THE EFFECT OF STORAGE CONDITIONS ON THE TRIBOLOGICAL PROPERTIES OF SOLID LUBRICANTS

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Abstract

Satellites, which usually have a lot of solid-lubricated parts, are stored on the ground for a long period before they are launched. During this storage period, degradation of solid lubricants induced by an air atmosphere becomes a major issue. But it is not clear how much influence these storage environments, especially humidity and temperature, have on solid lubricants. In this research, we stored typical solid lubricants for prescribed periods at constant humidity and temperature, and evaluated the effects of storage environments on their tribological properties by conducting pin-on-disk friction tests and XPS surface analysis. Noticeable differences resulted from environmental conditions were obtained.

1. Introduction

Today various solid lubricants are used for mechanical components of spacecraft. They show good tribological performance in vacuum conditions. However, it is known that generally solid lubricants are vulnerable to moisture, which can be a cause of increasing both friction and wear[1][2]. Therefore, proper storage environment must be prepared when they are stored on the ground before they are launched. Actually, for mechanical components coated with solid lubricants, their storing humidity during this period is regulated. In recent years, in some big projects such as the International Space Station program, this storage period reaches several years due to the delay in the launch schedule. But when considering a long storage period, not enough data have been collected to figure out what kind of storage environment we should provide in order to prevent lubricants' functional deterioration, and how much and what kind of influence that storage environment have on lubricants' tribological performance[3].

In this research, we stored disk specimens coated with typical solid lubricants used for space mechanical components for prescribed period at a constant humidity and temperature. Then we evaluated the effect of storage environment on their tribological properties by conducting pin-on-disk friction tests and surface analysis by XPS.

2. Friction Tests

2.1 Specimens

Materials selected for solid lubricating films are bonded MoS_2 (MoS_2-B), magnetron-sputtered MoS_2 (MoS_2-S) and ion-plated silver (Ag-IP). MoS_2-B consists of MoS_2, polyimide-amide binder and a few percent of additives. These materials were deposited on 440C stainless steel disk substrates which were lapped to 0.05 Ra for surface roughness. The characteristics of the films and the photograph of the disk specimen are presented in Table 1 and Figure 1, respectively. Counterpart pin specimens were 6mmϕ 440C stainless steel balls.

Table 1. Tested Solid Lubricating Films

Solid Lubricant	Film Thickness (μm)	Surface Roughness of Films (nm)	
		Mean	Standard Deviation
Bonded MoS_2	10±4	$1.2x10^3$	$2.4x10^2$
Magnetron-sputtered MoS_2	1.0±0.2	32	4.0
Ion-plated silver	0.5±0.2	30	3.2

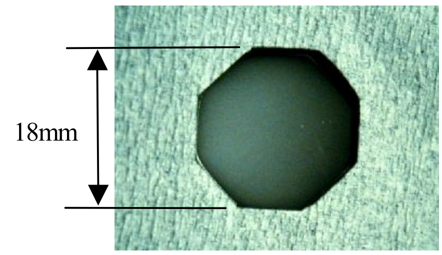


Figure 1. Test Specimen (Magnetron-sputtered MoS_2)

2.2 Test Facilities

"Ultra High Vacuum (UHV) Surface Test Facility", one of "High Vacuum Test Facilities for Mechanical Components[4]", was used for this research. This facility, shown in Figure 2, consists of two test chambers, two surface analyzers and a vacuum transfer system, which is capable of carrying out all the experimental procedures under high vacuum conditions.

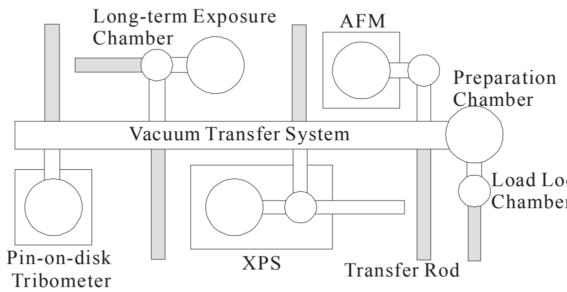


Figure 2. UHV Surface Test Facility

2.3 Testing Conditions

At first, the disk specimens were stored in air-conditioning containers at constant humidity and temperature for prescribed periods. Storage conditions employed were presented in Table 2. Then pin-on-disk friction tests were conducted at room temperature in UHV (< $1x10^{-5}$ Pa).

Load was 10N (the maximum Hertzian contact pressure was approximately 1.2GPa). The sliding speed was constant at 0.2m/s. The friction force was continuously monitored during the tests. The wear life of lubricants was defined to be the number of passes at which the coefficient of friction (COF) rose to 0.3. The summary of testing conditions is listed in Table 3.

Before and after storage, the surface of disk specimen was analyzed by XPS.

Table 2. Storage Conditions

Storage Period	1 month ~ 24 months
Temperature	25°C
Humidity	40%RH, 60%RH, 80%RH

Table 3. Testing Conditions

Friction Mode	Unidirectional Sliding Friction
Load	10N
Sliding Speed	0.2 m/s
Friction Radius	5mm or 7mm
Pressure	Below $1x10^{-5}$Pa
Temperature	Room Temperature (around 25°C)
Number of Passes to Terminate Tests	$2.0x10^6$

3. Results

3.1 Wear Life

Tables 4 and 5 show wear lives of MoS_2-B and Ag-IP, respectively. Long wear lives over 2 million passes in all the storage conditions indicate that both lubricants have good tolerance for humid conditions.

In contrast, wear lives of MoS_2-S varied widely according to storing humidity and storage period. Table 6 and Figure 3 show their wear lives. MoS_2-S without storage (as-received MoS_2-S) had wear lives ranged from $1.55x10^5$ to $2.03x10^5$ passes. Following is the tendency of wear lives of stored MoS_2-S compared with those of as-received MoS_2-S.

(1) Storage Period: 1 month

Wear lives decreased for all storage conditions.

(2) Storage Period: 3 months

Wear life for 40%RH decreased, while wear lives for 60%RH and 80%RH increased. In particular, MoS_2-S stored in 60%RH achieved a long wear life of $8.88x10^5$ passes, which is more than four times as long as that of as-received MoS_2-S.

Table 4. Wear Life of MoS_2-B

		Storing Humidity		
		40%RH	60%RH	80%RH
Storage Period	As Received (No Storage)	over $2.0x10^6$		
	1month	over $2.0x10^6$	over $2.0x10^6$	over $2.0x10^6$
	3months	over $2.0x10^6$	N/A	over $2.0x10^6$
	15months	N/A	N/A	over $2.0x10^6$

Table 5. Wear Life of Ag-IP

		Storing Humidity		
		40%RH	60%RH	80%RH
Storage Period	As Received (No Storage)	over $2.0x10^6$		
	1month	over $2.0x10^6$	N/A	over $2.0x10^6$
	3months	over $2.0x10^6$	N/A	over $2.0x10^6$
	6months	N/A	N/A	over $2.0x10^6$
	12month	N/A	N/A	over $2.0x10^6$

(3) Storage Period: 6 months

MoS_2 stored in 40%RH recovered the same level of wear life as that of as-received. MoS_2-S for 60%RH kept long wear life. Wear life for 80%RH dropped to the same level of wear life as that of as-received.

Elongations of wear life for oxidized MoS_2 and for MoS_2 rubbed in humid air condition have been reported by Fleischauer[5] and Suzuki[6]. They considered that MoO_3, a relatively soft oxide of MoS_2, has contributed to the elongation. Long wear lives after 3-month- and 6-month-storage in our tests may be due to the same reason.

(4) Storage Period: 12 months and more

Tests for 40%RH and 80%RH have been scheduled in future because most Japanese spacecraft are stored in a clean-room environment of less than 60%RH and thus the storage in 40%RH and 80%RH for more than a year are unrealistic. Wear lives for 60%RH scattered. They varied from $2.73x10^4$, 1/6 of that of as-received MoS_2-S, to $2.52x10^5$. Looking into their COF profiles in detail, disorder of COF could be seen around $2x10^4$ passes in every profile (Figure 4). Those sudden changes in COF have not been observed for specimens stored for shorter period at low humidity. The reason for this has not been clear yet, but we infer that at these points high wear of MoS_2-S occurred, which caused the wide variation of wear lives.

Table 6. Wear Life of MoS_2-S

Storage Period	Storing Humidity		
	40%RH	60%RH	80%RH
As received (No storage)	1.78×10^5, 2.03×10^5, 1.55×10^5		
1month	1.46×10^5	1.09×10^5	1.29×10^5
3months	1.30×10^5 1.34×10^5	8.88×10^5	3.14×10^5 2.09×10^5 2.35×10^5
6months	1.81×10^5	8.77×10^5	1.76×10^5
12months	N/A	2.73×10^4 2.52×10^5	N/A
18months	N/A	3.0×10^4	N/A
24months	N/A	2.12×10^5	N/A

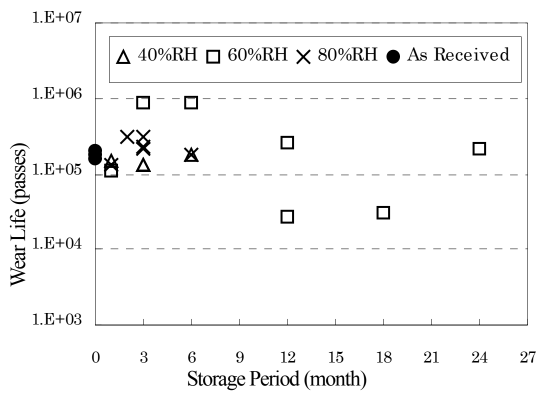
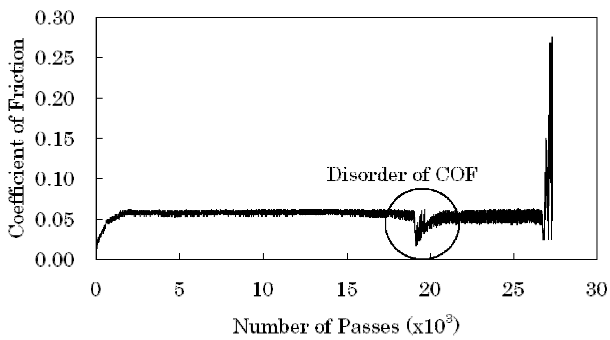
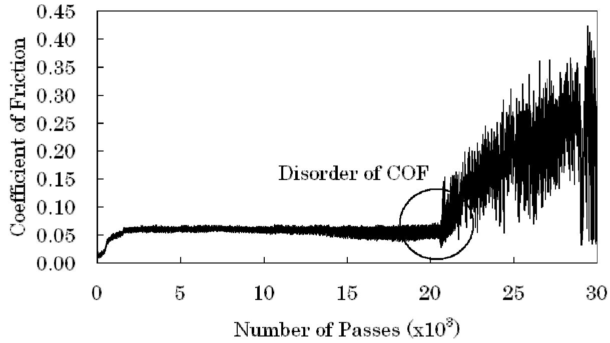


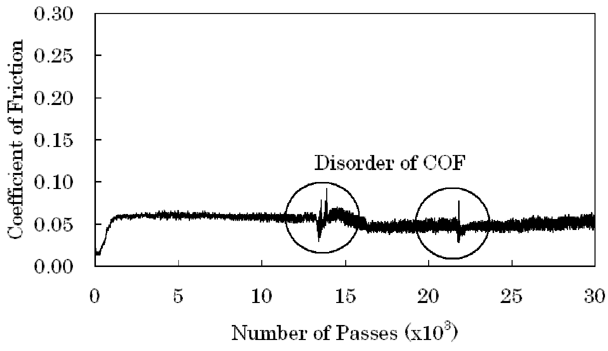
Figure 3. Wear Life of MoS_2-S



(a) Storage Period: 12 months



(b) Storage Period: 18 months



(c) Storage Period: 24 months

Figure 4. COF Profile for MoS_2-S Stored in 60%RH

3.2 Surface Analysis

Figures 5 and 6 show XPS spectra of unrubbed MoS_2-B and unrubbed Ag-IP surfaces, respectively. Five spectra for different conditions are overlapped. Horizontal axis represents binding energy and vertical axis represents signal intensity. Both figures show five overlapped spectra. These two figures indicate that MoS_2-B and Ag-IP are not chemically changed even after being exposed to humid conditions for a long period. The reason for MoS_2-B chemically stable like this seems to be due to antioxidant additives such as SbO_3, and for Ag-IP is that Ag-IP forms a very thin oxide layer of Ag on its surface which prevents more oxidation. These are the reasons why wear lives of both solid lubricants reached 2×10^6 passes in all the conditions.

XPS spectra for MoS_2-S (Figure 7) show existence of MoO_3 on the surface of MoS_2-S stored in humid conditions. The ratio

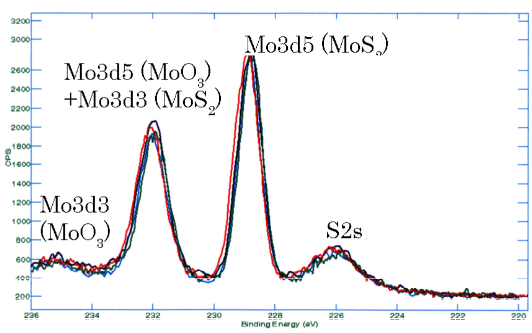


Figure 5. XPS Spectra for MoS_2-B (Mo3d Peak)

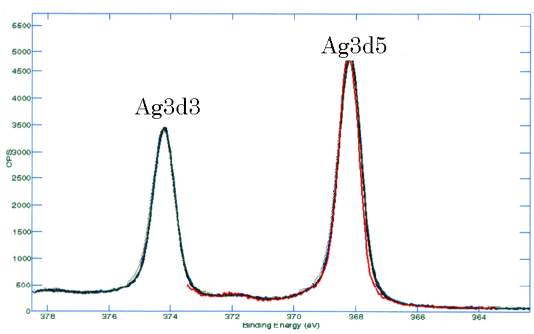


Figure 6. XPS Spectra for Ag-IP (Ag3d Peak)

of MoO_3 to MoS_2 increased as storing humidity and storage period increased.

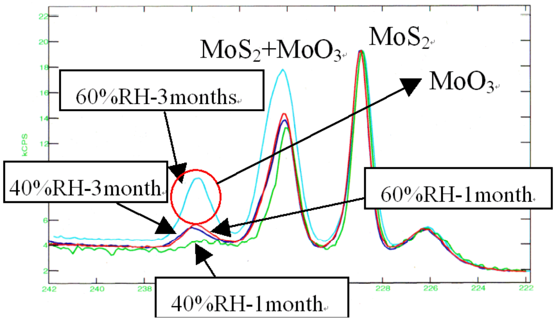
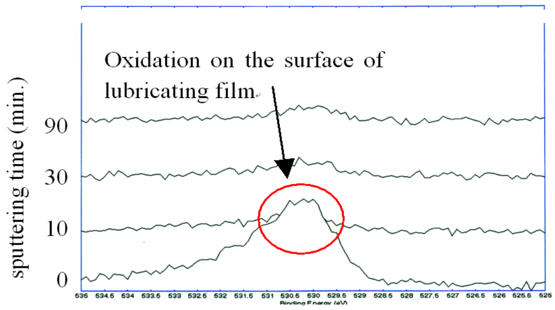
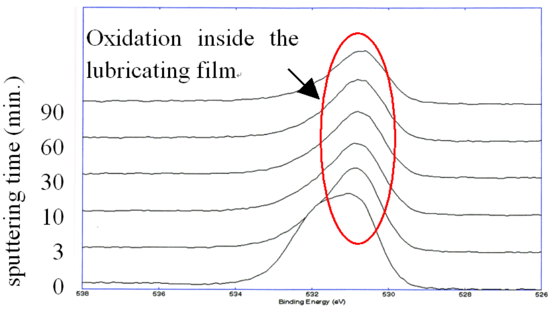


Figure 7. XPS Spectra for MoS_2-S (Mo3d Peak)

To collect more information on the oxidation condition, XPS depth profiling was conducted for MoS_2-S (Figure 8). Sputtering rate was approximately 5nm/min. Figure 8 shows that storage in high humidity for a long time generates high oxidation in deeper level of MoS_2-S. High oxidations were also observed in other spectra of MoS_2-S stored in 60%RH and 80%RH for 3 months and more.



(a) As Received (No Storage)



(b) After Storage for 3 Months in 80%RH

Figure 8. XPS Depth Profiling for MoS_2-S (O1s Peak)

Also iron oxides were found in spectra of MoS_2-S before friction tests (Figure 9). Since they were found before frictions tests, these iron oxides are considered to have come up to the surface from the substrate by diffusion.

As mentioned above, unlike other two solid lubricants, MoS_2-S has a weakness for moisture and is chemically unstable when exposed to moisture.

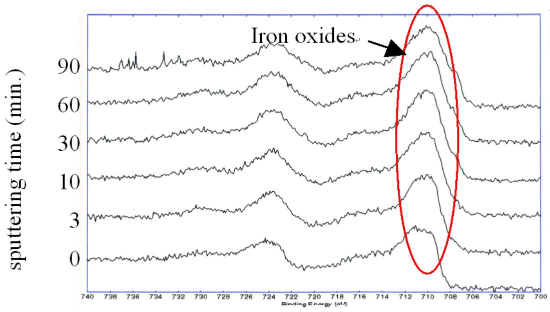


Figure 9. XPS Depth Profiling for MoS_2-S after Storage for 3 Months in 80%RH (Fe2p Peak)

4. Conclusion

In order to clarify the effect of storage environment on tribological properties of solid lubricants, pin-on-disk friction tests and XPS surface analysis were conducted after specimens were stored in air-conditioning cabinet for prescribed periods. The following is a summary of the results:

(1) MoS_2-B and Ag-IP are tolerant to humid storage environments, showing long wear lives of over 2 million passes in all the conditions. These lubricants are chemically stable even after long-term storage in 80%RH.

(2) MoS_2-S is weak for moisture and can easily oxidize in humid conditions.

(3) There are the cases that storing MoS_2-S in high humidity increases their wear lives compared with those of as-received MoS_2-S. The periods of these cases depend on storing humidity.

(4) Exceeding the period described in (3) results in decrease of wear life. In our conditions, wear life can decrease to approximately 1/6 of that of as-received MoS_2-S.

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