# **Evaluation of Solid Lubricative Coatings after Space Environment Exposure Test**

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Moving components materials in orbit environment require surface modification with stable lubrication for preventing increase in friction due to oxidation and irradiation damages. We have prepared a stainless steel substrate and four kinds of lubricative coatings such as TiN, MoS2, mixture of Cu and BN and Cu on the substrates and installed them on the International Space Station in orbit for exposure test from about a year to three years. We have analyzed tribological properties, surface roughness, chemical concentration and so on. Most of a year exposed substrates generally decrease friction in a vacuum as well as at an atmospheric pressure and over years exposure increased friction of almost substrates. XPS analysis shows Si based contamination layer that might affect the change in tribological properties.

Keywords: MPAC&SEED, ISS Service Module, coatings, friction, vacuum, TiN, MoS2, mixture of Cu and BN, Cu

### 1. Introduction

Orbit environment about 400km above the ground of a space shuttle and a space station is an ultrahigh vacuum under 10<sup>-5</sup> Pa and usually suffers from atomic oxygen attack at a high speed as well as some particles irradiation. Moving components materials therefore require surface modification with stable lubrication for preventing increase in friction due to oxidation and irradiation damages. After preparation of a stainless steel substrate and four kinds of such lubricative coating films as titanium nitride, molybdenum disulfide, mixture of copper and boron nitride and copper on the stainless steel substrates, these substrates have been exposed in orbit for a year ,two years and three years. Analyses have been carried out on tribological properties, surface micro roughness, structure, chemical concentration and so on in order to understand change in properties of lubricant coating by the exposure test in orbit and to develop surface modification for advanced smooth and reliable space solid lubrication.

### 2. Experimental

Commercial type 304 austenitic stainless steel sheet substrates (size; 14 mm x 14 mm x 1mm) have been coated with titanium nitride (TiN), molybdenum disulfide (MoS<sub>2</sub>), mixture of copper and boron nitride (Cu/BN) and copper (Cu) using a rf magnetron sputter deposition system with a film thickness about 50nm to 250nm [1] and were set on a panel then were installed onto the SM/MPAC&SEED (Service Module/ Micro-Particles Capturer and Space Environment Exposure Device) prepared by JAXA on the Russian service module in the International Space Station (ISS), then have been exposed in orbit and have recovered to the Earth.

The frictional properties of the substrates with and without exposure to orbit were observed with a system developed for measuring vacuum friction before and after annealing in a vacuum of  $10^{-5}$  Pa or less level. The design of the friction measurement system comes from the Bowden-Leben type as shown in Fig.1 [2]. The sample stage can move in x-y direction by two axes stepping motor and a manual axis in an ultrahigh

vacuum. A stainless steel ball or sapphire ball prove above the substrate holder has a load weight holder with 0.49N and has two strain gauges to detect frictional force.

Surface chemical characterization for the substrates was carried out on TiN coated substrate after exposure test for a year with a X-ray photoelectron spectroscopy with X-ray of excited from Al K  $\alpha$  (1486.6eV), constant pass energy of 69eV, X-ray beam size of 100  $\mu$  m in diameter and so on. Depth profiles of the substrates were also obtained with XPS using an argon ion sputtering.



Fig.1 Schematic view of a vacuum friction measurement system and photo of a center system.



### 3. Results

#### 3.1. Friction properties

Figure 2 shows the effect of vacuum annealing on vacuum friction coefficients of coatings on substrates with a variety of exposure test years. A year exposure test made substrates keep almost low coefficient of friction ( $\mu$ ) even after vacuum annealing but two years exposure test made some substrates increase  $\mu$ . Three years exposure test makes obvious difference among substrates in increase in  $\mu$ . SUS304 substrate, TiN coating and Cu coating show a large increase in  $\mu$  and MoS2 coating shows little increase in  $\mu$  keeping good lubrication in spite of long time exposure in orbit.

### 3.2. Surface analyses

Surface chemical condition and depth profile were obtained with a X-ray photoelectron spectroscopy for two surface areas of TiN coated substrates, one for exposure and the other for screening against exposure test in orbit for a year.



Fig.2 Effect of vacuum annealing on vacuum friction coefficients ( $\mu$ ) of substrates with a variety of exposure term; (a) SUS304 substrate, (b) TiN coating, (c) MoS2 coating, (d) Cu coating,(e) mixture coating of Cu and BN.

Figure 3 shows survey spectra of exposed and screened stainless steel sheets coated with TiN. Silicon (Si) peaks and large oxygen (O) peaks are observed on the exposed area while large fluorine (F) peaks are observed on screened area of the TiN coated substrate. Figure 6 shows depth profiles of exposed and screened area of the substrate coated with TiN. Silicon and oxygen concentrated layer is observed at the surface of TiN coating layer at the exposed area in orbit.



Fig.3 Survey spectra of exposed area of TiN coated substrate.

in a vacuum.

Si2p

12000





layer that can offer smooth and stable sliding against annealing

Si2p

Fig.4 Depth profiles of a year exposed (A) and screened area (B) of TiN coated substrates.

Changes in chemical condition were obtained for elements composing TiN coated stainless steel sheets after exposure in orbit as shown in Fig.7 with main binding energies [7]. Titanium forms TiN with nitrogen mainly and also forms  $TiO_2$  with oxygen in part. Silicon forms  $SiO_2$  with oxygen at the surface of TiN coating layer.

Comparison between these surface analytical results of exposed and screened area of TiN coated stainless steel sheet indicates as follows. TiOx layer formed during TiN film preparation. Silicon evaporated from silicone adhesives used for space station structure might react the atomic oxygen to form  $SiO_2$  during exposure in orbit. The mixed structure of  $SiO_2$  in coating film is considered to form a good lubricant

Fig.5 Changes in XPS spectra for silicon and oxygenwith sputter time for obtaining depth profile

### 4. Conclusion

Lubricative coatings such as TiN, MoS2, mixture of Cu and BN and Cu were prepared on the stainless steel substrates and installed them on the International Space Station in orbit for exposure test from about a year to three years. Most of a year exposed substrates generally decrease friction in a vacuum as well as at an atmospheric pressure but over years exposure increased friction of almost substrates except MoS2 coating. XPS analysis shows Si based contamination layer that might affect the change in tribological properties and furthur analyses are required to understand the detail mechanism on change in friction by exposure test in orbit.

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