properties of materials can be influenced by a variety of factors [1], such as high vacuum, cosmic radiation, thermocycling, atomic oxygen (AO), microgravitation, micrometeorites, and man-made debris, etc. National Aeronautics and Space Administration (NASA) report [2] summarized the effects of space environment on spacecrafts and pointed out that the AO was the most important factor in LEO environment. MoS<sub>2</sub>, a typical two-dimensional material, has potential applications as catalysts [3–5], energy materials [6–8] and lubricants [9–11] due to its special layer structure. In space technology, sputtered MoS<sub>2</sub> based films have been widely used as solid lubricant of moving parts [12]. Sometimes, they must undergo AO irradiation in space environment. Therefore, increasing attentions have been paid on the AO effects on the MoS<sub>2</sub> based films to ensure the service performance and high reliability of space mechanisms.

Two experimental methods (ground simulation and space exposure) have been used to investigate the space environment effects on the MoS<sub>2</sub> based films [13,14]. In fact, the space environment is the integration of different factors, which are very difficult to be synchronously simulated on the ground. Sometimes, there existed some deviations between the results of ground simulation and space exposure experiments. For example [15], Dugger investigated the effects of space exposure and ground simulation on the MoS<sub>2</sub>/Ni multilayer films and found the delamination phenomenon from the space exposed sample, which was absent for the ground

simulated sample. Thus, it can be said that the space exposure experiment is the most effective method to evaluate the ability of materials resistant to space environment.

The wear resistance of MoS<sub>2</sub> films can be improved by adding some amount of metals or oxides [16]. However, it is worth noticing that a majority of metal elements are reactive with the AO [17], and hence the oxidation resistance of MoS<sub>2</sub>-metal composite films is a worrying problem if they will be used in the LEO environment. Previous study [18] revealed that the structure and wear resistance of MoS<sub>2</sub> film could be improved by adding Sb<sub>2</sub>O<sub>3</sub>. Our previous study [19] investigated the AO irradiation effect on MoS<sub>2</sub>-Sb<sub>2</sub>O<sub>3</sub> films by the ground simulation method. In this study, the sputtered MoS<sub>2</sub>-Sb<sub>2</sub>O<sub>3</sub> composite film was exposed in LEO space environment aboard China Shenzhou-7 Manned Spaceship (SZMS-7) and the capability of this composite film resistant to space environment was clarified.

## 2. Experimental detail

## 2.1. Film deposition

The MoS<sub>2</sub>-Sb<sub>2</sub>O<sub>3</sub> composite film was co-deposited on AISI 440C steel substrate (25 × 25 × 5 mm<sup>3</sup>,  $R_a \leq 0.03 \mu\text{m}$ ) using a MoS<sub>2</sub>-Sb<sub>2</sub>O<sub>3</sub> composite target with a diameter of 85 mm by rf-sputtering technology. Before deposition, the vacuum chamber was evacuated to a base pressure  $\leq 1.0 \times 10^{-3}$  Pa and then the substrate was surface-etched in Ar plasma to eliminate possible contamination. Afterwards, the film was deposited under Ar pressure of  $\sim 5.0$  Pa and sputter power of  $\sim 500$  W. During film

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deposition, the substrate surface was parallel to the target surface and not rotated. The film thickness was controlled to be  $\sim 1 \mu\text{m}$ .

## 2.2. Space exposure experiment

Eight samples of the  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite films were space-exposed for 43.5 h in LEO environment, which were fixed on the Space Exposure Experiment Device (SEED) aboard SZMS-7. The SEED's position on the SZMS-7 was reported in elsewhere [20]. As shown in Fig. 1, the samples were randomly divided into two groups and fixed onto the SEED's front and back, respectively. The SZMS-7 was launched at 21:30 on September 28, 2008 from Jiuquan Satellite Launch Centre (JSLC). The orbit altitude and inclination of the SZMS-7 were about 343 km and 42.4 degree, respectively. The atomic oxygen flux calculated from the altitude and attitude of the SZMS-7 was about  $1.19 \times 10^{18} \text{ atom}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$  at the flight attack angle of 0 degree.

## 2.3. Characterization

After the flight mission, the samples returned to the ground and then were analyzed. X-ray diffraction (XRD, Philips X'Pert Pro) with Cu Ka radiation (40 kV, 30 mA) was performed to identify the film phase structure. The surface morphology was observed by scanning electron microscope (SEM, JEOL JSM-6701F). The chemical composition was analyzed using X-ray photoelectron spectroscopy (XPS, PHI Quantera Scanning X-ray Microprobe) equipped an Ar ion sputtering gun with a monochromatic Al Ka X-ray source of 1486.6 eV (hv). The spectra were referenced with respect to C 1s line at 284.6 eV.

## 3. Results and discussion

The structure of as-deposited  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite film was reported in elsewhere [19]. It was well known that the sputtered  $\text{MoS}_2$  film normally exhibited a porous columnar microstructure. However, it was found that the  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite film displayed an amorphous-like structure, indicating that the growth of  $\text{MoS}_2$  micro-platelets was significantly suppressed due to the incorporation of  $\text{Sb}_2\text{O}_3$ . As a result, the  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite film showed a very compact microstructure.

Fig. 2 gives the typical SEM surface micrographs and XRD patterns of space-exposed  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite films. Similar to the non-exposed film [19], the space-exposed  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite films still showed a featureless morphology, which was independent of the sample position. Previous studies [11] revealed that the sputtered  $\text{MoS}_2$  film normally presented two textures of (002) and (hk0) in XRD analysis due to its crystal anisotropy. However, in the present study the XRD patterns of  $\text{MoS}_2\text{-Sb}_2\text{O}_3$

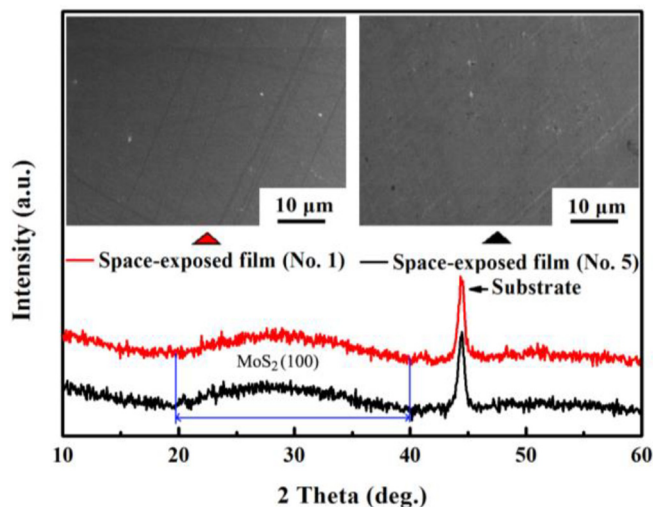


Fig. 2. Typical SEM surface micrographs and XRD patterns of space-exposed  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite films.

composite films only displayed a very broad peak at  $2\theta = 20\text{--}40^\circ$  either before or after the space exposure, indicating that the  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite film was amorphous-like and almost not influenced by the space exposure. XRD and SEM results suggested that the influence of space exposure on the structure of  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite films seemed to be negligible.

To further clarify the effects of LEO space environment on the  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite film, XPS analysis was implemented. Fig. 3(a) shows the typical XPS sputter profile of space-exposed  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite film. At the film surface, the O content was remarkably high, while the contents of Mo, S and Sb were relatively low. After the sputter of 1 min, the O content was obviously decreased along with the increase in the contents of Mo, S, and Sb, and then the contents of Mo, S, O and Sb were almost not influenced by the sputter time. Evidently, the effect of space exposure on the  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite film was restricted into the film surface layer. According to the previous study [21], the sputtering rate was  $\sim 3.5 \text{ nm/min}$  for the  $\text{MoS}_2$  film using an argon ion beam of 3 keV in XPS profile analysis. In this study, the used argon ion beam in the XPS profile analysis had a lower energy of 2 keV, suggesting that the sputtering depth of 1 min was lower than 3.5 nm. Namely, the affected depth by the space exposure was less than 3.5 nm for the composite film.

The chemical states of Mo, S, O and Sb element at the surface and inner of space-exposed  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  film were also analyzed and the typical results were shown in Fig. 3(b–d), respectively. At the film surface, the binding energies of Mo  $3d_{5/2}$  and  $3d_{3/2}$  were

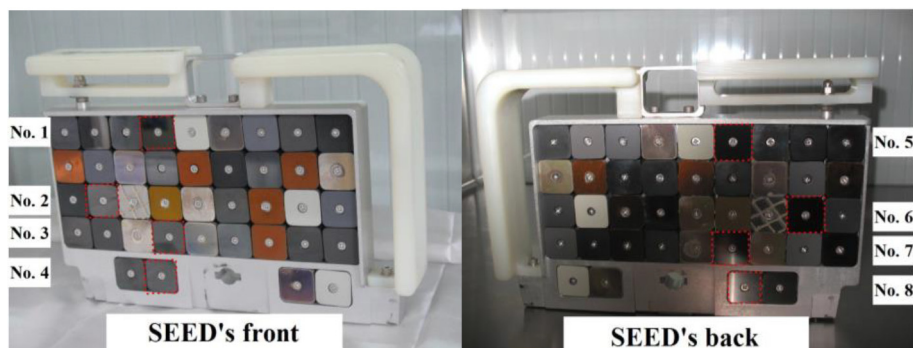


Fig. 1. Samples of  $\text{MoS}_2\text{-Sb}_2\text{O}_3$  composite films fixed on the SEED.

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