Evaluations of Solid Lubricant in JEM/SEED Experiment

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The degradation of materials in extreme environments is a critical issue affecting the reliability of mechanical systems in space. In the low earth orbit (LEO) space environment, factors such as atomic oxygen (AO), ultraviolet rays (UV), and radiation strongly affect materials. A number of materials for space applications were exposed to an LEO space environment by the Japan Experimental Module / Space Environment Exposure Device (JEM/SEED) experiment aboard the International Space Station (ISS) to evaluate how they would be affected by a real space flight environment. A solid lubricant coating was one of the experimental materials evaluated in the JEM/SEED experiment. In addition to the orbital evaluation, the same type of coating was irradiated with atomic oxygen (AO), and ultraviolet rays (UV) on the ground. The fluences of AO and UV irradiation corresponded to the exposure in LEO environment around the ISS during the SEED experiment. Samples from LEO exposure, ground irradiation with AO and UV, and a reference (non-flight and non-irradiated) sample were then subjected to friction tests in a vacuum and surface analyses. The results were compared to elucidate the effects of the various factors on the characteristics of the solid lubricant film.

Key Words: Solid Lubricant, MoS2 Bonded Film, JEM/SEED, Low Earth Orbit Environment

1. Introduction

The degradation of materials in extreme environments is a critical issue affecting the reliability of mechanical systems in space. In the low earth orbit (LEO) space environment, factors such as atomic oxygen (AO), ultraviolet rays (UV), and radiation strongly affect materials. A number of materials for space applications (e.g., thermal coatings, paints, lubricants and so on) were exposed to an LEO space environment in the Service Module / Space Environment Exposure Device (SM/SEED) experiment aboard the International Space Station (ISS) to evaluate how they would be affected by a real space flight environment 1–2). Three sets of pallets containing the same specimens were exposed to the space environment for different durations. A molybdenum disulfide (MoS2) film acting as solid lubricant coating was selected as one of the experimental materials in the experiment 3–8). Although longer exposure resulted in lower friction coefficients, much contamination was detected on the specimens. Therefore, the main factors affecting the observed tribological properties, whether contamination, AO, UV or another factor, were not clear.

The Japanese Experiment Module / Space Environment Exposure Devices (JEM/SEED) experiment was also exposure experiment using JEM Exposure Facility. The same MoS2 specimen as used in the SM/SEED experiment was selected and exposed to a LEO environment in the JEM/SEED experiment. The Service Module is located at the tail of the ISS, an area that appears to receive much contamination. The JEM is located on the front of the ISS, which is thought to be a cleaner, less contamination environment. Friction behavior and surface analysis results for the MoS2 specimen are reported.

2. Project Outline

The JEM / Micro-Particles Capturer and Space Environment Exposure Devices (JEM/MPAC&SEED) experiment was carried out as part of the Space Environment Data Acquisition equipment-Attached Payload (SEDA-AP) mission launched on Space Shuttle 2J/A (STS-127) in July 2009. After being mounted aboard the JEM exposure facility, the SEED pallet, which held specimens of materials for space applications, was exposed to the space environment from July 23. Fig. 1 shows photographs of the SEED pallet during exposure in LEO and after retrieval. The SEED pallet was retrieved to inside the ISS on April 9, 2010 and then returned to Earth by Space Shuttle 19A (STS-131). The exposure duration was 8.5 months (259 days), and the average altitude of the ISS estimated from flight data for these days was 343.4 km, at an inclination of 51.6 deg. The quantities of the effective factors, AO and UV, calculated from the altitude and attitude of the ISS and monitor materials, are listed in Table 1. The monitor materials were Vespel for AO and indium tin oxide (ITO) coated urethane sheet for UV. (In Table 1, Equivalent Solar Day (ESD) as a unit of UV, with 1 ESD being equal to $9.2 \times 10^6$ J/m².)

The SEED pallet included a lubricant specimen. It is commercially available bonded MoS2 films coated onto a titanium alloy substrate. An organic binder, polyamide-imide, constitutes about 65% of the film. The film thickness was about 10 µm. The specimen shape and the exposed area of the
coating are shown in Fig. 2.

The same types of specimen were also used in a ground-based experiment that simulated space exposure conditions. The same types of MoS₂ sample were exposed to AO and UV in a facility at JAXA. Some samples were exposed to only one factor (AO or UV) while others were exposed to both factors sequentially in different orders (i.e., AO followed by UV and vice versa). The AO environment was simulated by a laser-detonation source, while the UV environment was simulated by Xe short arc lamp. The levels of these irradiations are also listed in Table 1.

### Table 1. Quantities of effective factors in flight and ground experiment.

<table>
<thead>
<tr>
<th>Factor</th>
<th>JEM/SEED flight experiment</th>
<th>ground tests</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>calculation</td>
<td>monitor materials</td>
</tr>
<tr>
<td>Atomic oxygen</td>
<td>1.40 x 10⁷</td>
<td>5.91 x 10²⁰</td>
</tr>
<tr>
<td>[atoms/cm²]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ultraviolet rays [ESD]</td>
<td>56.85</td>
<td>&lt; 30</td>
</tr>
</tbody>
</table>

### 3. Evaluation Methods

#### 3.1. Friction test

Friction tests were performed using a tribometer, which has a pin-on-flat configuration. Sliding between a flat and counterpart specimen was done using a reciprocating linear motion. The counterpart specimen was a 7.94 mm-diameter ball composed of 440C stainless steel. The sliding speed and length were, respectively, 10 mm/s and 10 mm, and the applied load was 2 N. Friction tests were performed for each sample. The tests were interrupted at 100, 2,000, 20,000, and 100,000 sliding strokes and the wear tracks were examined to understand the mechanism of any friction changes.

#### 3.2. Surface analysis

X-ray photoelectron spectroscopy (XPS) analyses were carried out on the surfaces of a reference (non-flight and non-irradiated) sample, a flight sample, an AO-irradiated sample, and a UV-irradiated sample. The rubbing tracks after the friction tests were also analyzed by XPS for these samples. X-rays images of monochromatic Al Kα were used, focused to φ100 μm for the film surfaces and φ20 μm for the rubbing tracks.

### 4. Results and Discussion

#### 4.1. XPS analysis of film surfaces

Fig. 3 shows wide-range XPS spectra of the film surfaces of a flight sample, a reference sample and samples irradiated with either AO or UV, and Fig. 4 shows Mo 3d photoelectron spectra of the flight sample. The surface compositions of the
samples from the XPS analysis are presented in Table 2.

The surface of the flight sample showed a high oxygen content, and two peaks were observed in the Mo 3d spectra at 232 and 236 eV in the surface of the sample. These peaks indicate Mo(VI) and the presence of MoO₃. These results show that the surface of the sample was oxidized. In contrast, the flight sample showed lower carbon content than the reference sample. The AO-irradiated sample showed similar results, whereas the composition of the UV-irradiated sample was not significantly different to that of the reference sample. It is inferred from these results that molybdenum was oxidized and carbon was selectively etched in the surface of the flight sample by atomic oxygen in orbit.

Silicon and fluorine, which were not present in the film originally, were also detected in the surface of the flight sample. These must be contamination. The proportion of silicon in the surface of the JEM/SEED flight sample was less than in a sample from the earlier SM/SEED experiment. The JEM/SEED experiment environment seems to be cleaner, with less contamination than the earlier experiment. On the other hand, fluorine was not detected in the SM/SEED flight sample, so it seems to have come from a different source to the silicon. However, it is not clear what the source of this contamination was, or when it was deposited. Some other metals were detected in the surface of the AO-irradiated sample, and silicon was detected in the surface of the UV-irradiated sample. It is known that metallic elements came from the AO source, but the source of the silicon contamination of the UV-irradiated sample is unknown.

### Table 2. Surface composition of the samples by XPS. [\%]

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>O</th>
<th>S</th>
<th>Mo</th>
<th>Sb</th>
<th>Si</th>
<th>F</th>
<th>other metals</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flight</td>
<td>17.0</td>
<td>43.1</td>
<td>3.4</td>
<td>8.9</td>
<td>1.6</td>
<td>6.2</td>
<td>19.6</td>
<td>0.2</td>
</tr>
<tr>
<td>Reference</td>
<td>63.7</td>
<td>15.6</td>
<td>12.1</td>
<td>7.4</td>
<td>0.4</td>
<td>0.8</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>AO</td>
<td>22.7</td>
<td>52.1</td>
<td>5.6</td>
<td>11.7</td>
<td>2.0</td>
<td>3.1</td>
<td>-</td>
<td>2.7</td>
</tr>
<tr>
<td>UV</td>
<td>53.3</td>
<td>29.9</td>
<td>2.6</td>
<td>1.7</td>
<td>12.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

4.2. Friction behaviors

Fig. 5 depicts friction behaviors of the flight sample, the reference sample, and AO, UV singly irradiated samples. All the samples showed high friction coefficients at the start of friction tests due to the effects of surface roughness and so on, and then they decreased. The friction coefficient of the flight sample decreased sharply from start of the test and reached a steady state earlier than the reference sample. However, the downward curve was gentler than that of the SM/SEED flight sample \(^7,8\). The AO-irradiated and UV-irradiated samples also showed low friction coefficients from beginning of the tests. The AO-irradiated sample had similar behavior to the flight sample, but showed a lower friction coefficient than the flight sample at the beginning of the test. The friction coefficient of the UV-irradiated sample increased slightly and became stable after decreasing at beginning of the test.

The friction behaviors at the beginning of the tests of samples dual-irradiated sequentially with AO and UV are shown in Fig. 6, comparing with the behaviors of singly irradiated samples. Both samples exposed first to atomic oxygen and then ultraviolet rays (AO-UV in the figure) and ultraviolet rays and then atomic oxygen (UV-AO) showed low friction coefficients and showed similar friction behavior to the AO-irradiated sample.
4.3. Observation and analysis of wear tracks

To understand wear process and mechanism of friction change, friction tests were interrupted at 100, 2,000 and 20,000 sliding strokes and spot XPS analysis of wear tracks of the films and microscope observation of the contact area of counterpart were carried out.

The contact areas of 440C stainless steel counterpart ball after 100 strokes and 20,000 strokes for each specimen are presented in Fig. 7. From photographs of the counterpart after 100 and 20,000 sliding strokes of the reference sample, it can be recognized that transfer film built up strongly. Similarly for the UV-irradiated sample, hard transfer film adhered to the counterpart after 100 and 20,000 sliding strokes. In contrast, for the AO-irradiated sample, transfer film was recognized only at the entrance and exit sides of the contact area, and had not adhered to the center of the contact area of the counterpart after 100 and 20,000 sliding strokes. This indicates that mechanism of friction change is different between AO-irradiated and UV-irradiated sample, although both showed a sudden decrease in friction. On the counterpart of the flight sample, a little transfer was found after 100 sliding strokes, but transfer film was not recognized at the center of contact area after 20,000 strokes. The friction change mechanism of the flight sample seems to be the same as that of the AO-irradiated sample, atomic oxygen contributing to the decrease in friction. The morphologies of counterpart contact area for dual-irradiated samples were also similar to one of the AO-irradiated samples. Atomic oxygen must have strongly affected the friction behavior of the film in this study.

Fig. 8 shows the proportions of elements detected by XPS on the film surface and wear tracks after 100 and 20,000 times sliding strokes for each sample. The listed elements typically occur in the films or contaminants. The surface of the flight sample was oxidized, and the proportions of elements were similar to that of the AO-irradiated sample except for contaminants. The oxygen concentration was approximately 10% in the 100-stroke wear tracks of all samples, regardless of the proportions of oxygen on the film surfaces. This ratio of 10% was unchanged after 20,000 strokes, indicating that the oxide layer was removed by sliding at an early stage. This is also apparent from Mo 3d spectra of the wear tracks of the flight sample in Fig. 4. The carbon ratio in the flight sample and the AO-irradiated sample increased with sliding. This appears also to be due to the removal of the oxide layer. The carbon concentrations in the reference and UV-irradiated samples decreased by 100 sliding strokes and then returned to nearly the same value at 20,000 sliding strokes as that of the film surface. For the contaminant elements, silicon was removed by a small number of sliding strokes, but the ratio of fluorine did not change by 100 sliding strokes. The influence of fluorine may persist longer than silicon, although the contribution of the fluorine contamination to friction behavior is not obvious.

![Image of wear tracks and XPS analysis results]

Fig. 7. Wear tracks of counterpart specimens for each sample after 100 strokes and 20,000 strokes.
5. Summary

A bonded MoS₂ film was exposed to a low earth orbit environment in the JEM/SEED experiment. Due to the location of the specimen pallet in a relatively clean environment, less contamination was expected than in an earlier SM/SEED experiment. Samples of the same film were irradiated with atomic oxygen and ultraviolet rays on the ground. The tribological properties and surface composition of the films after exposure were evaluated, with the following finding:

1. Silicon and fluorine contaminations was detected on the JEM/SEED flight sample. The amount of silicon contamination, which was detected also on the SM/SEED flight sample, was small.

2. The JEM/SEED flight sample and samples irradiated with AO and UV on the ground showed lower friction coefficients than a reference sample at the beginning of the tests.

3. The mechanism of the decrease in friction of the flight sample was similar to that of the AO-irradiated sample.

Acknowledgments

We appreciate the work of all people involved in the development and operation of the JEM/MPAC&SEED experiment.

References

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