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Degradation of Mechanical Properties of Polyimide Film Exposed to Space Environment

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Polymer materials used in spacecraft are susceptible to space environmental effects, which degrade not only their surface characteristics but also their mechanical properties. For application as structural materials, for example, as a base film for deployable structures, understanding of the degradation of mechanical properties is particularly important. Tension-applied polyimide films were exposed to the space environment of low Earth orbit using the Microparticles Capturer and Space Environment Exposure Device on the International Space Station Russian service module. After space exposure for approximately one and two years, mechanical properties of the flight samples were evaluated using tensile tests and were compared with degradation behaviors of samples that had been irradiated, respectively, by atomic oxygen, ultraviolet radiation, and electron beams. Results showed that tensile strength and elongation of the flight samples decreased concomitantly with increased exposure duration. Of the factors, atomic oxygen attack was considered to be a main degrading factor in a space environment; the samples exhibited no marked degradation of mechanical properties by either ultraviolet or electron beam irradiation. The tension (below 7.0 MPa) that had been applied to the samples had no marked effect on the degradation of the flight samples or any irradiated sample.

Nomenclature

A_K	=	exposure area of the atomic oxygen fluence monitor, Kapton H, 3.14 cm^2
A_s	=	exposure area of samples
E_K	=	erosion yield of the atomic oxygen fluence monitor, Kapton H, 3×10^{-24} $cm^3/atom$
F	=	atomic oxygen fluence
Δm_K	=	mass loss of the atomic oxygen fluence monitor, Kapton H
Δm_s	=	mass loss of the samples
Δt_s	=	thickness loss of samples
ρ_K	=	density of the atomic oxygen fluence monitor, Kapton H, 1.42 g/cm^3
ρ_s	=	density of samples

I. Introduction

THE factors causing degradation of polymer materials in a space environment include radiation, which are ultraviolet (UV), solar flare x rays, solar wind electrons, and protons trapped in Earth's magnetic field, in addition to temperature variations greater than 200°C, orbital thermal cycling, micrometeoroids, and orbital debris. Furthermore, in low Earth orbit (LEO), atomic oxygen (AO) collides with exposed materials at high velocities, about 8 km/s, thereby eroding their surfaces. Because of such *AO attacks*, the exposed surfaces of polymer materials are transformed into a *needlelike* surface [1]. Polymers used for spacecraft suffer chemical and physical damage from these space environment effects, which alter surface characteristics and degrade mechanical properties.

Of all polymer materials, polyimide has considerable resistance to high temperatures and large operating temperature ranges; it also has a large tolerance against intense UV and radiation. For those reasons, polyimide has been applied predominantly to thermal control films attached where they are exposed directly to a space environment. In addition to these applications, polyimide films have been used as construction materials for deployable structures because of their high specific strength and rigidity, high dimensional accuracy, and low rate of thermal expansion, such as the base film of solar sails and large flexible solar paddles for the International Space Station (ISS) or artificial satellites [2–5]. Especially on the outermost layer film of multilayer insulation (MLI), it is important to estimate the influence of space-degrading factors on the thermooptical properties of the exposed surface. To design a reliable deployable structure for spacecraft, recognizing the degree of mechanical property degradation in a space environment is indispensable. In addition, for application as a structural material, polyimide films are used under tension to maintain their structural shape at low gravity [2–5]. Where applied, tension hastens degradation of mechanical properties of structural materials; structural design necessitates consideration of the tension impacts. However, experiments to investigate degradation of the mechanical properties in exposed polyimide films, including tension-applied ones, have not been conducted frequently. Further study is necessary to demonstrate which space factors impart serious damage to mechanical properties, how much the degradation of mechanical properties proceeds, and whether the applied tension affects degradation or not.

The tension-applied polyimide films were exposed to a space environment with the Microparticles Capturer and Space Environment Exposure Device (MPAC&SEED) on the ISS Russian service module (SM): polyimide films were included in samples of the SEED experiment [6]. The SM/MPAC&SEED was launched on 21 August 2001, and carried to the ISS by the Russian Progress spacecraft. Subsequently, the SM/MPAC&SEED was set on the exterior wall of the Russian Zvezda SM by extravehicular activity (EVA) on 10 October 2001 (Fig. 1). The SM/MPAC&SEED comprises three units, including identical sets of samples. Each year, one of the three units was retrieved and returned to the Earth by the Russian Soyuz to evaluate aging phenomena in exposed materials. After space environment exposure, tensile tests for exposed polyimide films were conducted to evaluate the changes of mechanical properties.

Numerous degradation factors exist in the LEO environment, as described in the preceding paragraphs. Materials exposed in the LEO are degraded by effects of the respective environmental factors and their synergetic effects. Therefore, from the evaluation of the flight sample alone, it is difficult to distinguish degradation factors. Ground

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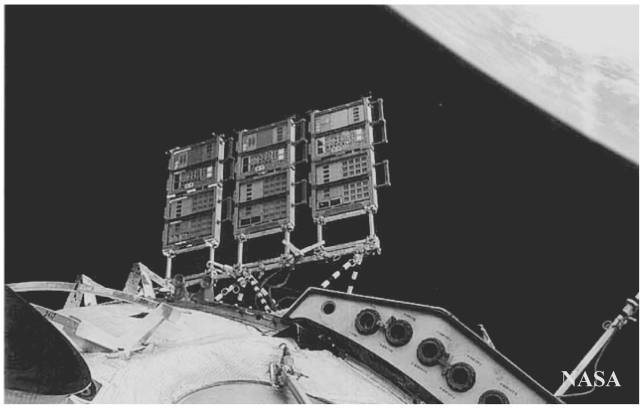


Fig. 1 Photograph of the SM/MPAC&SEED attached to the SM exterior wall.

reference experiments were conducted for comparison with flight experiment results. Major space environmental factors related to degradation of polymers' mechanical properties, that is, AO, UV, and electron beams (EBs), were simulated.

This paper reports evaluation results of tension-applied polyimide films exposed to an actual space environment, in addition to degradation mechanisms of mechanical properties and the applied tension effects, which are discussed through comparison with the degradation behavior of each irradiated sample.

For flight samples, evaluation results from the first and second retrieved samples are described. The third samples are under investigation; those results will be described in future reports.

II. Experimental Procedures

A. Material

Highly heat-resistant polyimide film, UPILEX-S (UBE Industries, Ltd.), was used as a tested material. UPILEX-S has been adopted for applications as a base film for flexible solar paddles of the Space Flyer Unit (Japanese space experimental unit) and the Advanced Earth Observing Satellite I and II (ADEOS-I and ADEOS-II) [4,5].

Dog-bone-shaped samples were used in this experiment to evaluate tensile strength and elongation. The sample shape is similar to that of the "Type IV" specimen of the American Society for Testing and Materials Standard D-638-03. The dog-bone-shaped samples were punched out using a die from a 125-μm-thick sheet. The sample dimensions are presented in Fig. 2.

For polymer sheet production, sheets are drawn to the rolling direction and polymer chains are aligned in the same direction. Therefore, tensile strength and elongation might depend on the drawing direction. The longitudinal direction of all samples was arranged to the drawing direction to prevent the influence of anisotropy.

The sample erosion depth by AO during the experimental duration of approximately 3 years was estimated to be greater than 200 μm. The maximum thickness of the commercial product UPILEX-S was 125 μm. Therefore, each sample consisted of four stacked films in this experiment.

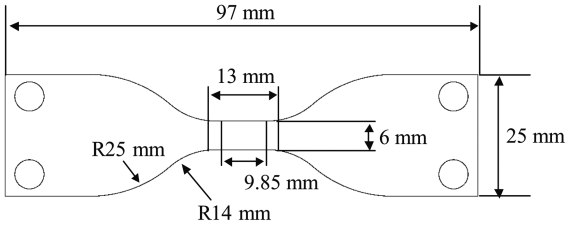


Fig. 2 Schematic view of the sample.

B. Service Module/Microparticles Capturer and Space Environment Exposure Device Experiment

The tension-applied polyimide films were exposed to the LEO environment using SM/MPAC&SEED. After 315, 865, and 1403 days of exposure, one of the three units of SM/MPAC&SEED was retrieved [6]. For this discussion, the first and second retrieved samples are designated, respectively, as "flight 1" and "flight 2."

In the SM/SEED experiment, the four-layer stacked UPILEX-S samples were mounted with a tension-loading mechanism. Figure 3 shows postflight photographs of the mechanism with the sample, and Fig. 4 shows a cross-sectional schematic view of the mechanism. The mechanisms can apply unidirectional tension to samples by pulling one end of a sample, using a spring with exposure of the samples to a space environment. The tension applied to the samples was set to 0 MPa (no tension), 1.4 MPa (low tension), and 7.0 MPa (high tension) by adjusting the spring elongation. Low tension was based on the nominal stress of the base films of the ADEOS-I solar paddles; high tension was set 5 times larger than the low tension. The mounting locations of the samples in the SM/MPAC&SEED are depicted in Fig. 5.

C. Ground Reference Experiments

In ground reference experiments, the samples were irradiated by AO, UV, and EBs. The sample configuration for the ground reference experiments was identical to that for the SM/SEED experiment samples: dog-bone shape and four-layer stack. Three levels of tension, 0 MPa, 1.4 MPa, and 7.0 MPa, were also applied to the samples during irradiation tests.

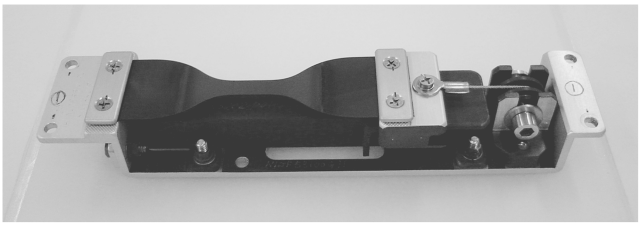


Fig. 3 Postflight photograph of the tension-loading mechanism with a sample.

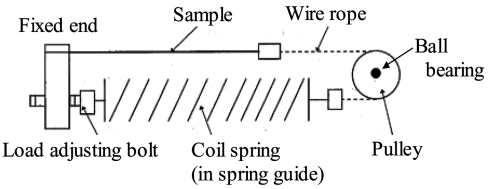


Fig. 4 Cross-sectional view of the tension-loading mechanism.

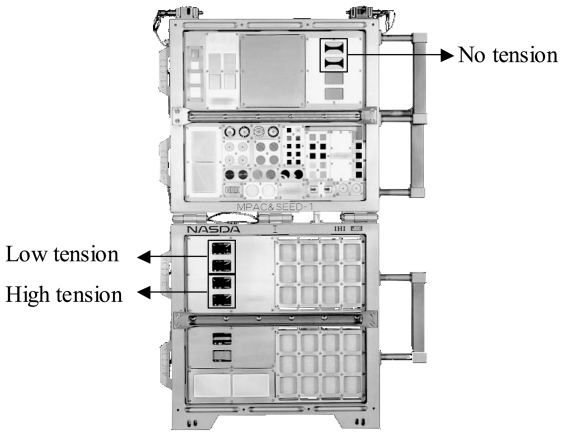


Fig. 5 The SM/MPAC&SEED samples and the location of tension-applied polyimide films.

The AO irradiation testing was performed using a laser detonation AO beam source. The translational energy of the hyperthermal atomic oxygen was approximately 5 eV to replicate the LEO environment. The AO irradiation conditions are listed in Table 1. Kapton H (DuPont) films were mounted with irradiation samples in the same sample holder to monitor the AO fluence. The AO fluence was calculated from the mass loss of Kapton H; it is described as the following Eq. (1):

$$F = \Delta m_K / A_K \rho_K E_K \tag{1}$$

A space chamber equipped with a Xe lamp was used for UV irradiation tests. Table 2 presents the UV irradiation conditions; UV flux and fluence levels were obtained from the spectral radiant intensity at a wavelength of 200–400 nm, as measured using a multispectral radiometer. The Xe lamp light includes an infrared wavelength region. Therefore, the backsides of the samples were cooled by water flow to prevent sample heating. The irradiation area temperature was monitored using thermocouples attached to the reference sample.

Table 3 shows the EB irradiation conditions. Because the total dose of the test is too small to be measured using cellulose triacetate (CTA) films, the total dose for EB irradiation tests was calculated using the irradiation time, as extrapolated from the relation between irradiation times and the total measured dose to CTA films.

D. Surface Observation

Surface microstructure observation was conducted for exposed areas of flight samples and the irradiated areas of ground reference samples using a scanning electron microscope (SEM), JSM-6340F (JEOL, Ltd.), and EPMA-1600 (Shimadzu Corp.).

E. Calculation of Thickness Change

Thickness changes of each sample were calculated using Eq. (2) from the mass loss after space exposure or ground reference experiments:

$$\Delta t_s = \Delta m_s / A_s \rho_s \tag{2}$$

where $\rho_s = 1.47$ g/cm^3 for UPILEX-S.

Table 1 AO irradiation test conditions

AO flux, atoms/cm^2 · s	1.0–5.0 × 10^{15}
AO fluence, atoms/cm^2	3.0 × 10^{20}
	1.3 × 10^{21}
	4.1 × 10^{21}
AO velocity, km/s	8.0
Vacuum, Pa	10^{-2}–10^{-3}

Table 2 UV irradiation test conditions

UV flux, ESD[a]/day	10
UV fluence, ESD[a]	20
	35
	69
Sample surface temperature, °C	10–30
Vacuum, Pa	10^{-4}–10^{-5}

[a]Equivalent solar day, 1 ESD = 1.02 × 10^7 J/m^2.

Table 3 EB irradiation test conditions

EB dose, kGy (irradiation time, s)	1.6 (26)
	3.3 (53)
Accelerating voltage, kV	200
Electron current, mA	2.0
Vacuum, Pa	10^{-4}–10^{-5}

F. Tensile Test Procedures

The tensile strength and elongation of flight samples and ground reference samples were evaluated using universal material testing machines: Autograph AG-5kNI (Shimadzu Corp.) and 5565 (Instron Corp.). Each tensile test was conducted using four stacked films. Grip sections of the samples were mutually bonded using adhesive to prevent interlayer slippage during tensile tests. Before testing for the four-layer stacked samples, it was confirmed that the load was distributed equally to each layer during tensile tests by comparison with tensile test results for one-layer samples; no difference was apparent between the tensile strength and elongation of four-layer stacked samples and those of one-layer samples.

Testing was performed under a constant strain ratio of 50 mm/ min at room temperature and 50 ± 5% relative humidity. The sample strain was calculated based on the crosshead travel distance. The tensile strength was determined as the maximum stress in the stress-strain curve, and elongation was defined as the strain at the first failure of four-layer films. The stress of samples was calculated considering the thickness changes defined by Eq. (2).

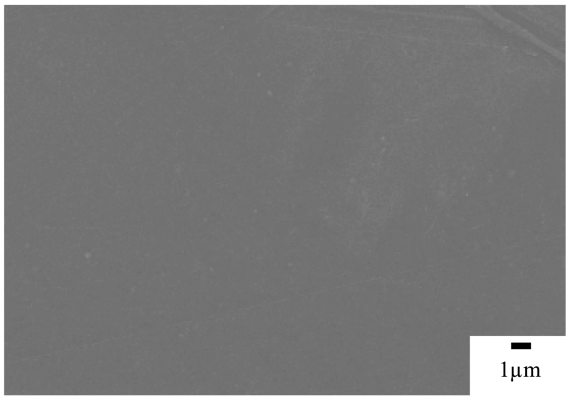
III. Results and Discussion

The surface exposed to the space environment and the surfaces irradiated by AO are shown in Fig. 6. Compared with the control sample's surface, these surfaces were deeply eroded, exhibiting a rough texture. This surface texture is producible by oxidative decomposition and gasification reaction attributable to AO attacks [1,7]. Some contamination is visible on the flight sample's surface. The other samples on SM/SEED were also contaminated by outgassing from organic materials used in the ISS, and the main components of contamination were analyzed to SiO_x [8,9]. The surface asperity of the AO irradiated sample, 1.3×10^{21} atoms/cm^2, was apparently remarkable compared to that of the AO irradiated sample, 3.0×10^{20} atoms/cm^2. This result indicates that surface asperity would be developed as AO fluence increases. The surface microstructure of samples irradiated by UV or EB changed little.

Waters et al. [10] measured the height of cones that developed on a needlelike surface and erosion depth for various polymer films irradiated by AO at an average energy of 70 eV. Results of their study showed that the cone height has a linear relationship with the product of AO fluence and the square root of the erosion yield. The erosion yield is assumed to be constant under fixed translational energy in AO irradiation [11]; the cone height is considered to be equivalent to the surface asperity. Consequently, according to that relationship, the surface asperity can grow along with the increase of AO fluence. Although the AO energy used for their experiment [10] differs greatly from that of our experiment, this relationship between the AO fluence and surface asperity correlates with our current results.

Cross-section observation was also conducted to identify the surface aspect for flight samples and AO irradiated samples, as shown in Fig. 7. The samples for cross section observation were embedded with epoxy and then cut with a microtome. The boundary between the sample and embedding agent is traced in Fig. 7 to clarify the surface aspect eroded by AO. Numerous blunt and short cones are formed on the flight sample surface. The cones on the AO irradiated sample's surface are high, sharp, thin, and aligned at close intervals. The surface roughness of AO irradiated samples, 3.0×10^{20} atoms/cm^2, is much higher than that of the flight 2 samples; the surface roughness R_z is about 2.5 and 0.9 μm for each sample. However, the flight sample's surface exhibits some extremely deep concavities compared with surroundings, as indicated by the white arrow in Fig. 7a.

After space exposure and AO irradiation tests, the mass of the samples was decreased by AO erosion. There was almost no variation in the mass of UV and EB irradiated samples. The thickness changes of flight samples and AO irradiated samples which were calculated from mass loss with Eq. (2) are shown in Fig. 8. The thickness loss of flight samples was smaller than the estimated value, which is greater than 200 μm for three years of space exposure. In addition, there is no remarkable difference of the thickness loss



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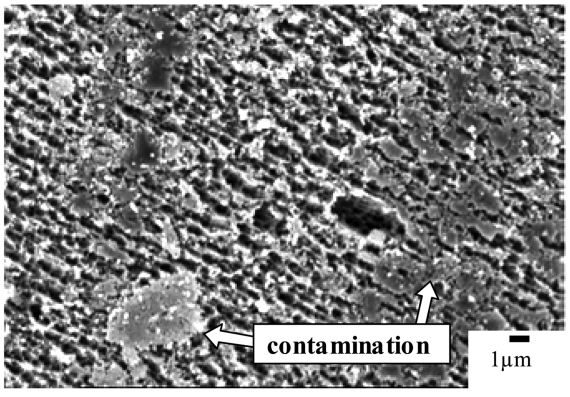

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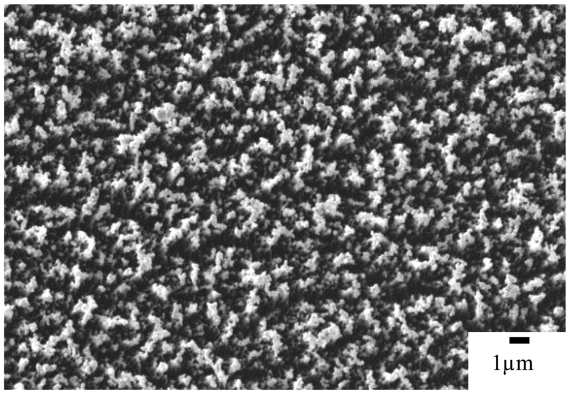

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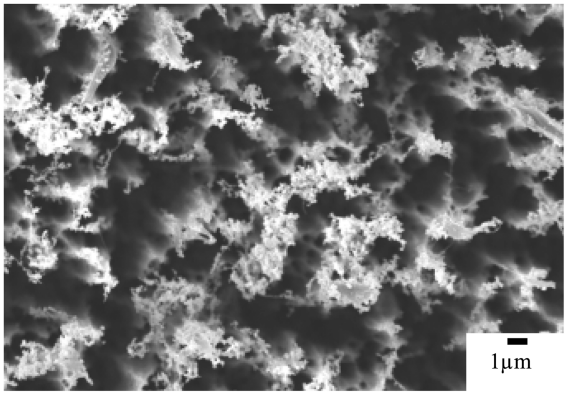
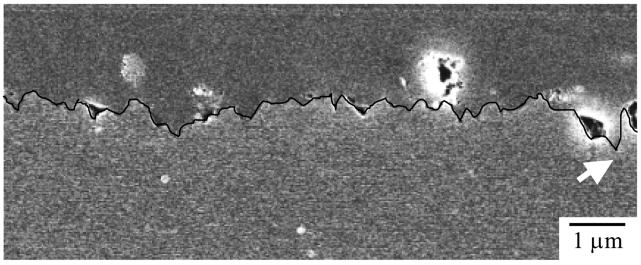


d)

Fig. 6 SEM images of a) the control sample's surface, b) the exposed surface of flight 2 (no tension), c) the AO irradiated sample's surface (no tension) at 3.0×10^{20} atoms/cm^2, and d) the AO irradiated sample's surface (no tension) at 1.3×10^{21} atoms/cm^2.



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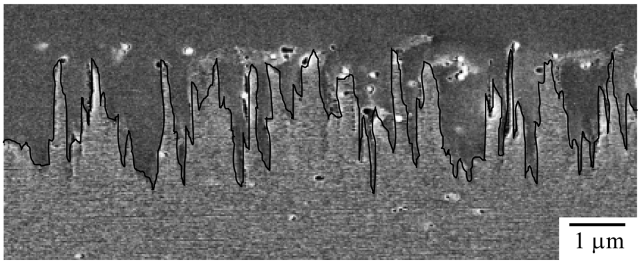


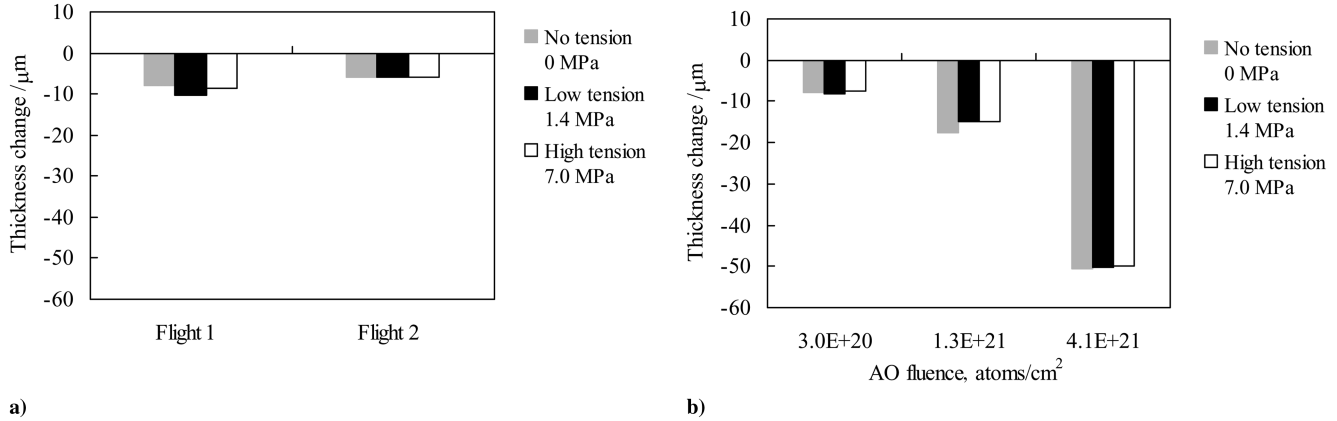
b)

Fig. 7 Cross section of a) flight 2 (no tension), and b) the AO irradiated sample (no tension) at 3.0×10^{20} atoms/cm^2. The boundary between the sample and embedding agent was traced to clarify the surface aspect.

between flights 1 and 2. These results show that the SiO_x contamination layer on the flight sample surface can serve as the anti-AO coating [9]. There is concern that the thickness loss is estimated as smaller than the actual loss in calculation using Eq. (2) because of the mass increase by contamination attachment. Through comparison of the thickness calculated with Eq. (2) and that measured directly from the cross section observation, the influence of contamination attachment to the thickness loss is negligible; the calculated thickness is approximately equivalent to the actual measurement value. The changing of contamination thickness was evaluated for other samples of SM/SEED; the contamination thickness was 80 nm after 865 days of space exposure [8]. The thickness of AO irradiated samples decreases concomitantly with the increase of AO fluence. No considerable change attributable to applied tension was found in either flight or AO irradiated samples.

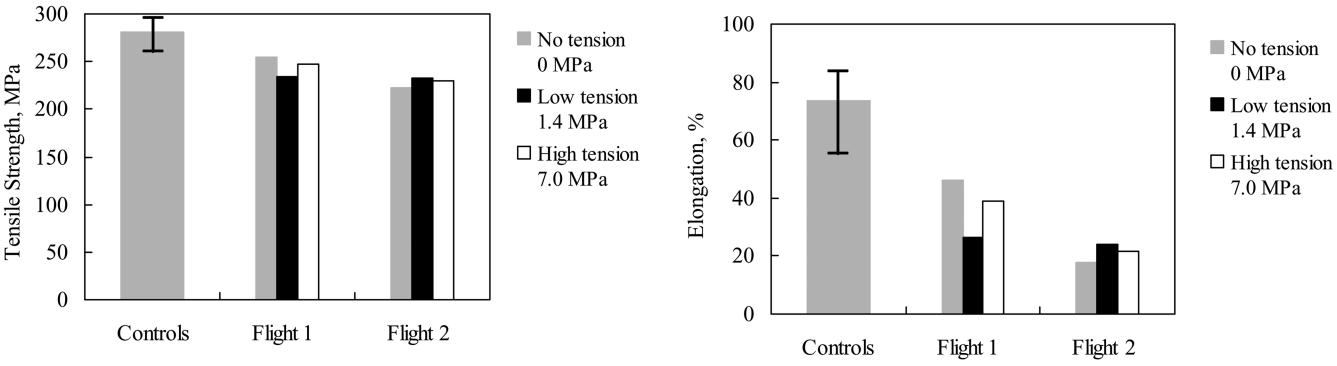
Tensile strength and elongation changes of the flight samples are shown in Fig. 9. Compared with the control samples, the tensile strength of the flight samples decreased slightly. A major reduction in elongation was detected, showing a decrease to 25–60% of the value of the control sample. In terms of both tensile strength and elongation, the reduction of flight 2 was greater than that of flight 1; the longer the exposure duration, the more mechanical properties of exposed materials were degraded.

The changes of mechanical properties for the AO, UV, and EB irradiated samples are shown, respectively, in Figs. 10–12. Figure 10 shows that the mechanical properties of AO irradiated samples declined considerably with increasing AO fluence. The tensile strength and elongation decreased, respectively, to 60 and 20% of control samples' values at AO fluence of 4.1×10^{21} atom/cm^2. As shown in Figs. 11 and 12, minor changes of tensile strength and elongation occurred in UV and EB irradiated samples. Although the chemical structure of polyimides can be transformed by UV irradiation [12], these results suggest that the chemical reaction layer by UV irradiation is too thin to affect the mechanical behavior. Generally, polyimide has a high concentration of imide rings in the main chain, rendering it resistant to EB irradiation because of pi-electron conjugation. The EB dose level, by which the elongation of UPILEX-S declines to 50% of its initial value, is approximately 30 MGy [13]. The dose levels in present EB irradiation tests, 1.6 and 3.3 kGy, are small compared to 30 MGy. Therefore, noticeable degradation of mechanical properties by EB irradiation was not seen at these EB dose levels. In the ground reference experiments, marked degradation of mechanical properties was found only in AO



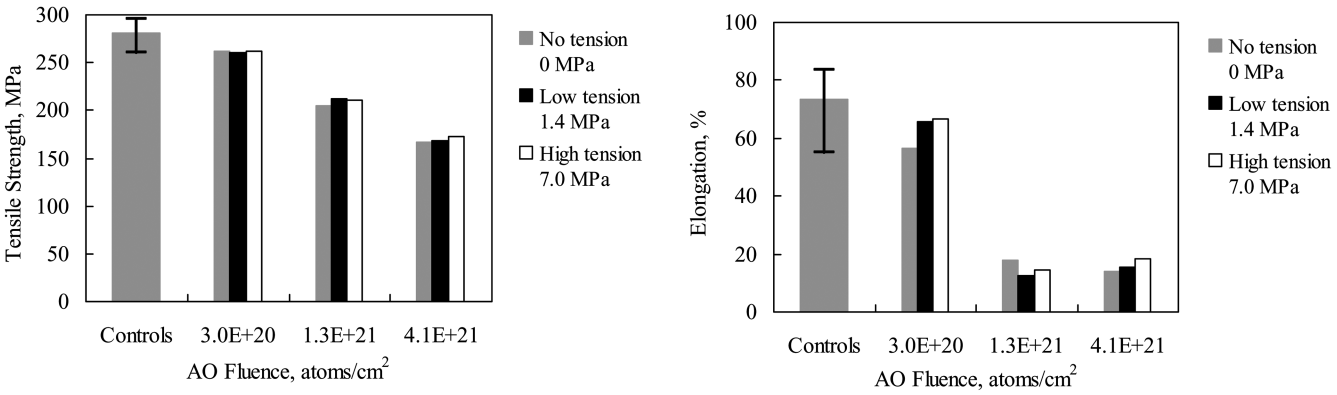
a) b)

Fig. 8 Thickness change of a) the flight samples and b) the AO irradiated samples.



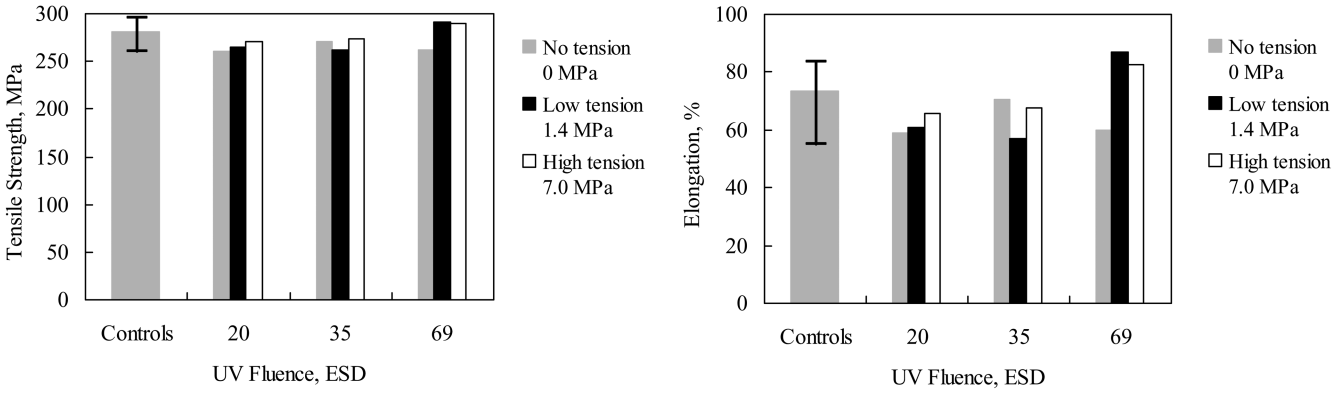
a) b)

Fig. 9 Flight samples' a) tensile strength and b) elongation.



a) b)

Fig. 10 AO irradiated samples' a) tensile strength and b) elongation.



a) b)

Fig. 11 UV irradiated samples' a) tensile strength and b) elongation.

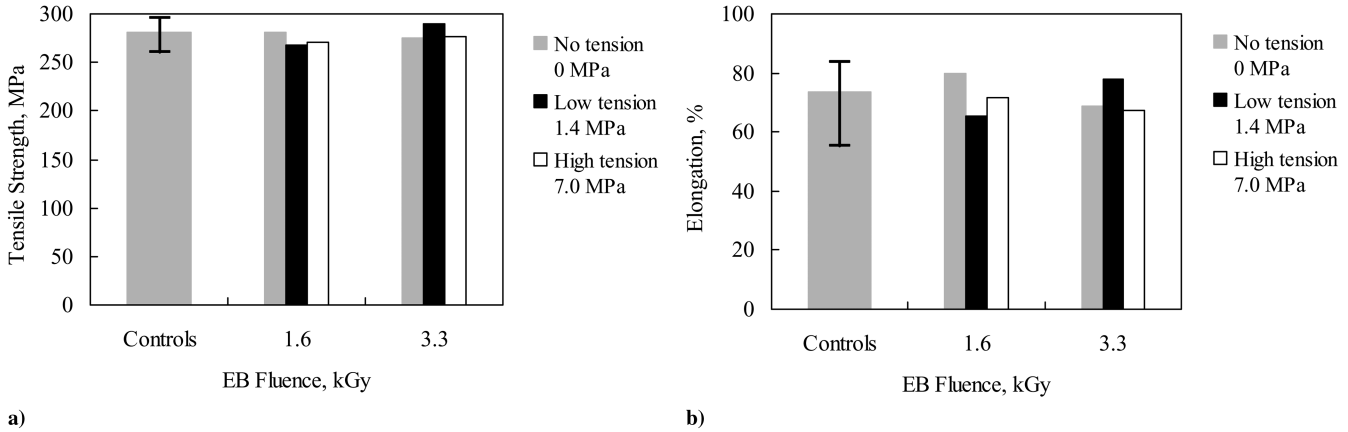


Fig. 12 EB irradiated samples' a) tensile strength and (b) elongation.

irradiated samples. Consequently, AO attack is considered to be the main degrading factor in a space environment.

Applied tension had no marked effect on tensile strength and elongation in either the flight or the ground reference samples. The tension level applied to samples is 3% of the yield stress of control samples. It is conceivable that the applied tensions are too small to affect the samples' tensile strength and elongation. Further experiments are needed with higher tension to clarify tension effects of degradation during space environment exposure.

The changes in tensile strength and elongation of flight samples and AO irradiated samples, which were set to 7.0 MPa (high tension) during space exposure or irradiation tests, are plotted for AO fluence in Fig. 13. As described previously, no marked difference was found among applied tension levels. The AO fluence of flight samples was determined by evaluation of the AO monitoring sample, Vespel (DuPont), and the simulation using the Space Environments and Effects System (SEES; the database system for providing data and models related to space environments and effects of space environments) [14]. The AO monitoring sample was mounted in the SM/MPAC&SEED with other samples: the respective AO fluences for flights 1 and 2 were 2.04×10^{20} atoms/cm^2 and 2.57×10^{20} atoms/cm^2. The SEES simulation deduced the AO fluence levels for each flight sample as 2.85×10^{21} atoms/cm^2 for flight 1 and 5.70×10^{21} atoms/cm^2 for flight 2, taking into account the exposure period of SM/SEED, and the orbit and altitude of the ISS during the SM/SEED experiment. An enormous discrepancy exists between the AO fluence calculated by the AO monitoring samples and those deduced from the SEES simulation. The discrepancy would stem from the SiO_x contamination layer observed on the flight sample surface; the AO monitoring samples were prevented from AO erosion because of the SiO_x contamination layer, resulting in the low AO fluence [14]. Therefore, the AO fluence of flight samples includes large error.

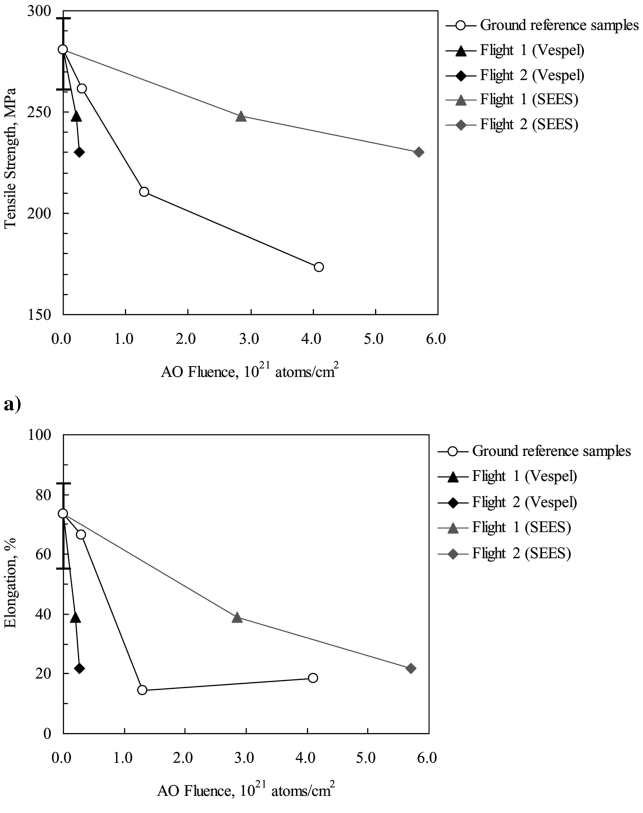


Fig. 13 Changes in a) tensile strength and b) elongation of flight samples (high tension) and AO irradiated samples (high tension) by AO fluence.

In Fig. 13, the tensile strength of the ground reference samples was shown to decrease gradually to 60% of the pristine one as AO fluence increases. Elongation of the ground reference samples showed a critical reduction at the AO fluence of less than 1.3×10^{21} atoms/cm^2; it indicated considerable degradation as a polymer material. The tensile strength and elongation of flight samples were also decreased as the AO fluence increased, denoting the same tendency of the ground reference samples. However, it is difficult to read for the consistency of degradation degree between the ground reference samples and the flight samples from Fig. 13 because of the large error of the flight samples' AO fluence.

As a result of SEM observation, the amount of surface asperity increases concomitant with the AO fluence. In addition, the degradation of tensile strength and elongation of samples was enhanced by increasing AO fluence. From these results, surface morphology transformation to greater roughness might exert a strong influence on tensile strength and elongation; the increased surface asperity is inferred to cause a greater opportunity for destruction. It is generally assumed that excessive stress concentrates at a concave region on the rough surface; then the concave region can develop into surface cracks and become the initiation point of the polymer film's destruction. The flight samples showed the significant decrease of more than AO irradiated samples, 3.0×10^{20} atoms/cm^2, in tensile strength and elongation, in spite of the fact that the surface roughness R_z of flight samples was less than that of AO irradiated samples. A stress concentration would be produced easily at extremely deep concavities compared with surroundings observed on the flight sample's surface.

IV. Conclusions

Tensile tests were used to investigate degradation of mechanical properties for tension-applied polyimide films exposed to a space environment. The tensile strength and elongation of the flight samples decreased as their exposure period increased. Ground

reference tests demonstrated that the AO irradiated samples underwent considerable degradation of tensile strength and elongation. This result clearly suggests that AO attack is the main cause of decreased tensile strength and elongation in a space environment. The space exposed surface of flight samples and the surface irradiated by AO in ground reference examination developed a rough texture. The rough surface is regarded as a cause of degradation of mechanical properties. Tension of less than 7.0 MPa, which was applied to the samples during space exposure and irradiation tests, had no marked effect on the degradation of mechanical properties of any sample.

Acknowledgments

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