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Results of Space-Environment Exposure of the Flexible Optical Solar Reflector

Eiji Miyazaki* and Ichiro Yamagata†
Japan Aerospace Exploration Agency, Ibaraki 305-8505, Japan

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This paper presents exposure results for the flexible optical solar reflector, a sample material in the International Space Station Service Module/Micro-Particles Capturer and Space Environment Exposure Device experiment mission. The flexible optical solar reflector, which has a conductive layer and a mirror layer coated on a polyetherimide base film, is a thermal control film material for spacecraft. It achieves low solar absorptance and high infrared emittance. Results indicate a mass increase: the flexible optical solar reflector was not eroded by atomic oxygen in low Earth orbit. Thermo-optical properties show no significant change. In fact, flexible optical solar reflector is confirmed to retain its initial properties after exposure on an International Space Station orbit for 46 months. Transmission electron microscopy observation of the cross sections including the exposed surface showed that a new layer had formed over the flight sample. Qualitative analysis of the new layer indicates that the layer mainly comprises silicon and oxygen. The layer is chemically produced by deposited silicone contamination and atomic oxygen, which would be SiO_2. This experiment also provides actual quantitative contamination data on the International Space Station, contributing to improvement of contamination control on the International Space Station in the future.

I. Introduction

MATERIALS, mostly organic materials, that are used on spacecraft are likely to be damaged by exposure to a space environment. Surface characteristics (e.g., optical properties and/or bulk characteristics such as mechanical properties) can be altered by such damage. The factors inducing such damages are known to include thermal cycling, radiation, ultraviolet (UV) rays, and atomic oxygen (AO) in low Earth orbit (LEO) [1–3]. To understand the tolerance of candidate materials for use in a mission to space environments, ground tests have been developed and have been applied to the materials. However, cases have been documented in which the ground-test results did not match exposure results obtained in an actual space environment [4]. Therefore, on-orbit materials exposure experiments were performed to elucidate the effects of a real space environment on materials and to verify the materials' tolerance of the space environment [5–9].

Japan Aerospace Exploration Agency (JAXA) has conducted experiments under the Service Module/Micro-Particles Capturer and Space Environment Exposure Device (SM/MPAC&SEED) since October 2001, exposing materials to space on the exterior of the Russian service module of the International Space Station (ISS) [10,11]. It is a unique aspect of the SM/MPAC&SEED mission that three identical sets of samples with different exposed durations are obtained. Each experimental unit includes 3 sample types for the MPAC experiment and 28 sample types for the SEED experiment. Each set was retrieved at a different time: unit 1 was retrieved in August 2002, unit 2 in February 2004, and unit 3 in August 2005. We thereby obtained three sample sets with different exposure periods.

This paper specifically presents the results of the flexible optical solar reflector (F-OSR) installed on SM/MPAC&SEED as 1 of the 28 SEED samples. The F-OSR is a thermal control film for spacecraft such as satellites, which achieves low solar absorptance α_S and high infrared emittance ε with flexibility. The F-OSR is a five-layered and second-mirrored film with a polyetherimide base film. The F-OSR has a UV protection layer to protect the base film from UV degradation. The SM/SEED experiment is appropriate to investigate whether the function can be maintained for a year-long order period on orbit. For evaluation, the retrieved F-OSR samples were evaluated to determine their changes of mass and thermo-optical properties. Topographic analyses and qualitative analyses were also performed. Herein, we present results of those evaluations for the F-OSR and discuss the influence of the space environment of the ISS orbit on the F-OSR for up to 46 months.

II. Experimental

A. Exposure Experiment on the ISS

1. *SM/MPAC&SEED Experiment Mission*

The exposure experiment was carried out in the SM/MPAC&SEED mission. The mission consists of three identical units that were installed on the outside wall of the Russian service module in ISS, as shown in Fig. 1. Samples were installed on both sides of the unit, which were facing fore and aft of the ISS. The fore-facing side is called the *ram side*; the aft-facing side is the *wake side*. Two F-OSRs were installed on the ram side of each unit. The respective appearances of the ram side of the SM/MPAC&SEED unit and the location of the F-OSR installed on the SM/MPAC&SEED unit are shown in Fig. 2. The dimensions of SM/MPAC&SEED unit are 570 mm wide by 875 mm high by 158 mm deep.

In the original mission design, respective SM/MPAC&SEED units were planned for exposure to space of 1 year, 2 years, and 3 years on the ISS. Table 1 describes the actual exposure durations: approximately 10, 28, and 46 months, respectively. The experiment units were launched by the Progress on 21 August 2001 and installed on the outside wall of the SM on 15 October 2001. Each set was retrieved separately by crew extravehicular activities from outside of the ISS and brought into the ISS. Inside the ISS, the sample trays were removed from the SM/MPAC&SEED frame and stowed into the special return cassette for return to the ground by Soyuz.

2. *Exposure Environment*

SM/MPAC&SEED is a completely passive mission with no electric power used for on-orbit operation of the experiment.

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*Engineer, Space Materials Section, Advanced Materials Group, Aerospace Research and Development Directorate, 2-1-1 Sengen, Tsukuba; miyazaki.eiji@jaxa.jp.

†Currently Engineer, Materials Monitoring Section, Fuels and Materials Department, Oarai Research and Development Center, Japan Atomic Energy Agency; yamagata.ichiro@jaea.go.jp.

Fig. 1 Photograph of SM/MPAC&SEED installed on the ISS.

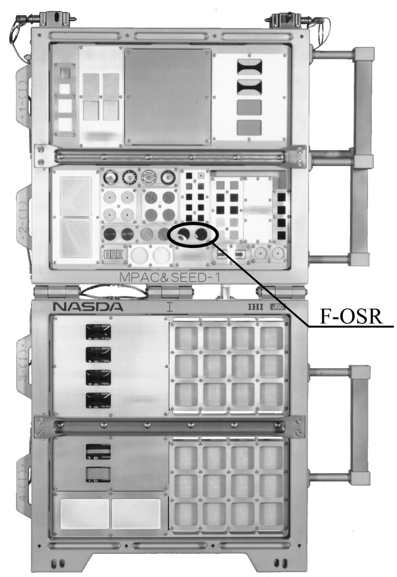

Fig. 2 SM/MPAC&SEED unit (ram side) and the location of the F-OSR.

Therefore, environmental monitoring is carried out using seven kinds of passive monitoring techniques that operate without a power supply. The individual passive monitoring devices and samples are installed along with SEED samples on the sample tray. Monitoring objects include materials and devices that provide passive records of maximum temperature, AO fluence, UV fluence, and total ionizing radiation dose. After retrieval from the space environment and return to the ground, the monitoring samples are evaluated to determine the cumulative values for their exposed conditions. The names of monitoring samples and their measured results of unit 1 and unit 2 are summarized in Table 2 [12]. Data from unit 3 are still under investigation and will be given in a future paper. Details of monitoring samples and their evaluation methods are described in other papers [12,13].

B. Sample Preparation

The F-OSR, manufactured by Sumitomo Bakelite Co., Ltd., is a film with multilayered coatings for thermal control of spacecraft such as satellites. As schematically illustrated in Fig. 3, the F-OSR has five layers: a conductive layer, an UV-ray protection layer, a base film polyetherimide, a mirror layer, and a corrosion protection layer. The maximum usage temperature of the F-OSR is 150°C. The base film polyetherimide is UV-degradable; for that reason, CeO_2, which is known to be UV-absorbent, is deposited. Silver is used as a second surface mirror; the layer is covered completely by Ni alloy to prevent corrosion. With such features, F-OSR achieves low solar absorptance α_s and high infrared emittance ε with good flexibility.

The F-OSRs for SM/MPAC&SEED used a 100-μm-thick base film and were cut into 25-mm-diam circles, which were mounted on the sample holder with a frame opening a 20-mm-diam window for exposure. The mass and thermo-optical properties of the F-OSR provided for the SM/MPAC&SEED experiment are mass= 48.5 mg, $\alpha_S = 0.156$, and normal infrared emittance $\varepsilon_N = 0.812$. Two samples (A and B) were mounted on each set (1–3); that is, six samples (1-A–3-B) were put on the ISS.

C. Evaluation Items

For mass measurement, we used a microbalance (readability 1 μg, maximum load 5100 mg, MX5; Mettler Toledo International, Inc.). Solar absorptance α_S was measured using a JAXA custom-ordered spectrophotometer (NASDA-PSPC-7844; Jasco, Inc.). The value of α_S is calculated by measured spectral reflectivity of a sample of 250–2500 nm and solar spectral irradiance [14]. Normal infrared emittance ε_N was measured using a total-emittance reflectometer (DB-100; Gier Dunkle Instruments, Inc.).

The cross-sectional microstructure was observed using transmission electron microscopy (TEM). The samples stained using RuO_4 were sliced by ultramicrotomy to make a cross section including the exposed surface. To obtain microstructural information, TEM observation was performed at the acceleration voltage of 100 kV and magnification of $\times 200,000$. Scanning TEM and energy dispersion x-ray analysis (STEM-EDX) was conducted to elucidate the qualitative analysis of small areas (approximately 1 nm diameter) in regions of interest found in the cross-sectional images.

III. Results and Discussion

A. Mass

The mass changes of exposed samples are depicted in Fig. 4, which shows the relationship between exposed duration and actual mass change. The results indicated a slight mass increase of less than 0.1 mg for all retrieved samples with 48.5 mg initial mass. In addition, the mass change shows a tendency of increasing concomitant with the exposure duration: that is, 0.04 mg gain for 10 months exposure, 0.07 mg gain for 28 months, and 0.09 mg gain for 46 months. In general, the AO-attacked polymer material is eroded, resulting in mass loss. For instance, the third-retrieved AO

Table 1 Exposed durations of SM/MPAC&SEED (GMT denotes Greenwich Mean Time)

Unit	Exposed duration, days	Launch (GMT)	Exposure		Landing (GMT)
			Beginning (GMT)	End (GMT)	
1	315	21 Aug. 2001	15 Oct. 2001	26 Aug. 2002	10 Nov. 2002
2	865			27 Feb. 2004	30 Apr. 2004
3	1403			18 Aug. 2005	11 Oct. 2005

Table 2 Summary of monitoring materials of SM/MPAC&SEED [12]

	Maximum temperature, °C	AO fluence, atoms/cm^2		UV fluence, ESD[a]	Total ionizing dose, Gy[b]		
Unit	Thermolabel[c]	Polyimide Vespel	PAMDEC[d]	Polyurethane	Alanine dosimeter	RADFET[e]	TLD[f]
1	50	1.7×10^{20}	2.4×10^{21}	18.1	1.95	0.44	1.46×10^{-3}
2	50	2.1×10^{20}	1.9×10^{21}	15.8	15.3	5.99	0.12

[a]Equivalent solar day, 1 ESD = 1.02×10^7 J/m^2.
[b]Gray, 1 Gy = 1 J/kg.
[c]Measured at approximately 5 mm below the exposed surface of tray 2, in which F-OSRs were installed.
[d]Passive atomic-oxygen monitoring device equipped with carbon film.
[e]Radiation-sensitive field-effect transistor.
[f]Thermoluminescent dosimeter.

monitor polyimide sample Vespel lost 4.087 mg during exposure [13]. The F-OSR data suggest that erosion does not occur; that is, it is verified that F-OSR has sufficient AO tolerance for almost 4 years on the ISS orbit. On the other hand, such a mass increase suggests absorption or deposition of some substances during exposure. That point is discussed later using the results of cross-sectional analysis.

B. Thermo-Optical Properties

The changes of thermo-optical properties of the exposed samples during exposure are depicted in Figs. 5 and 6. No significant change of thermo-optical properties was observed; that is, α_S increased 0.01 from the initial value of 0.156 for 46 months' exposure, which is almost entirely included in the uncertainty of the measuring device. For ε_N, less than 0.005 changes were observed from the initial value of 0.812 for 46 months on orbit, which is also included in the uncertainty of the equipment. Such a small change suggests that F-OSR has UV and radiation tolerance. Lack of UV protection might induce a color change of the base film, thereby changing α_S.

However, some papers have described recovery: the degraded optical properties that have changed because of the space-environment exposure are subsequently recovered by exposure to air [15,16]. In the present mission, it cannot be determined whether recovery might have occurred or not. Because of the retrieval procedure, air exposure before measurement on the ground is unavoidable. Compared with other SEED samples of the F-OSR, several samples show changes that are visible on the exposed surface. Although they might have recovered, such visible changes persist even after long exposure to air on the ground. These results suggest that F-OSR can retain its thermo-optical properties of beginning of life for 46 months on the ISS orbit.

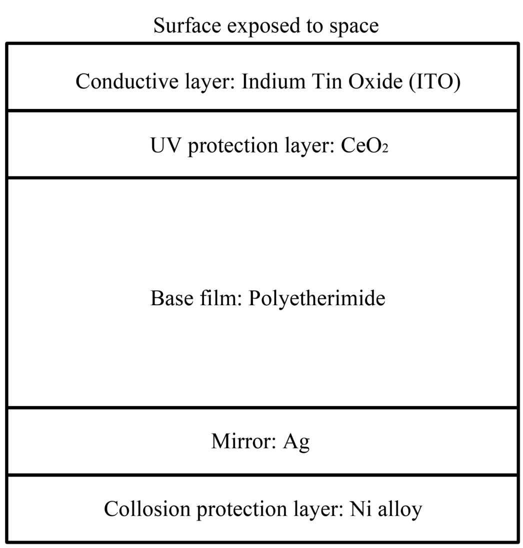

Fig. 3 Cross-sectional illustration of the F-OSR.

C. Cross-Sectional Analysis of the Exposed Surface

Cross-sectional TEM images of the near exposed surface are shown in Fig. 7. In the figures, the dark area appearing in the lower

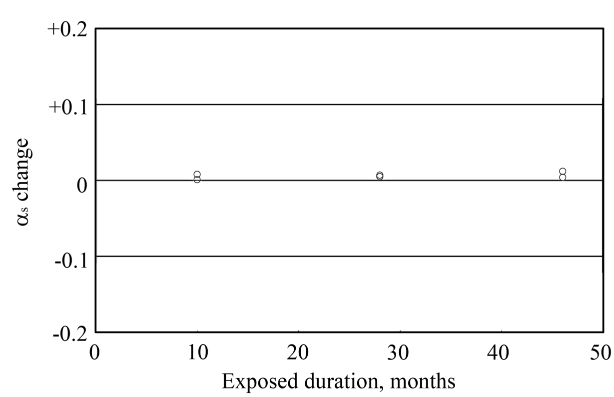

Fig. 5 Results of solar absorptance α_S change of retrieved F-OSRs from SM/MPAC&SEED exposure.

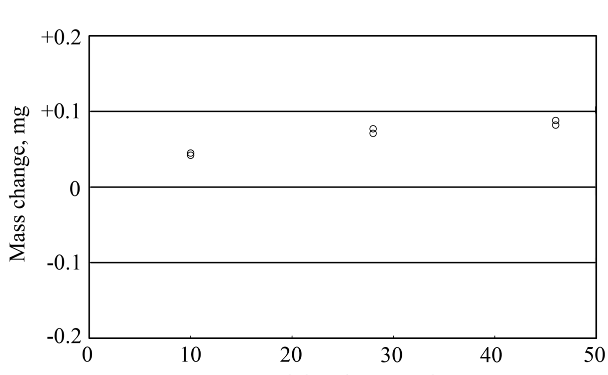

Fig. 4 Results of mass change of retrieved F-OSRs from SM/MPAC&SEED exposure.

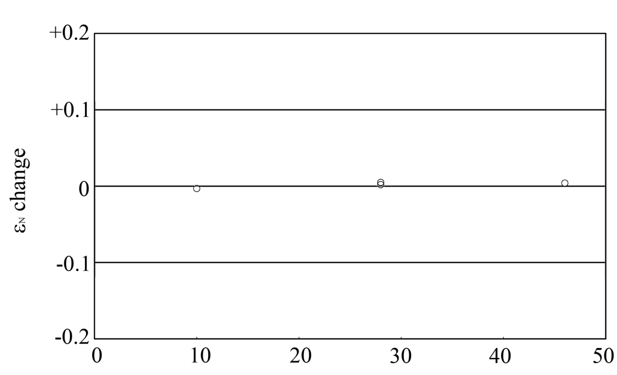

Fig. 6 Results of normal infrared emittance ε_N change of retrieved F-OSRs from SM/MPAC&SEED exposure.

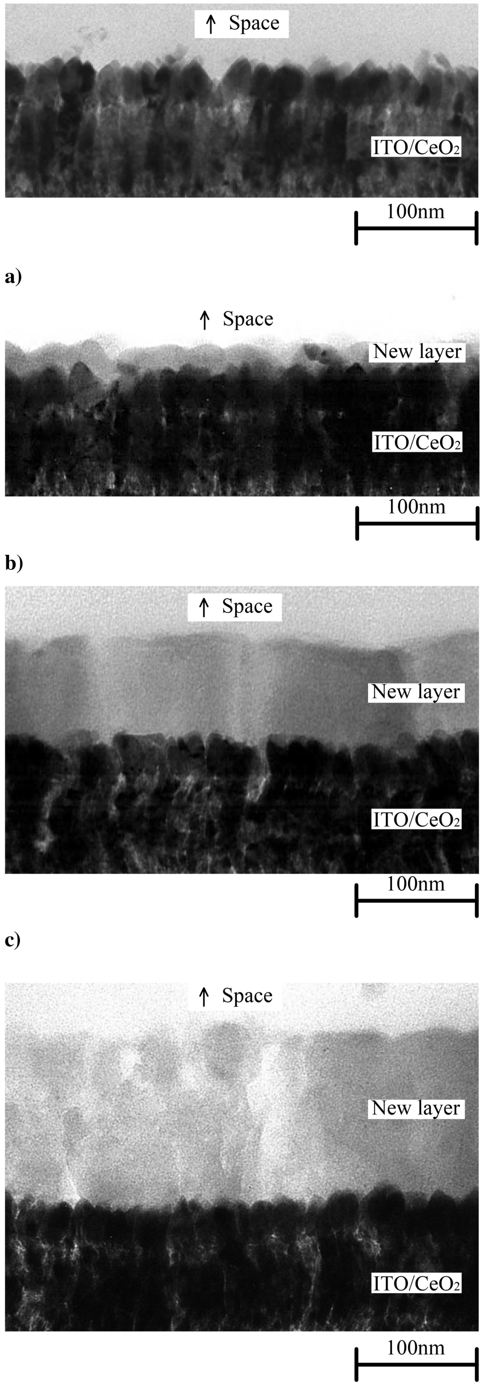

Fig. 7 Cross-sectional TEM images of the F-OSRs for SM/MPAC&-SEED of a) blank sample, b) sample of unit 1, c) sample of unit 2, and d) sample of unit 3.

half is the ITO/CeO_2 layer; the bright area appearing in the upper half is a mount material for TEM observation that is not a part of the F-OSR. These areas can be seen in both blank sample and retrieved samples, whereas a gray area is found between the dark area and the bright area only in retrieved-sample images. The gray area would be a new layer, which was built during exposure and covered the flight samples. For the sample of unit 1 shown in Fig. 7b, the thickness of the new layer can be estimated as approximately 20 nm. No obvious change was observed on the original surface (i.e., the border between the new layer and ITO/CeO_2 layer), compared with the surface of the blank sample. The surface shape of the new layer, the border between the gray area and bright area, is found to trace that of the original F-OSR's ITO/CeO_2 layer's surface. The cross section of the sample of unit 2 is shown in Fig. 7c: the thickness of new layer is approximately 80 nm, and the original surface is kept its original shape. The shape of the new layer surface does not trace finely, but smoothly. Figure 7d shows the cross section of the sample of unit 3: the thickness is approximately 120 nm, the surface of the new layer is smooth, and the original surface indicates no obvious difference from its initial shape.

In general, the AO-attacked surface shows a distinctive shape with μm-sized asperity, a so-called *carpet* shape or *needlelike* shape. The F-OSR indicated neither apparent mass loss nor an AO-attacked shape at the original surface observed at a magnification of nanometer-scale resolution. From the view, F-OSR might have tolerance against AO attack on the ISS orbit. Though there is a possibility that the new layer might play a role to protect the surface from AO attack, such protection was not observed in the result that the other retrieved polyimide films installed on the same SM/MPAC&SEED unit were damaged by AO [17]. Thus, the tolerance of the F-OSR itself against AO attack is confirmed to be sufficient enough.

Then, to examine the new layer, we performed STEM-EDX analysis in terms of investigating the layer qualitatively. For the analysis, the electron beam was focused around the center of the new layer in the depth direction, as shown in Fig. 8. Consequently, the result shows that carbon, silicon, and oxygen are detected, which is commonly observed in any retrieved F-OSRs. The sample 3-A spectrum is shown as representative in Fig. 8. First, it could not be determined whether the detected carbon was derived from the exposure on orbit. Carbon was also detected in the ITO/CeO_2 layer, though the layer that was made by deposition at manufacturing should not contain carbon. Second, silicon and oxygen detected on the new layer are discussed. Because F-OSR contains no silicon, the silicon must originate from sources other than F-OSR (e.g., paints for thermal control or adhesives used for solar arrays containing silicon). Outgassed silicone from such sources would deposit on the other components on the ISS, including SM/MPAC&SEED. Silicone deposition is one of known phenomena occurring in spacecraft on orbit [5]; in the SM/MPAC&SEED mission, it was also observed and reported previously [18]. Oxygen would originate from AO existing around the ISS orbit. The deposited silicone on the surface of the F-OSR would be oxidized by collision with AO, resulting in production

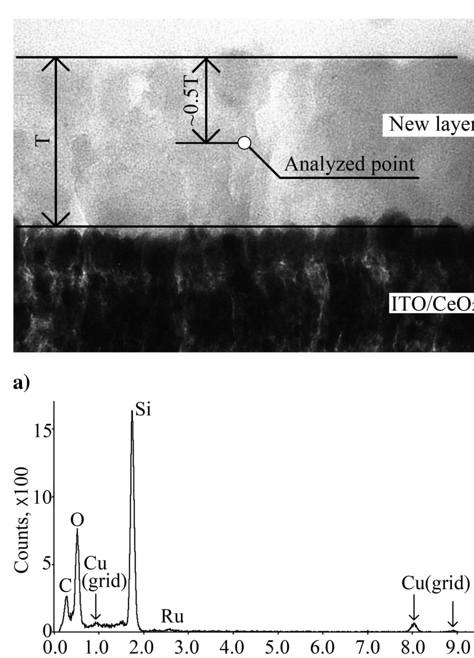

Fig. 8 STEM-EDX analysis of the new layer on F-OSRs for SM/MPAC&SEED: a) details of analyzed point and b) the spectrum of EDX for sample 3-A.

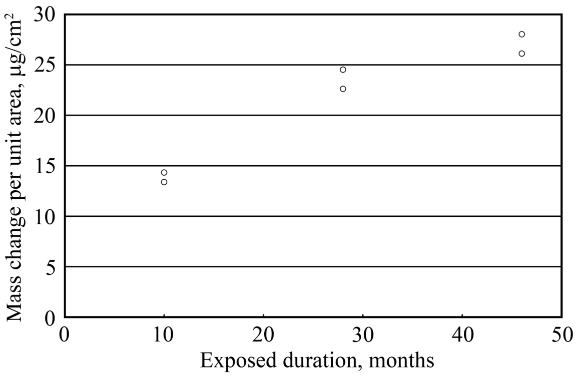

Fig. 9 Relationship between the measured mass change per unit area of the F-OSRs and the exposed duration for SM/MPAC&SEED.

of silicon dioxide (SiO_2). The process of silicon deposition and oxidation would happen continuously on orbit.

Both mass measurements and thickness measurements of the new layer discovered by cross-sectional observation using TEM revealed mutual agreement, suggesting that the mass increase is the result of new layer deposition. Figure 9 shows the relationship between the measured mass increase per unit area and the exposure duration: the deposition is expected to become saturated as the exposure duration lengthens. This result suggests that such saturation might result from the outgassing rate behavior of the contaminant source [19]. Contamination deposition rate can be calculated from the present result. Average rates are 1.7×10^{-5} g/cm^2/year for sample of unit 1, 1.0×10^{-5} g/cm^2/year for sample of unit 2, and 7.1×10^{-6} g/cm^2/year for sample of unit 3. It is higher than the requirement values, which are less than 1.0×10^{-6} g/cm^2/year at 300 K surface for molecular deposition [20,21]. Obtaining actual measured data will contribute to the improvement of contamination control for ISS and other spacecraft in the future.

IV. Conclusions

The F-OSR was exposed to the space environment as one SEED sample of the SM/MPAC&SEED mission carried out on the ISS for 46 months from October 2001. The retrieved samples were investigated for their mass change and thermo-optical properties. Cross-sectional TEM observation and STEM-EDX analysis were also performed.

A slight mass gain of less than 0.1 mg was observed for all retrieved samples, showing that F-OSR has tolerance against AO (i.e., erosion does not occur). The thermo-optical properties retained their initial values, although these results might also reflect recovery by exposure to air; nevertheless, beginning-of-life thermo-optical properties of the F-OSR were retained for 46 months in ISS orbit. Cross-sectional TEM observation revealed that a new layer, which does not exist on the blank sample, was built up on the exposed surface of the F-OSR. The new layer thickness increased concomitantly with the exposure duration. A STEM-EDX analysis reveals that the new layer consists mainly of Si and O, which suggests that the new layer is silicon dioxide that is chemically produced from silicone contamination and AO.

We conclude that the experiment described in this paper verified F-OSRs' tolerance to the low-Earth-orbit space environment around ISS for an exposure period of nearly 4 years. In addition, the experiment provides actual quantitative contamination environmental data on an exterior surface of the ISS, contributing to improvement of contamination control for ISS and other future spacecraft.

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J. Minow
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