EFFECTS OF LEO ENVIRONMENT ON MECHANICAL PROPERTIES OF PEEK FILMS UNDER TENSILE STRESS

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The environment of LEO is extremely harsh for polymer materials because of detrimental factors such as atomic oxygen (AO), ultraviolet light (UV), and charged particle radiation. Although many researches regarding LEO environment on physical and chemical properties of polymers were reported, those on mechanical behaviors are still insufficient. For the long term use of polymers in LEO, it is necessary to elucidate the strength properties systematically. This study exposed Poly-ether-ether-ketone (PEEK) films under tensile stresses into LEO by using ISS Russian service module. In parallel, ground tests were carried out by using JAXA facilities, which irradiated AO, UV, and electron beam (EB) to the specimens individually. By comparing the results of flight tests with ground tests, degradation properties and the mechanisms were studied.

Keywords: Atomic Oxygen, Ultraviolet Light, Electron Beam, PEEK, Tensile Properties, International Space Station

1. Introduction

In recent years, deployable structures have been acknowledged to be a leading edge technology for the construction of space facilities. Their use is expected in many space structures, such as large space antennas, photovoltaic generation systems, solar sails, etc. Polymeric films are one of the candidate materials for these deployable structures. In these applications, polymer films have to hold a certain amount of load in space for a long term. Therefore, the reliability of mechanical properties in space is one of the most important factors for them. However, space is extremely harsh to polymers due to the presence of several types of radiation and atomic oxygen (AO). In particular, it is well known that many polymeric materials are damaged by AO in LEO from 200 to 700 km altitude [1]. Although space exposure experiments of polymer films have been conducted [2]-[3], we have insufficient data of the relation between mechanical properties and LEO environment.

To investigate these phenomena, two different experiments were organized: one was a space exposure experiment and the other was a ground control experiment [4]. The space exposure experiment exposed Poly-ether-ether-ketone (PEEK) films with tensile loads in LEO environment. This research is a part of the Micro-Particles Capturer and Space Environment Exposure Device (MPAC&SEED) experiment implemented by the Japan Aerospace Exploration Agency (JAXA) [5]. The space experiment started in October 2001 utilizing the ISS Russian Service Module, and finished with the final retrieval of the samples in August 2005. The ground control experiments also started in 2001 by using JAXA facilities, which exposed individual irradiation of AO, ultraviolet light (UV) and electron beam (EB) to the reference PEEK films.

The exposed samples of both experiments were analyzed focusing on the following material properties at Hokkaido University:

(1) Physical properties: Change in mass, thickness, surface morphology.
(2) Chemical properties: Change in chemical structure.
(3) Mechanical properties: Change in tensile properties.

This study mainly introduced the change in mechanical properties of PEEK films after space exposure, and investigated the influential factors on the degradation by comparing the flight and ground experiments.

2. Experimental procedures

2.1 Flight tests

The material used was PEEK film with 0.4mm thickness (FS-1100C, Sumitomo Bakelite Co., Ltd.). The repeating unit of PEEK is shown in Fig.1. PEEK is not a commonly used spacecraft material compared with polyimide; however, it has many attractive properties due to thermoplastic characteristics. In particular, it is expected for heat resistant films and/or matrix of composite materials because of its high formability. Besides, by comparing the degradation of PEEK with those of polyimide (thermosetting polymer), useful insights can be derived on the relation between polymer structures and changes of mechanical properties.

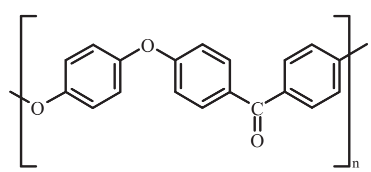
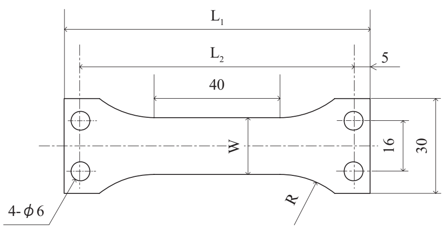


Fig.1 Repeating unit of PEEK.

Three types of specimens with different widths were machined as shown in Fig.2. The axial direction of the specimens was set to the extruded direction of the film. The specimens were loaded by a tension spring with initial setting stresses of 0, 1.6, and 4.7MPa during the experiments. These



Thickness : 0.4 unit in mm

W (mm)	R (mm)	L_1 (mm)	L_2 (mm)	Initial stress (MPa)
30	-	100	90	0
18	28	97	87	1.6
6	14	97	87	4.7

Fig.2 Specimens of flight tests.

values corresponded to about 0, 2 and 5% of yield strength (≈ 85MPa).

In the space exposure experiment, the three types of specimens were installed on a same sample holder, and three sets of the sample holders were attached on the exterior of the Russian service module. Each sample holder was retrieved to the Earth after 10-months (315 days), 28-months (865 days), and 46-months (1403 days) exposures. The average values of the altitude and the velocity of the ISS were 371km and 8.1km/s, respectively.

2.2 Ground tests

In the ground tests, individual irradiation of AO, UV, and EB were carried out [4] by using the facilities of JAXA. The Combined Space Effects Test Facility [6] was used for AO and EB irradiation, and the UV test system (Yamashita Denso Corporation) was used for UV irradiation. The condition of each irradiation is shown in Table 1. The irradiation level (Table 2) was set to be equivalent with 0.1, 0.5, 1 and 3 years in LEO environment based on the calculation by the Space Environments & Effects System (SEES) [7] of JAXA.

Table 1 Conditions of the AO, UV, and EB irradiations

AO	AO velocity	(km/s)	8.11
	AO flux	(atoms/cm^2·s)	1.95~3.50×10^{15}
	Vacuum pressure in the test chamber	(Pa)	≈ 10^{-3}
EB	Accelerating voltage	(kV)	200
	Vacuum pressure in the test chamber	(Pa)	< 10^{-8}
UV	Light intensity	(ESD/day)	10
	Wave length	(nm)	250-500
	Vacuum pressure in the test chamber	(Pa)	< 10^{-8}

ESD: Equivalent Solar Day = 1.02×10^3 J/cm^2

Table 2 Irradiance level of each irradiation

	Irradiance level				Unit
AO	2.79×10^{20}	1.31×10^{21}	(a)4.08×10^{21} (b)3.97×10^{21}	(a)7.55×10^{21} (b)7.75×10^{21}	atoms/cm^2
EB	–	1.64×10^{12} (1.63)	3.30×10^{12} (3.29)	9.89×10^{12} (9.84)	Electrons/cm^2 (kGy)
UV	–	34 (3.47×10^4)	69 (7.04×10^4)	–	ESD (J/cm^2)
Equivalent time in the LEO	0.1	0.5	1	3	year

Notes. (a):Stress=0MPa
(b):Stress=1.6MPa, 4.7MPa

2.3 Analyses methods

After the space and ground control experiments, specimens were analyzed by using mass measurements, AFM analyses, DSC analyses, EPMA analyses, and XPS analyses to investigate physical and chemical properties. The following equipments were used for the analyses.

Mass measurement: Mass of the specimens was measured by the electron balance (ME215P, Sartorius) at a temperature of 23±2°C and a humidity of 50 ±2°C after conditioned in the same environment for 48 hours. The repeatable precision is within ±0.015mg.

AFM analysis: Surface morphology was measured by using the atomic force microscope (SPA-400, SPI3800N, SII Nano Technology Inc.).

DSC analysis: Heat properties of the specimens were measured by the differential scanning calorimeter (DSC-6200, Seiko Instruments Inc.) to clarify the glass transition temperature, melting temperature, etc. Measurements were done in N_2 environment with a heating and cooling rate of 10°C/min.

XPS analysis: X-ray photoelectron spectroscopy was carried out to measure the chemical structure at specimen surface (ESCALAB 220i-XL, FI Surface Systems Inc.).

In addition to the physical and chemical analyses, mechanical properties were investigated by tensile tests. Specimens for tensile tests were cut into the width of 1mm from the samples by using a sharp razor blade. After conditioned at a temperature of 23 ± 2 ºC and a relative humidity of 50 ± 5 %, tensile tests were conducted in the same environment with a strain rate of 0.1 /min, conforming to ASTM D882-95a.

3. Results of flight tests

Various changes were seen in the specimens after space exposure compared with pristine ones. In this section, main features of the flight specimens will be shown focusing on the surface appearance, thickness, and tensile properties.

3.1. Surface appearance

Fig. 3 shows the surfaces of the flight samples after space exposure. The color of the exposed areas clearly changed into brown. The browning became deeper with increasing exposure period; however, the amount of browning had no relation with tensile stress. In addition to the browning of the sample, specimen surfaces were slightly covered with the pale whitish matter. The detail of this feature is shown in Fig. 4. The pale whitish region was observed in about 70~80% of the exposed area regardless of applied stress and exposure period (the arrow A in Fig.4). The EPMA analyses showed that Si and O element were mainly detected; therefore, this region can result from the chemical reaction between Si-derived contamination and atomic oxygen.

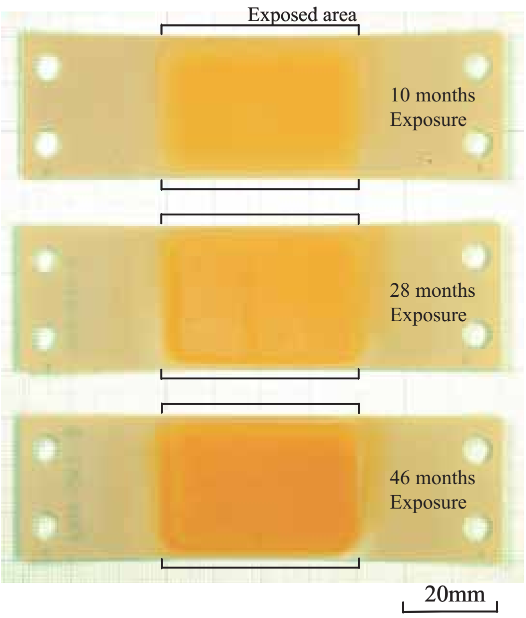


Fig.3 Surface appearances after space exposure under no tension.

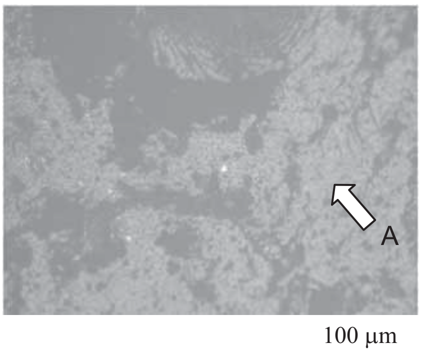


Fig.4 Magnified photograph of the 28-months flight sample under 1.5MPa. The arrow A shows the pale whitish region.

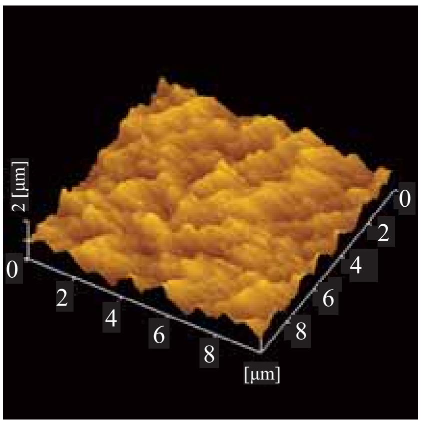


Fig.5 Surface morphology of the flight sample after 10-months exposure.

Fig. 5 shows the AFM surface topography of the sample exposed for 10 months. The exposed area was distinctively eroded showing a cone-like pattern. This eroded feature is generally observed on the polymer surfaces attacked by AO. The maximum difference in the height of the conical pits was 2.5 μm in the area of 20 μm × 20 μm. The amount of the roughness seemed to have no significant relation with tensile stress during exposure.

3.2 Thickness reduction

The mass of the specimens decreased with increasing exposure period, which resulted from thickness reduction caused by AO attack. The average thickness decrease, Δh, was calculated by using next equation,

$$\Delta h = \frac{\Delta m}{\rho \cdot A} \qquad \cdots (1)$$

where Δm: mass loss, ρ: density of PEEK=1.3g/cm^3, A: exposed area. Fig.6 shows the relation between Δh and exposure period. Δh increased with increasing exposure period; however, it tended to deflect from a straight line. Stresses during exposure seemed to have no significant effect on Δh.

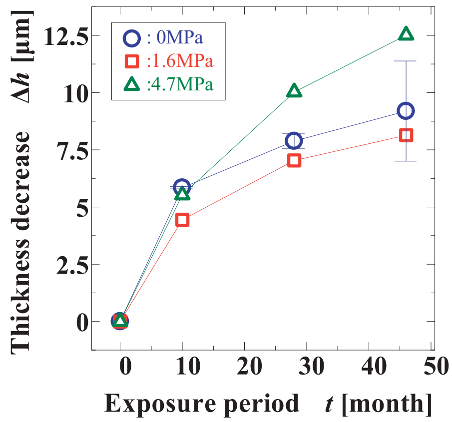


Fig.6 Thickness decrease of the flight samples.

3.3 Tensile properties

Fig. 7 shows the stress strain curves. For making stress-strain curves, stress was calculated by using the reduced thickness (h-Δh) after exposure by subtracting thickness

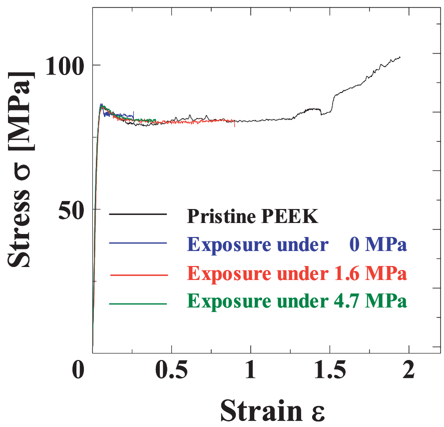


Fig.7 Stress-strain curves of the flight samples after 28-months exposure.

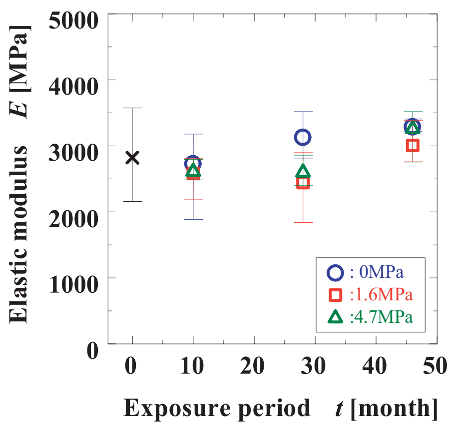


Fig.8 Relation between elastic modulus and exposure period of the flight samples.

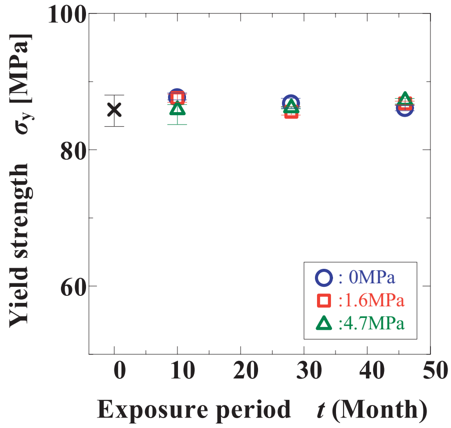


Fig.9 Relation between yield strength and exposure period of the flight samples.

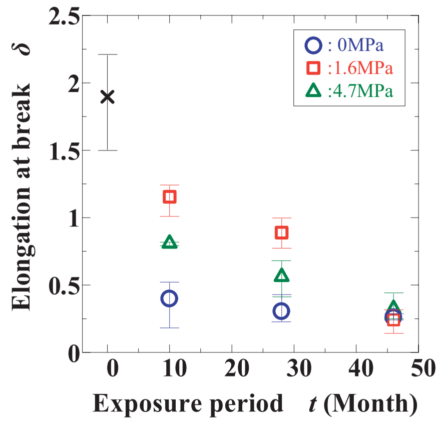


Fig.10 Relation between elongation at break and exposure period of the flight samples.

decrease Δh from initial thickness h. The stress strain curve of the pristine sample exhibited a clear yield point followed by a necking deformation. After the termination of the necking behavior, stress rose again and the specimen finally fractured. The strain at fracture point, elongation, was about 200 % showing large ductility of PEEK. The space exposed samples also exhibited yield point and necking deformation; however, elongation decreased compared with pristine sample.

The relation between elastic modulus and exposure period is shown in Fig.8. Although each elastic modulus had a wide distribution, the range of the flight samples was within that of the pristine ones. The space environment around the ISS had no clear effect on elastic behavior of PEEK. Fig.9 shows yield strength. Yield strengths of flight samples were almost same as the pristine one regardless of exposure period and tensile stress. Fig. 10 shows the relation between elongation and exposure period. Unlike with elastic modulus and yield strength, elongation was strongly affected by LEO environment. It decreased with increasing exposure time, and after 46 months, it dropped to about 15% of the pristine sample. The space environment clearly embrittled the specimens. As for 10- and 28-months exposures, elongations of the stressed samples were larger than those that were not stressed. However, elongations after 46-months exposure were similar regardless of stress. This suggests that a correlation between LEO environment and stress on mechanical properties are likely to exist in the early stage of exposures. Based on the results of the tensile tests, it can be said that elongation at break is the useful parameter to estimate the degradation of mechanical properties.

4. Results of ground tests

Significant changes were observed in the flight samples as mentioned in the previous section. The following introduces the main results of AO, UV, and EB irradiation in the ground control experiments to clarify which factors in LEO environment caused the phenomena.

4.1 Surface appearance

Fig.11 shows an example of the surface feature of AO irradiated sample in the ground tests. The cone-like morphology similar to the flight sample (Fig.5) was also observed. In contrast, there was no significant change in surface topography after UV and EB irradiations. Therefore, as anticipated, the surface erosion showing a cone-like pattern was caused by AO attack in LEO environment.

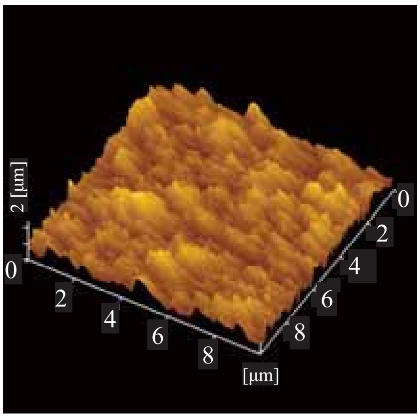


Fig.11 Surface morphology of the AO irradiated sample (2.79×10^{20} atoms/cm^2) .

After the AO irradiation tests, maximum difference in height in the surface area (100 μm × 100 μm) were 4μm, 8μm, and over 10μm with respect to the fluence of 2.79×10^{20}, 1.31×10^{21}, and 4.08×10^{21} atoms/cm^2. The surface roughness increased with increasing AO fluence. On the other hand, the maximum difference in height of the surface of the flight sample after 10-months exposure was only 2.5 μm (Section **3.1**). This value is similar to the roughness of the sample (4μm) irradiated with AO at 2.79×10^{20} atoms/cm^2. According to the calculation by the SEES program [7], 2.79×10^{20} atoms/cm^2 was equivalent to the fluence of about 0.1 year in LEO (Table 2). Therefore, the actual fluence which the flight sample suffered from during 10 months in LEO can correspond to only 0.1 year. This discrepancy will be discussed in Section **5**.

The surface color of the AO irradiated samples at 2.79×10^{20}atoms/cm^2 remained unchanged. The EB irradiated ones also showed no color change. In contrast, the UV irradiation changed the surface color into dark brown. Fig.12 shows the surface appearance after UV irradiation. The browning of the sample was clearly seen. Therefore, UV constituent in LEO can be the main reason for the surface browning of the flight samples.

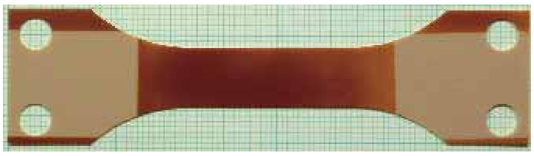


Fig.12 Surface appearance of the UV irradiated sample (34ESD (3.47×10^4 J/cm^2), under 4.7MPa) .

4.2 Thickness reduction

Mass change occurred only in the AO irradiated sample in the ground tests. The average thickness reduction *Δh* after AO irradiation was calculated by using Eq. (1). Fig. 13 shows the relation between *Δh* and AO fluence. *Δh* was directly proportional to the AO fluence. The tendencies in Fig.13 are very different from Fig.6, and the possible reasons for the discrepancy will be discussed in Section **5**. Both UV and EB irradiation caused no significant change in mass and in thickness; therefore, the thickness decrease of the flight samples resulted from AO constituent in LEO.

The stress during irradiation had no significant effects on thickness decrease. The tendency is different from the report by Verker et al., which showed that stress during AO irradiation increased the erosion damage [8]. The reason of the discrepancy is still under examination; however, the very low stresses used in this study (2, 5 % of yield stress,) compared with their experiment may relate with the behavior.

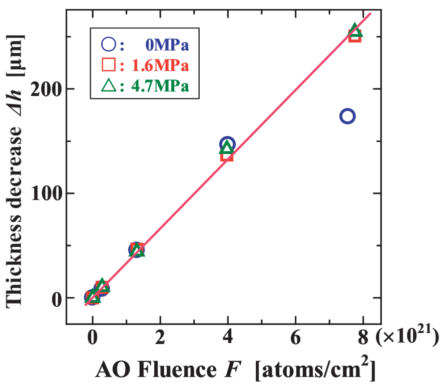


Fig.13 Relation between thickness decrease of the samples and AO fluence in the ground tests.

4.3 Tensile properties

As shown in Fig.7 and Fig.10, the elongation of the flight samples decreased after space exposure. Since the surface of the flight sample showed cone-like pattern, the concavities may relate with these phenomena due to stress concentration effects. To check this problem, stress-strain diagram of the AO irradiated samples were measured. Fig.14 shows the stress-strain curves of the samples irradiated with AO at 2.79×10^{20} atoms/cm^2. As mentioned in Section **4.1**, these samples had a similar roughness with 10-months flight samples, and are suitable for the comparison from the view point of stress concentration. From Fig. 14, stress-strain curves after AO irradiation are almost same with pristine sample regardless of tensile stress. The results suggested that the elongation at break was not affected by AO irradiation with this fluence level.

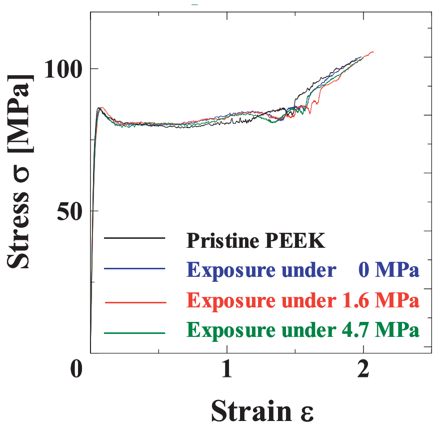


Fig.14 Stress-strain curves of the AO irradiated samples with a fluence of 2.79×10^{20} atoms/cm^2.

In addition to AO irradiation, EB irradiation also had no effect on stress-strain curves of PEEK. Some researchers reported that EB irradiation decreased the elongation of PEEK; however, it was obtained under the high dose level over 100kGy ~100MGy [9]-[10]. The EB dose used in this research (10kGy, Table 2) was much less than these values, therefore, it

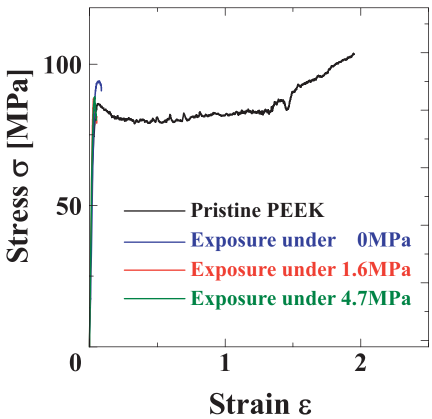


Fig.15 Stress-strain curves of the UV irradiated samples with a fluence of 34ESD (3.47×10^4 J/cm^2).

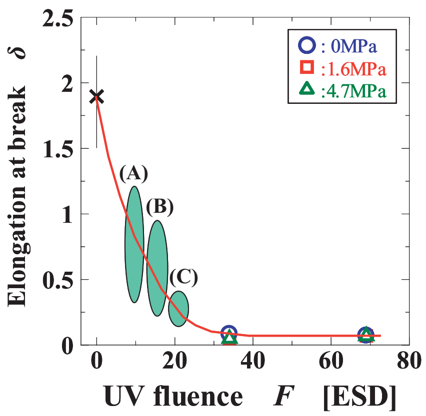


Fig.16 Relation between elongation at break and UV fluence. (A), (B) and (C) corresponds to the range of elongation of flight samples after 10, 28, and 46-months exposure, respectively.

was too low to affect the tensile properties of PEEK.

Different from the results of AO and EB irradiation, UV had significant effects on tensile properties. Fig.15 shows the stress-strain curves after UV irradiation with a fluence of 34ESD (3.47×10^4J/cm^2). The elongation at break decreased dramatically. Fig.16 shows the relation between elongation at break and UV fluence. The UV irradiation decreased the elongation to 3~5% of that of pristine sample. The reduction of the elongation seems to be already saturated at the fluence of 34ESD. Although the decrease of elongation was larger in ground UV tests (Fig.16) than in flight tests (Fig.10), the main factor which degrades mechanical properties of PEEK can be attributable to the UV constituent in LEO.

5. Discussion

5.1 The mechanism of degradation of PEEK induced by UV constituent in LEO environment

Based on the comparison between flight tests and ground tests of PEEK films, the harshest factor in LEO affecting the tensile properties was considered UV. It caused the embrittlement of the material as shown in Fig.10 and Fig.16. To investigate the reason, the XPS analysis of the UV irradiated sample in the ground tests [11] was shown in Fig.17. A new chemical structure of O=C–O was generated as well as the reduction of the C–O and C=O bonding after UV irradiation. This result suggested that both scissions and crosslinking of polymer chains occurred. The results of DSC analyses were given in Table 3. Glass transition temperature (T_g) increased while heat of crystallization ΔH_c, melting temperature T_m, and heat of melting ΔH_m decreased after UV irradiation. These data corresponds to the result of XPS analyses (Fig.17). In particular, both the increase of T_g and the decrease of ΔH_c indicated the formation of crosslinking [12]-[13]. Crosslinking generally results in the embrittlement of polymer materials [12]; therefore, the changes in mechanical properties of the flight samples were mainly caused by this mechanism.

As for the 10-months and 28-months exposures in space, elongations of the stressed samples were larger than those that were not stressed. The detail mechanism of the phenomenon is still unclear; however, the similar tendencies were reported in the mechanical properties of polypropylene exposed with UV [14]. In the literature, tensile stress during UV exposure tended to reduce the decrease of elongation, especially in the early stage of UV irradiation. This suggests that tensile stress has the possibility to prevent the crosslinking during UV irradiation. However, when the fluence of UV increases, the ability to cause crosslinking can be much larger. As a result, the effect of tensile stress to prevent the decrease of elongation might diminish as shown in the 46-month exposed samples.

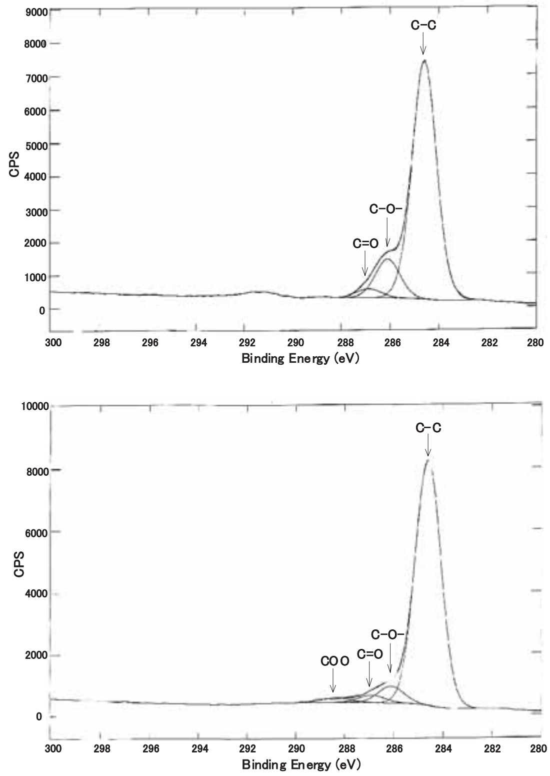


Fig.17 XPS analyses before and after UV irradiation.

Table 3 Heat properties before and after UV irradiation measured by DSC (34ESD, under no tension).

	T_g (°C)	ΔH_c (J/g)	T_m (°C)	ΔH_m (J/g)
Pristine	142.3	14.5	340.5	45.3
After UV irradiation	153.1	6.08	333.7	33.8

5.2 The discrepancies between the degradation of flight samples and those of reference samples in the ground tests

Comparison between flight and ground tests clarified that AO in LEO environment caused surface erosion and thickness decrease, and that UV induced the degradation of mechanical properties. However, there are several discrepancies between flight and ground samples quantitatively. As for thickness decrease *Δh*, the tendencies were different between the flight (Fig.6) and the ground tests (Fig.13). In the flight tests, *Δh* tended to deflect from a straight line with increasing exposure period; however, *Δh* was proportional to AO fluence in the ground tests. In addition, *Δh* in flight samples without tension were only 5.9μm, 7.9μm, and 9.2μm with respect to 10, 28, and

46-months exposures. In contrast, *Δh* of AO irradiation with a fluence of 7.6~7.8×10^{21} atoms/cm^2, which corresponded to 3 years in ISS orbit (Table 2), was about 250μm. To investigate the discrepancies, the actual AO fluence of the flight samples were estimated by using both *Δh* of flight tests (Fig.6) and the results of AO irradiation tests (Fig.13). Then, 1.8×10^{20}, 2.4×10^{20}, and 2.8×10^{20} atoms/cm^2 were obtained with 10, 28, 46-months flight, respectively. These values are much smaller than the AO fluence calculated by SEES program [7] of JAXA (Table 2). In addition, the roughness of the flight samples (Fig.5) was similar to that of AO irradiated sample with a fluence of 2.79×10^{20} atoms/cm^2 (Fig.11). From these results, the actual fluence of the flight samples can be about 2~3×10^{20} atoms/cm^2. These values correspond to the AO fluence of about 1month in LEO environment.

As for tensile properties, the elongations at break after UV irradiation over 34ESD (Fig.16) were smaller than those of flight samples (Fig.10). To compare the difference, the range of elongation of flight samples were replotted as (A)~(C) on the curve in Fig. 16. As a result, the actual UV fluences of flight samples were estimated to be less than 20ESD. These values correspond to only about 2~3.5 months in LEO environment (Table 2).

Based on the above discussion, it can be said that the actual fluence of AO and UV which flight samples suffered from in LEO was much smaller than the value estimated by SEES program. The following two were possible reasons for the discrepancy. One was the attitude change of the ISS during the flight and the other was the attachment of SiO_x contamination on the sample. The ISS had been under construction during the space experiment; therefore, the actual time, for which the normal of the sample surface coincided with the ISS traveling direction, was not always proportional to the exposure period. Besides, EPMA analyses confirmed that sample surfaces were covered by SiO_x layer. This contamination has the ability to prevent the AO attack. In addition, SiO_x has very low light transmission [15]. Therefore, the contamination composed of SiO_x has the possibility to reduce the effect of AO and UV on the flight samples.

6. Conclusion

To clarify the effects of LEO environment on mechanical properties of polymers, exposure experiments were conducted utilizing the International Space Station Russian Service Module. Poly-ether-ether-ketone (PEEK) films under tensile stresses were exposed to LEO, and reference samples were irradiated with AO, EB, and UV by using ground facilities of JAXA. Comparing the results of flight and ground tests, degradation behaviors of the samples and those influential factors were investigated. The main results obtained in this study were summarized as follows:

(1) The surface color of the flight samples changed into dark brown, and the browning became deeper with increasing exposure period. The similar phenomenon was not seen in AO and EB irradiations but in UV irradiation of ground tests. UV constituent in LEO environment can be the main reason of the browning of the sample surface.

(2) The thickness of the flight samples decreased with showing a surface morphology of cone-like pattern. These phenomena were only observed in AO irradiation of ground tests; therefore, it can result from AO in LEO environment.

(3) Elastic modulus and yield strength of the flight samples remained unchanged; however, the elongation at break decreased to about 15% of pristine sample after 46-months exposure. The UV irradiations in the ground tests only showed similar tendencies. XPS and DSC analyses suggested that crosslinking induced by UV in LEO was the main factor for the change of elongation.

(4) As for 10-months and 28-months exposures, the decrease of elongation of stressed samples was smaller than that of unstressed samples; however, the phenomena diminished after 46-months exposures. This indicated that tensile stress had a tendency to prevent the embrittlement in the early stage of exposure.

(5) Based on the ground tests, it was clarified that EB constituent in the ISS orbit hardly influenced the tensile properties.

(6) The amount of degradation of the flight samples was much smaller than estimated by using SEES program. The attitude change of the ISS during flight and the formation of contaminated layer of SiO_x were presented for the reason.

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