Contamination Growth Observed on the Micro-Particles Capturer and Space Environment Exposure Device

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Peer postretrieval observation of the three Micro-Particles Capturer and Space Environment Exposure Device units revealed the growth and distribution of external contamination. All units were remarkably contaminated. Their surfaces had turned a uniform brown color and had numerous colored spots. The contamination conditions differed between the ram and wake faces and were affected by their respective exposure durations. X-ray photoelectron spectroscopy and time-of-flight secondary ion mass spectroscopy analyses were performed to determine the atomic contents and contamination conditions. Comparison of the three units indicated how the contamination layer grew on the external surface in the low-Earth-orbit environment. Although contamination on the ram face expanded continuously at the rate of 39–18 nm/year, the contamination layer thickness observed on the wake face was very thin and remained virtually unchanged.

Introduction

THE Japan Aerospace Exploration Agency (JAXA) deployed three identical Micro-Particles Capturer and Space Environment Exposure Device (MPAC&SEED) units for two passive experiments on the exterior of the Russian service module (SM) on the International Space Station (ISS) [1]. The three units were retrieved individually, after 315, 865, and 1403 days of exposure. To capture micrometeoroids or debris, very lightweight materials such as silica aerogel and polyimide foam were mounted on the both faces of the MPAC units. The SEED exposed 28 organic and inorganic samples to investigate their performances on low Earth orbit. The overall dimensions of one MPAC&SEED unit are W 570 mm, H 875 mm, and D 158 mm. Each unit consists of four sample holders which are compact enough to be retrieved via extra vehicular activity.

Contamination Environment of Service Module/ Micro-Particles Capturer and Space Environment Exposure Device

The SM of the International Space Station is located on the aft end, where visiting Russian vehicles dock. External contamination from vehicle thruster plumes and propellant purges had been predicted, in addition to outgassing from organic materials on the service module Micro-Particles Capturer and Space Environment Exposure Device [2]. The ram faces of SM/MPAC&SEED units point toward the ISS velocity vector when the ISS flies in the *X* axis in the velocity vector. The ISS spent more than 40% of its time in other attitudes while SM/MPAC&SEED was onboard; the ram and wake face orientations also changed [3].

Pankop et al. performed induced contamination analyses of the ISS, including MPAC&SEED [4]. The field of view from and to

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MPAC&SEED was analyzed using an ISS geometric model, indicating that a large fraction of the view from the ram face was occupied by the SM, the SM solar array, the functional cargo block, and docked vehicles such as Soyuz and Progress. Only the SM and Progress docking on the SM aft port entered into the view from the wake face. They estimated that more contamination on the wake face was induced by the thruster rather than by outgassing. In contrast, the predicted thruster-induced contamination on the ram face was negligible [4].

Contamination of external surfaces is a complicated process that is generated by molecular deposition, crosslinking induced by ultraviolet (UV) radiation, and reaction with and erosion by atomic oxygen (AO). Passive space environment monitors were mounted on the MPAC&SEED to measure the total dose of AO, UV, space radiation, and the temperature.

Thermal analysis predicted the maximum temperature as 102°C , and minimum as -105°C at the ram surface. Only the maximum experienced temperatures were recorded using passive thermal labels on orbit. Temperature indicator 8E-50 (Nichiyu Giken Kogyo Co., Japan), mounted 1 mm under the ram surface, recorded 90°C . The maximum temperatures were $50\text{--}60^{\circ}\text{C}$ at other measurement points, 5 mm under the ram surface. The observed temperatures suggest that the thermal analysis estimated the on-orbit temperature almost precisely.

Contamination Observations

Optical Observation

All retrieved trays had brownish surfaces. The different coloration among units with three different exposure durations was not significant (Fig. 1).

On each unit, the wake face was a much darker color than the ram face (Fig. 2). The white doughnut around the fastener hole was covered by a fastener head on orbit. The brown color of the contaminated areas was deeper at the rim of the white doughnut.

The aerogel tile surfaces were darkened, with a roughened texture [5]. Originally, it was expected that the flat and semitransparent texture of the aerogel surface make the visual investigation easy. At the postretrieval observation, we found that the aerogel on the wake face suffered much greater damage than that on the ram face. The cracks on the crispy altered surfaces were increased by exposure duration. Even the aerogel on the ram face had a rough surface after extended exposure.

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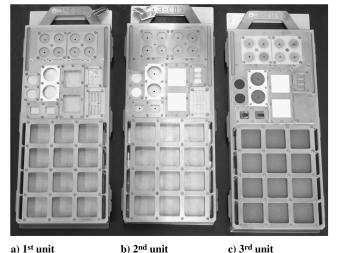
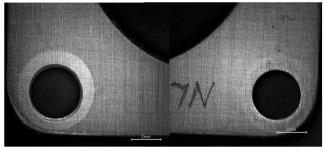


Fig. 1 Retrieved SM/MPAC&SEED units.



a) Wake b) Ram

ig. 2 Aluminum plates removed from the third retrieved unit.

The numerous colored spots found on the samples were roughly able to be categorized into 1) white clouds, 2) brown clouds, 3) brown spots, 4) gray spots, 5) black flakes, and 6) others.

X-Ray Photoelectron Spectroscopy and Time-of-Flight Secondary Ion Mass Spectroscopy Analysis

Contamination of the aluminum plates that had been mounted on both the ram and wake faces of the three units was analyzed using x-ray photoelectron spectroscopy (XPS) and time-of-flight secondary ion mass spectroscopy (TOF-SIMS). The plates, which were used to fix the SEED samples, were made of A6061-T6 alloy with MIL-A-8625 type I anodic coating. The coating must be completely sealed because it has a porous structure. Cobalt or nickel acetate are often used in a sealing agent.

First, XPS analyses were performed to obtain the atomic composition of the top surfaces using Quantera SXM (Physical Electronics, Inc.). The analyses points were selected from uniformly contaminated areas and colored spots on each plate. Moreover, XPS depth profiling using Ar-ion etching was performed on the uniformly contaminated parts, thereby revealing the internal structure of the contamination. The sputtering rate was calculated using measurement data obtained by etching with a standard sample of SiO₂ film.

TOF-SIMS analyses were also performed using TFS-2000 (Physical Electronics, Inc.) on the uniform contamination to facilitate identification of the contaminant molecules: 15 kV Ga+ was used as the primary ion; the raster size was $80 \times 80~\mu m$.

Analysis Results

Uniform Contamination

The XPS analysis results are presented in Fig. 3. The analyzed area was 200 $\,\mu{\rm m}$ in diameter and less than 10 nm deep. Therefore, the detected elements are inferred to include both terrestrial and on-orbit contamination.

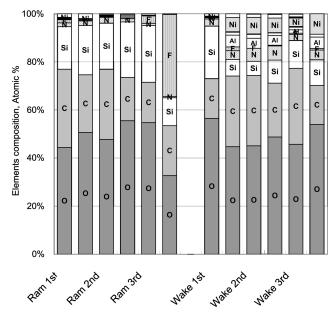


Fig. 3 Elements composition of uniform contamination analyzed by XPS (at. %).

At all analysis points on the ram and wake faces, O, C, Si, N, Al, and Ni were commonly detected. About 70% of the carbon appeared as CHx. Others were C-O, C=O, and COO. In addition, Zn, F, Na, S, Ca, Sn, P, K, and Pb were found on some analysis points.

In general, more Si was detected from the ram face; the wake face had more Al and Ni. Depth profile charts revealed the balance of Si, Al, and Ni. A thick SiOx layer covered the original surface on the ram face, whereas the SiOx layer on the wake face was very thin (Fig. 4). Therefore, components of an aluminum anodic coating and Ni from the sealing agent appeared immediately below the very top surface, even after 1403 days' exposure.

Nitrogen concentrations on the wake faces were considerably higher than on the ram faces (Fig. 3). Whereas N does not often appear in outgassed molecules, thruster plumes contain a certain amount of nitrogen-bearing substances produced from the propellants. This supports the prediction of thruster plume-induced contamination [4]. Sulfur concentrations were 1.1–1.6% on the wake faces, but S was scarcely detectable on the ram faces, for unknown reasons.

Layered Structure of Contamination

A C-rich layer, which was less than 2 nm thick, covered the uppermost surfaces of all specimens. The discontinuation of the depth profile suggests that the layer was the result of terrestrial contamination (Fig. 4). The next layer, which was rich in Si- and O-, was considered to be a SiOx contaminant layer that was produced by AO reaction with siloxane. Within the SiOx layer, the ratio of Si to O was 2.01–2.30. Binding energy of -Si2p and -O1s corroborate the identification of the layer as SiO₂. The layered structure closely resembled the contamination noted at the Passive Optical Sample Assembly I, one of the Mir environmental effect payloads, postretrieval analysis [6]. Subsequent TOF-SIMS analysis detected fragmentary ions of polydimethylsiloxane (PDMS) on both ram and wake faces of all three units. This result suggests that the SiO₂ layer was formed with Si originating from PDMS contamination effused from silicone products.

Growth of the Contamination Layer

Figure 5 indicates the contamination layer thicknesses of the three units observed by XPS depth profiling. The boundary surface between the contamination layer and the aluminum plates was not sharply defined because of the porous structure of the anodic coatings on the aluminum plates. We considered the depth at which the Si

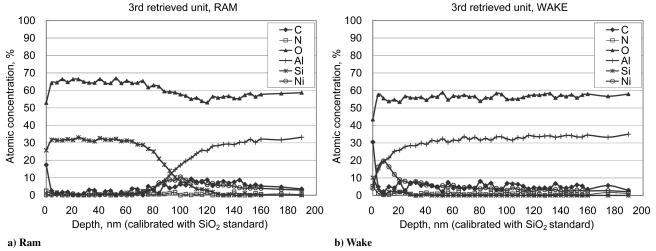


Fig. 4 Depth profiles from XPS analyses of the third unit.

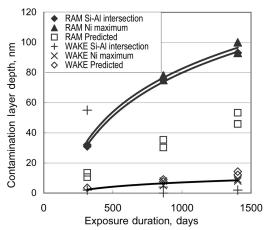


Fig. 5 Contamination layer growth with the exposure duration.

concentration exceeded Al to be the contamination layer thickness. The existence of Ni also indicates the position of the original surface. The Ni that appeared between the SiO₂ layer and Al-O layer is attributable to nickel acetate in the sealing agent for the anodic coating. The maximum Ni concentration appeared at almost the same depth with the crossing of Si and Al concentration curves (Fig. 4).

The SiO₂ layer was observed more clearly on the ram face; its thickness increased in relation to the exposure duration (Fig. 5). This observation indicates a continuous supply of siloxane, even after a long duration of exposure. About 30 vehicles visited and docked to the ISS while MPAC&SEED was onboard. The vehicles were constructed by flesh materials and considered to have effused more outgassing. From February 2003 to July 2005, the later half of MPAC&SEED exposure, no module nor truss segment was attached to the ISS, owing to suspension of the space shuttle flight. The formation of the SiO₂ layer on the ram faces slowed as time progressed, perhaps because of a reduction of the outgassing rates of the source materials on orbit. One analyzed area on the wake face of the first unit had much thicker contamination than other areas on the wake face. This local characteristic was inconsistent with the others; the reason is unclear.

Distribution of the Contamination

Formation and growth of the SiO_2 layer was also observed on several SEED samples and monitoring samples [3,7,8]. These SiO_2 layer thicknesses were measured using transmission-electron microscope (TEM) or scanning TEM-electron energy loss spectroscopy (STEM-EELS). TEM analyses were performed using H-7100FA (Hitachi, Japan), with an acceleration voltage of $100 \, \text{kV}$.

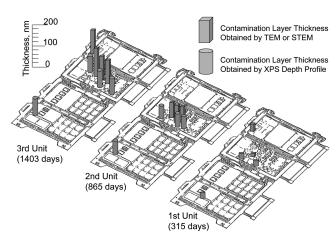


Fig. 6 Contamination thickness observed on the ram face.

The samples were prepared by ultramicrotome and RuO_4 stained. Figure 6 summarizes the contamination layer thicknesses observed on the three units. The SiO_2 layer thicknesses obtained by the TEM cross-sectional observation were 120–140 nm on the third retrieved samples. Though the TEM reported more thicknesses than XPS, the difference in analytical methods prevents equal comparison among the data. XPS depth resolution decrease related to the ion sputter time, because ion etching increased the surface roughness, and the etching rate must be affected by the sputtering angle. On account of the surface roughness of the samples, XPS depth resolution is not supposed to be as good as TEM for thicker contamination layer analysis. One data set obtained by a method indicates that the SiO_2 layer on a unit has almost even thickness for different positions. Boeing also predicted that the contamination thickness distributes within an 8 nm difference on a unit [4].

One sample from the ram face of the third retrieved unit had significantly more fluorine than the other samples (Fig. 3). In the analyzed area, 75% of the carbon was combined with F. The depth profile indicated that an F- and C-rich top layer, 6–7 nm thick, covered the next SiO_2 layer. TOF-SIMS analysis depicted the fluorine distribution.

Figure 7 presents a comparison of the major fluoride ion intensities that were analyzed using TOF-SIMS. The measured ion counts were normalized by the total count. Intensities of the CF^+ , CF_3^+ , $C_2F_4^+$, and $C_2F_5^+$ peaks were remarkably strong on the third retrieved unit compared to the first and second units. Negative ions of fluorides, such as CFO^- , $C_2F_5O^-$, and $C_3F_5O_2^-$, also had strong intensities on the third unit. They might be fragments of perfluoropolyether or polytetrafluoroethylene [9], etc. The intensities of the fluoride ions varied widely among the four analyzed areas on the third unit. This

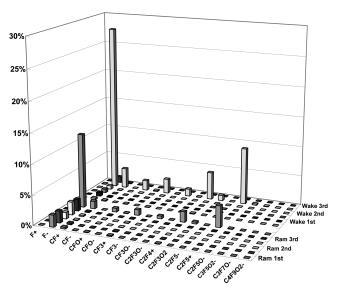


Fig. 7 Normalized fluoride ion spectra analyzed by TOF-SIMS.

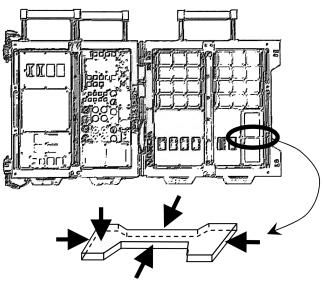


Fig. 8 XPS analysis points on an aluminum plate.

diversity suggests that fluorine was distributed unevenly within the same face of the third unit.

Five points were chosen from an aluminum plate on the wake face for XPS analysis to determine the contamination distribution on a smaller scale (Fig. 8). Contamination observed on the three sides was almost identical to that on the top, in spite of their different lines of sight. One side, closely faced to the frame that held the tray on orbit, presented a unique composition. This side had much more C and F, instead of O and Si. The XPS results depict the centimeter-scale unevenness in fluorine distribution within a small area, as observed on the ram face using TOF-SIMS.

The ESA Matroshka experiment facility is one possible source of outgassing fluorocarbon. Matroshka was covered by Teflon and was deployed in front of MPAC&SEED 18 days after the second unit retrieval. However, Matroshka's effects cannot fully explain the local unevenness within the third retrieved unit.

Spot Contamination

Numerous colored spots were detected on the retrieved units. They were of various shapes and colors; their diameters were of 1–1000 μ m. Though full attribution is not achievable, XPS analyses provided information to assume their origins. Some irregular shaped spots had unique elements, such as Fe or Pb. Spots that contain much

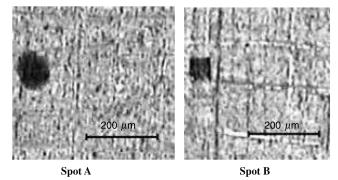


Fig. 9 Brown round spots on the wake face of the third retrieved unit.

more C binding F were assumed to be organic fluorides. Zinc oxide is a typical white pigment and widely used for thermal control paint. Paint chips were possible origins of the spots containing more Zn than normal surfaces. PO_4^{2-} and K^+ were found in a spot. Because these ions were considered to be of human origin, the water dumped from the transport vehicles was a possible source.

Among the spots, roughly circular spots were considered to be formed by the impact of low-velocity droplets. There was a known source of the droplets: thruster plume. Some of the spot contaminations would be produced by the thruster plume impingement. It is possible that the thick molecular contamination layer had covered up the particles and/or liquid droplet substances on the ram faces. Accordingly, we selected two similar brown, rounded spots from various shaped/colored spots on the wake face of the third unit (Fig. 9) and analyzed their element compositions using XPS. Table 1 presents the elemental compositions of the spots. The nitrogen concentration was remarkably greater than on normal surfaces. The two compositions of the spots were very similar, except for the F and N concentrations. The time of the droplet impingement is unknown. The F concentration would vary according to the thickness of the molecular contamination layer covering the droplet. There would be sufficient time for a droplet deposited long before retrieval to develop an F-rich molecular contamination layer that covers it.

Discussion

Contamination Thickness Versus Color Change

Although the contamination layers on the ram faces were much thicker than those on the wake faces, the brown color on the wake faces was deeper than that on the ram. One reason for this might be the magnitude of UV irradiation. Two passive UV monitors were mounted on both the ram and wake faces of each unit. Unexpected results were obtained from the calibration results of the monitoring samples. Data from the second unit suggest that the wake face was irradiated by 13 times more UV than the ram face [3]. The UV fluence on the MPAC&SEED is also calculated using the ISS geometric model and attitudes. Predicted UV fluence on the wake face was 6 times more than on the ram face. These results differ in their magnitudes. However, they are consistent with the optical appearances of the contaminated surfaces.

Thruster-induced contamination is another possible cause of the color difference. Brown coloration, which is inferred to be the result of thruster plume impingements, has been observed commonly on

Table 1 Element composition of the brown spots analyzed by XPS (unit, at. %)

	С	N	О	F	Al	Si	Ni	Zn	S
Spot A	45.2	23.1	22.6	2.4	1.1	2.9	2	0.3	0.4
Spot B	45.4	10.2	24.8	11.1	1.3	4.2	1.6	0.7	0.7
Normala	24.2	4.1	49.1	0.6	3.3	11.1	5.2	0.4	1.1

^aThe element composition of the normal surface shows the average of the wake surfaces from the three units.

Table 2 Contamination depth predicted [4] and measured (unit, nm)

		Unit 1	Unit 2	Unit 3
Ram	Predicted	10.6–13.5	30.3–35.4	45.9–53.3
	Measured	31, 32	75, 75	93, 93
Wake	Predicted	3.5	7.7–9.1	12.1–14.3
	Measured	2, 55	0, 4	2, 2

the Mir space station, the space shuttle, and the ISS. Boeing predicted that the wake face would have more thruster-induced contamination than the ram face [4]. The higher nitrogen concentration observed on the wake face confirmed the prediction (Fig. 3). The darker color on the wake face is attributable to the thruster plume.

Contamination Layer Growth

The observed contamination layer thicknesses on the wake faces were much less than the predicted values. The cause of this difference is explainable by the process of contamination layer formation.

Although C had greater concentrations at the top surfaces, the contamination layers observed on the ram faces were formed mostly from only Si and O. The most likely scenario is selective reaction of Si with AO. In fact, SiO₂ formation under vacuum-UV (VUV) irradiation is reported often [10]. Because VUV electromagnetic radiation has more energy than the binding energy of C-H or Si-C, VUV separates the methyl group from PDMS [11]. Remaining Si sites bind to AO and form rigid SiO₂. Other atoms, such as C, were rejected or formed volatile molecules, and did not remain on the surface. Although the molecular deposition rate on a cryogenic surface depends strongly on the surface temperature, the maximum temperature recorded on the SM/MPAC&SEED was 60–90°C; sufficiently high to be free from light-molecule deposition.

This scenario indicates that the permanent contamination layer thickness depends on the mass of Si in the contaminants, not on the total mass of contaminants. Table 2 summarizes levels of the outgassing-induced contamination predicted by Boeing [4]. The figures do not include thruster plume impingement, which does not contribute to SiO_2 formation. The predicted value agrees with the measured SiO_2 layer thickness of the order.

However, no significant difference is visible in fragmentary ion counts of PDMS between the ram and wake faces. It suggests that the wake face also had the siloxane supply.

The difference in AO radiation still poses an eminently plausible hypothesis, although it has not been confirmed by the AO flux measurement on MPAC&SEED. Two passive AO monitors of different types were mounted on both the ram and wake faces [3]. They indicated that AO irradiation on the ram face was 1.3–1.8 times greater than on the wake face. This difference seems to be too small when compared with the observed contamination thicknesses. The AO monitors measure mass loss and the change in electric resistance by AO erosion. However, a thick SiO₂ layer was also noted on the AO monitors on the ram face. Data must be revised to accommodate effects of deposits of contaminants and/or a shielding effect. The AO fluence on the imaginary plane on the ISS ram end was calculated using the MSIS-86 model in JAXA-developed Space Environment and Effects System (SEES).[‡] The ISS attitude changes were considered in the analysis. SEES predicted 10 times or more higher fluence than measured values. Even though the analysis did not take into account the screening by the ISS elements, the net AO fluence on the ram face could be much more than the measured value, and explain the difference in SiO₂ layer thicknesses.

The observed SiO₂ layer thicknesses did not depend on the contaminated surface material. In fact, STEM analysis presented a crosscut image of a graphite sample that shows that the SiO₂ layer did not conformally trace the original surface. Figure 10 was obtained using HF-2210 (Hitachi, Japan) with an acceleration voltage of 200 kV. Many voids were found under the SiO₂ layer covering

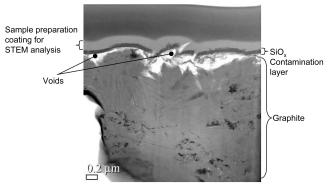


Fig. 10 Voids observed between ${\rm SiO_2}$ layer and the original graphite surface.

coarse surfaces, such as the graphite AO monitor or the AO eroded polyimide. This observation suggests that the SiO_2 formation process does not require absorption of silicone contaminants on a surface. Under such a SiO_2 forming environment, contamination proof surface modifications may not be effective to prevent the contamination layer growth.

Thruster Plume Impingement

Bipropellant thrusters on the ISS and visiting vehicles are used for reboost and attitude control. Fuel/oxidizer reaction products (FORP) are produced by an unsymmetrical dimethylhydrazine and nitrogen tetroxide reaction [12]. The Energia and the Keldysh Center investigated contamination of FORP by ground experiments and sample collection from the Mir space station [13]. They obtained very close figures for the final gross formula of an organic part of FORP, that is, $C_1H_{3.1}N_{0.8}O_{0.1}$ by a ground test and $C_1H_{2.3}N_{0.8}O_{0.1}$ by analysis of samples collected on orbit. Comparing the analyzed data, the elemental composition of the brown circular spots had sufficient compatibility to be attributable to FORP.

Conclusions

We retrieved three identical SM/MPAC&SEED units after different exposure durations. The units were covered uniformly with molecular contamination and had numerous colored spots. Contamination was much thicker on the ram faces than on the wake faces, and increased continuously with the exposure duration. XPS depth profiling revealed that the uniform contamination had a layered structure. Under the thin uppermost layer, which is considered to be terrestrial contamination, a Si- and O-rich layer followed. Within this SiO₂ layer, other atoms were barely detected. XPS and TOF-SIMS analysis identified the elements contained in both uniform and spot contamination. Siloxane, which forms rigid SiO₂ under AO radiation, was considered to be the dominant contaminant. The SiO₂ layer was formed without surface deposition, even on the voids of coarse surfaces. Severe UV irradiation and the repeated thruster plume impingement could cause the deep brown color specifically noted on the wake face. Brown spots found on the wake faces were presumed to have been formed by FORP impact.

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[‡]Details available at http://sees.tksc.jaxa.jp.

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