WEAR-LIFE OF THE MOLYBDENUM DISULFIDE SPUTTERED FILM UNDER HYPERTHERMAL ATOMIC OXYGEN BOMBARDMENT: IN-SITU WEAR-LIFE EVALUATIONS

Masahito Tagawa[1)]*, Masayuki Muromoto[1)], Syunsuke Hachiue[1)], Kumiko Yokota[1)], Nobuo Ohmae[1)], Koji Matsumoto[2)], and Mineo Suzuki[2)]

[1)] Kobe University, Kobe, Japan
[2)] National Aerospace Laboratory of Japan, Chofu, Japan

**Department of Mechanical Engineering, Faculty of Engineering, Kobe University*
Rokko-dai 1-1, Nada, Kobe, Hyogo 657-8501, Japan
TEL & FAX: +81-78-803-6126, E-mail: tagawa@mech.kobe-u.ac.jp

ABSTRACT

Wear-life of the MoS_2 film was evaluated by *in-situ* tribological testing under 5 eV atomic oxygen exposures which simulate LEO atomic oxygen environment. A combination of a laser-detonation atomic oxygen source and a conventional pin-on-disk friction tester was used to perform tribological tests. It was confirmed that the friction coefficient was not affected by atomic oxygen exposure when atomic oxygen fluence was low; however, the friction coefficient increased with increasing atomic oxygen fluence and it reached as high as 0.05 (seven times larger than the normal value) at the atomic oxygen fluence of 3.4 x 10^{16} atoms/cm^2/cycle. Effect of atomic oxygen on the wear-life of the film has much more drastic. With atomic oxygen fluence of 1.7 x 10^{16} atoms/cm^2/cycle, wear-life of the film was reduced one ninth of the ex-situ testing result of the same film. It was also observed that the wear-life of the film was inversely proportional to the atomic oxygen fluence between every sliding motion.

1. INTRODUCTION

The energetic atomic oxygen reaction with materials used in the exterior of a spacecraft is one of the key issues in future long-life spacecraft. Much attention has been paid to polymer degradation due to atomic oxygen attack in the last decade. Beside polymeric materials used in thermal control systems, lubricants are also one of the key materials to be tested. A lubrication malfunction in a space system influences a serious damage to satellite. In many cases, there exists no back-up system for such tribological failure. Therefore, it should be stressed that the influence of atomic oxygen on lubricants must be evaluated before the mission.

Molybdenum disulfide (MoS_2) is one of the most widely used lubricants in space systems. Its low friction coefficient (typically 0.02-0.05) and durability in vacuum are suitable for space applications. In order to evaluate the effect of hyperthermal atomic oxygen bombardment on MoS_2-based lubricants, some research groups have studied the tribological properties of the atomic oxygen-exposed MoS_2 lubricants. However, such research results were often contradictory: high friction [1] or low friction [2], short wear life [3] or no effect on wear life [1], isotropic oxidation [3] or anisotropic oxidation [1]. These discrepancies may be due to the difference in the experimental conditions in each experiment such as beam energy, fluence, sample preparation and so on. Therefore, it is important to clarify the experimental conditions, including beam characteristics, in order to access the tribological properties of MoS_2 lubricant in an LEO space environment.

Until today, most of the tribological tests regarding atomic oxygen effects have been carried out after atomic oxygen exposure was completed; i.e., post-exposure tests. It is always doubted whether or not ex-situ and in-situ tests give the same results. The in-situ tribological testing results were reported by Wei et al [4]. However, they used a thermal O-atom source wherein the impinging energy of 5 eV is neglected. Recent studies suggested the importance of the role of impinging energy on the surface reaction of atomic oxygen with polymers [5]. Thus, the role of impinging energy of atomic oxygen should also be considered in atomic oxygen effects on lubricants. Moreover, none of these studies reported the wear-life of the film under simulated atomic oxygen environment in LEO (impinging energy and flux).

In this study, we are reporting the first ground-based simulation results on the atomic oxygen effects on the wear-life of the MoS_2 sputter-deposited films in which simulates both impinging energy and flux of atomic oxygen in LEO space environment. Wear-life of the MoS_2 sputtered film under various atomic oxygen testing conditions, including conventional ex-situ and more realistic in-situ testing conditions, were evaluated and compared. The tribological results of the same type of sputtered MoS_2 film flown on STS-85 were also referred.

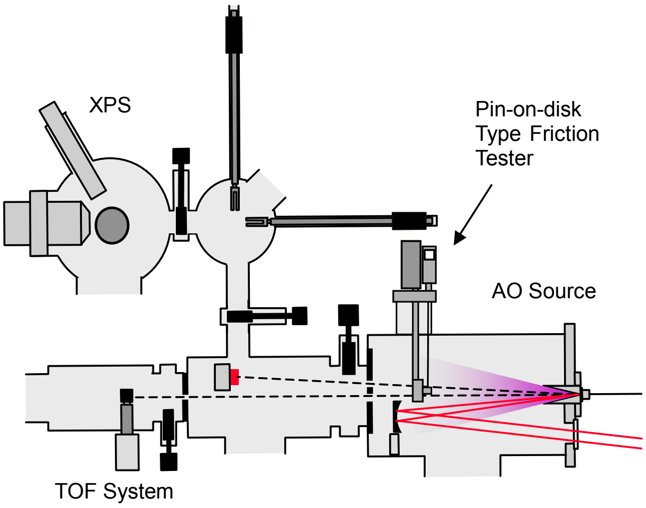

Figure 1. A Schematic drawing of the laser detonation atomic oxygen (AO) beam source used in this study. In-situ friction tester is installed in the AO source chamber.

2. EXPERIMENTS

The MoS_2 specimens used in this study were sputter-deposited MoS_2 Films. The sputtered films were prepared by the radio frequency (RF) sputtering technique at the National Aerospace Laboratory of Japan. Sputtering conditions of the RF sputtered specimens are reported elsewhere [2]. The samples prepared by the same procedure have also been aboard STS-85 and correlated with the ground-based experiments reported here.

The atomic oxygen source used in this study was based upon the laser-induced detonation phenomenon and originally developed by the Physical Sciences Incorporation [6]. The atomic oxygen source was attached to the testing facility developed in our laboratory [7]. The atomic oxygen beam was monitored by a time-of-flight measurement system consisting of a quadrupole mass spectrometer and a multi channel scalar. The mean energy of the hyperthermal atomic oxygen was calculated to be 4.5 eV. The atomic oxygen fraction in the beam was approximately 45 % and balance was molecular oxygen. Atomic oxygen flux of the beam was measured by an Ag-coated quartz crystal microbalance (QCM) with an accommodation coefficient of 1. A typical atomic oxygen flux at the sample position (47 cm from the nozzle) was estimated to be 2.4 x 10^{14} atoms/cm^2/s. Note that atomic oxygen flux is inversely proportional to the square of nozzle-sample distance.

The UHV-friction tester used in this study was especially designed for this facility. This UHV-friction tester is based on the conventional pin-on-disk layout. The UHV friction tester was installed in the atomic oxygen source chamber. The testing conditions are summarized in Table 1.

Table 1. Summary of the tribological test conditions of this study.

Pin	SUS440C ball (3/16 inch)
Disk	Sputtered MoS_2
Load	3.6 N
Sampling frequency	5 Hz
Track length	8 mm
Sliding speed	2.0 mm/s
AO energy	4.5 eV
flux	2.3x10^{14}AO/cm^2/s
repetition rate	1 Hz

3. RESULTS AND DISCUSSION

In a previous study, we have proposed a method for the tribological testing in analyzing the effect of atomic oxygen [8]. One of the points is that the initial high friction of atomic oxygen-exposed MoS_2 lubricants is due not only to the atomic oxygen effect, but also to the resistance of wear track formation at the beginning of friction. It has been demonstrated that the wear track formation before the atomic oxygen exposure is essential to analyze atomic oxygen effect. In this study, a wear track was created before the atomic oxygen exposure such that the effect of initial wear tack formation was eliminated. Indeed, without atomic oxygen exposure, no initial high friction was obvious. In contrast, after the atomic oxygen exposure high friction was observed. Thus, the initial high frictions in this study are attributed to the atomic oxygen effect.

The fluence dependence on the initial friction force of the MoS_2 sputtered film is shown in Figure 2. It was obvious that the initial friction force of MoS_2 film increased with increasing the atomic oxygen fluence on the friction surface. An atomic oxygen of 2.5 x 10^{18} atoms/cm^2 leads to a friction coefficient of more than 9 times larger than the steady-state value. It is therefore concluded that atomic oxygen-induced high friction requires an accumulation of amount of atomic oxygen at the friction track.

When atomic oxygen flux was high enough, not only the initial high friction, but also steady-state high friction was obvious. Figure 3 represents the friction coefficients at the onset of atomic oxygen exposures. In this experiment, repetition rate of the

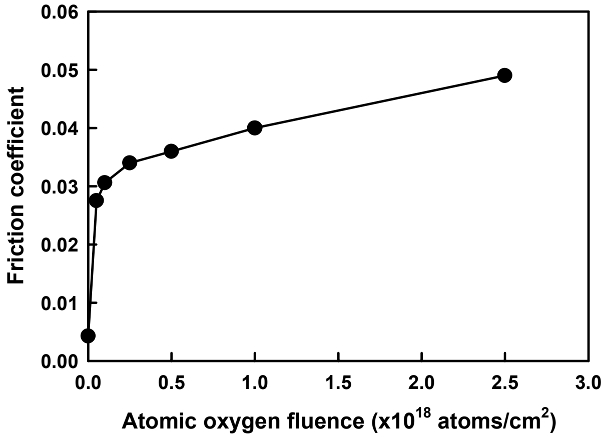

Figure 2. Atomic oxygen fluence dependence of the initial friction coefficient (ex-situ testing results).

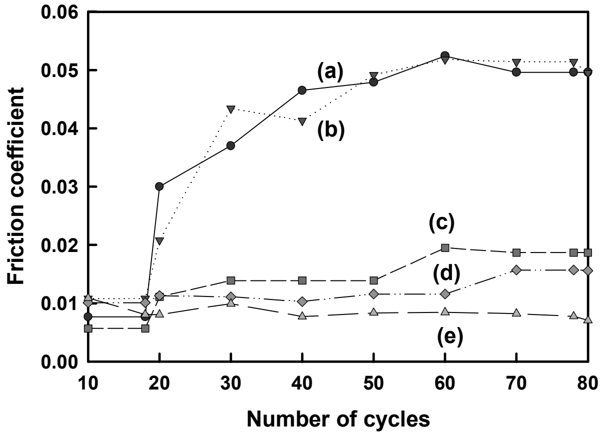

Figure 3. In-situ tribological testing of MoS_2 sputtered film at various atomic oxygen fluences; (a): 3.4 x 10^{16}, (b): 1.7 x 10^{16}, (c): 1.1 x 10^{16}, (d): 5.6 x 10^{15}, (e): 2.0 x 10^{15} atoms/cm^2/cycle. Recovery of friction coefficient is not observed.

sliding motion is reduced to 0.05 Hz so that atomic oxygen fluence between each sliding motion accumulated as high as 10^{16} atoms/cm^2. As is shown in Figure 3, friction coefficient was significantly affected by atomic oxygen exposures when its fluence exceeds 1.7 x 10^{16} atoms/cm^2/cycle. (Note that the unit of atoms/cm^2/ cycle represents the atomic oxygen fluence in a cycle of pin movement.) However, no significant effect of atomic oxygen in friction coefficient was observed in the lower fluence cases (in the order of 10^{15} atoms/cm^2 /cycle). It was also obvious in Figure 3 that the friction coefficient increased gradually with cycles and reached the saturated friction coefficient of 0.05 that is 6 times larger than the pre-exposure value. Friction coefficient reached 0.05 at the 60th cycle which corresponds to atomic oxygen fluence of 1.4 x 10^{18} atoms/cm^2. These atomic oxygen fluence and friction coefficient are identical to those observed in the initial high friction in ex-situ testing [8].

From a series of experiments carried out in this study, it was suggested that accumulation of atomic oxygen is needed to affect friction coefficient of MoS_2 sputtered film. It was also demonstrated that the recovery of friction coefficient due to sliding could be expected, but only in the case of high repetition rate.

Wear-life of the sputtered MoS_2 film was evaluated under the atomic oxygen exposures. Without atomic oxygen exposure, wear-life of the film in ultrahigh vacuum was approximately 50,000 cycles. In-situ wear-life testing, atomic oxygen fluences between sliding were adjusted by changing repetition rate of sliding motion of the pin; atomic oxygen flux was fixed to 2.4 x 10^{14} atoms/cm^2/s. Figure 4 represents the results of wear-life tests of MoS_2 sputtered film. The open circles (b) indicate the friction coefficient of MoS_2 under atomic oxygen exposure of 1.3 x 10^{15} atoms/cm^2/cycle; i.e., in-situ testing results. The wear-life of the films was 34,280 cycle which corresponds to the atomic fluence of 4.6 x 10^{19} atoms/cm^2. In contrast, the solid circles (a) show the result of ex-situ test; i.e., wear-life evaluation was carried out by the same sample after atomic oxygen exposure of 4.6 x 10^{19} atoms/cm^2 was completed. The wear-life by the ex-situ testing was evaluated to be 41,000 cycle, which is 20 % greater than the in-situ testing result. Effects on atomic oxygen fluence between sliding cycle were also evaluated. The solid triangles (c) in Figure 4 represent the in-situ friction test under atomic oxygen

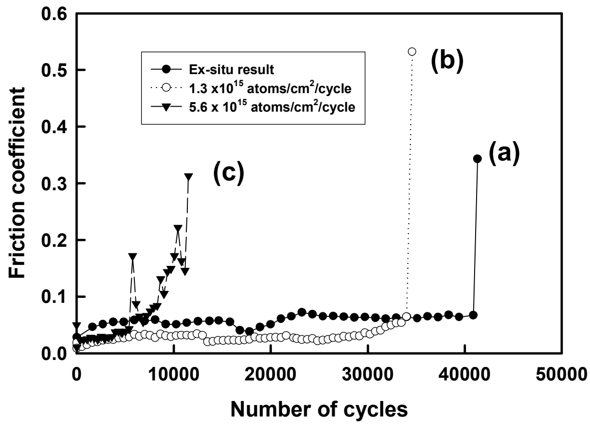

Figure 4. Wear-life of MoS_2 sputtered film under various atomic oxygen exposure conditions. (a): ex-situ testing result after atomic oxygen fluence of 4.6 x 10^{19} atoms/cm^2, (b) in-situ testing with 1.3 x 10^{15} atoms/cm^2/cycle, and (c) in-situ testing with 5.6 x 10^{15} atoms/cm^2/cycle.

exposure of 5.6 x 10^{15} atoms/cm^2/cycle, i.e., atomic oxygen fluence was 4.3 times greater than the case in (b). The wear-life in the case (c) was only 11,340 cycles which was one third of the case (b). It was thus concluded that atomic oxygen effect on MoS_2 was atomic oxygen fluence-dependent; the wear-life is inversely proportional to the atomic oxygen fluence between sliding motion of the pin. This is due to the fact that the atomic oxygen effect is limited at a few tens of nanometers from the surface of MoS_2. These oxide layer, which is consisted of MoO_3 [9], are delaminated by sliding and a certain thickness of MoS_2 was lost by friction.

Similar results were obtained in the flight experiment aboard STS-85. In the ESEM program, same type of MoS_2 film was exposed to a real space environment. The atomic oxygen fluence in orbit was estimated to be 1.0 x 10^{20} atoms/cm^2. The wear-life measurement was made with the same Hertzian contact pressure and similar sliding conditions reported in this ground-based study. The wear-life of the flight and the control samples thus measured were reported to be approximately 50,000 and 40,000 cycles, respectively [2]. In that report, atomic oxygen fluence is twice as large as in this study, but 20 % of shortage of the wear-life was resulted in both studies. This is due to the fact that the oxidation of MoS_2 is restricted only in the surface region (less than 20 nm from the surface) and at the atomic oxygen fluences of 4.6 x 10^{19} and 1.0 x 10^{20} atoms/cm^2 oxidation would be close to saturated thickness [10].

4. CONCLUSIONS

The tribological properties of MoS_2 were evaluated under hyperthermal atomic oxygen beam exposures. Friction coefficient at the beginning of sliding increased with the accumulation of atomic oxygen fluence. An atomic oxygen of 2.5 x 10^{18} atoms/cm^2 leads to a friction coefficient of more than 9 times larger than the normal value. When atomic oxygen flux was high enough, not only the initial high friction, but also steady-state high friction was obvious. It was also evident that the atomic oxygen effect on wear-life of sputtered MoS_2 film was atomic oxygen fluence-dependent; it was inversely proportional to the atomic oxygen fluence between each sliding motion of the pin. This is considered due to the termination of oxide thickness at the MoS_2 surfaces that were delaminated by the friction.

ACKNOWLEDGMENTS

A part of this study was supported by the Hyogo Science and Technology Association; by the Gant-in-Aid of Scientific Research from the Ministry of Education, Culture, Sports Science and Technology, Japan; and by the Space Utilization Research Promotion from the Japan Space Forum.

REFERENCES

1. Arita M., Yasuda Y., Kishi K. and Ohmae N., Investigation of Tribological Characteristics of Solid Lubricants Exposed to Atomic Oxygen, *Proceedings of STLE/ASME Tribology Conference*, St. Louis, **91-TC-3D-1**, 1991.

2. Matsumoto K., Suzuki M., Imagawa K., Okada Y. and Tagashira M., Evaluation of tribological characteristics of sputtered MoS_2 films exposed to LEO environment, *Proc. 21st Int. Symp. Space Technology and Science*, Omiya, Japan, **98-b-30**., 1998.

3. Martin J. A., Cross J. B. and Pope L. E., MoS_2 Interactions with 1.5 eV Atomic Oxygen, *Materials Research. Symposium Proceedings.,* **140**, pp.271-276, 1989.

4. Wei R., Wilbur P. J., Bughholz B. W. and Kustas F. M., In situ Tribological Evaluation of Greases and Solid Lubricants in a Simulated Atomic Oxygen Environment, *Tribology Transaction*, **38**, pp.950-958, 1995.

5. Kinoshita H., Tagawa M., Umeno M. and Ohmae N., Surface reaction of a low flux atomic oxygen beam with a spin-coated polyimide film: Translational energy dependence on the reaction efficiency, *Transactions of the Japan Society for Aeronautics and Space Sciences*, **41**, pp.94-99, 1998.

6. Caledonia G. E., Krech R. H. and Green B. D., High flux source of energetic oxygen atoms for material degradation studies, *AIAA Journal.*, **25**, pp.59-63, 1987; U.S. Patent No.4894511 (16, January, 1990).

7. Kinoshita H., Ikeda J., Tagawa M., Umeno M. and Ohmae N., A Fast Atomic Oxygen Beam Facility with In-situ Testing/analysis Capabilities, *Review of Scientific Instruments*, **69**, pp.2273-2279, 1998.

8. Tagawa M., Ikeda J., Kinoshita H., Umeno M. and Ohmae, N., Effect of Atomic Oxygen Exposures on the Tribological Properties of Molybdenum Disulfide Lubricants, *Protection of Space Materials from Space Environment*, Kleiman J., and Tennyson R. C., Eds., Kluwer Academic Publishers, pp.73-84, 2001.

9. Tagawa M., Kinoshita H., Umeno M., Ohmae N., Matsumoto K. and Suzuki M., Ground-based tribological testing of the sputter-deposited molybdenum disulfide films in hyperthermal atomic oxygen exposure, *Proceedings of 8th European Space Mechanisms and Tribology Conference*, Toulouse, France, **ESA SP-438** pp.291-296, 1999.

10. Tagawa M., Yokota K. and Ohmae N., unpublished.

Back to Contents