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Measurement and Evaluation of Thermal Control Material with Polyimide for Space Use

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Spacecraft orbiting around Mercury demand thermal control materials with ultraviolet ray resistance, particle irradiation resistance, and high-thermal stability. The chemical thermal stability of the polyimide film *s*-BPDA/PDA (Upilex-S®; Ube Industries, Ltd.), generated from biphenyltetracarboxylic dianhydride (*s*-BPDA) and *p*-phenylenediamine (PDA), is known to be higher than that of established thermal control materials, such as Kapton-H® and Upilex-R®. Based on experimental verification of the resistance to ultraviolet ray and protons and electrons irradiation, *s*-BPDA/PDA film is deemed appropriate for spacecraft aimed at interior planet explorations. Recovering of the degradation of the optical properties of the film after the protons irradiation is described. The role of radiation-generated radicals in the degradation of the film by protons is discussed.

Nomenclature

d	=	thickness of thin film, μm
k	=	extinction coefficient
n	=	refractive index
R	=	spectral reflectance when a light beam is incident vertically on a thin film
R_0	=	spectral reflectance when a light beam is incident vertically on a infinite extent surface, $[(n-1)^2+k^2]/[(n+1)^2+k^2]$
T	=	spectral transmittance when a light beam is incident vertically on a thin film
T_g	=	glass transition temperature, °C
$T_{20,000}$	=	temperature corresponding to the half-life period extrapolated to 20,000 h of tensile strength, °C
α	=	absorption coefficient, $4\pi k/\lambda$, cm^{-1}
α_S	=	solar absorptance
ε_H	=	total hemispherical emittance
ε_N	=	normal emittance
λ	=	wavelength, μm
σ	=	standard deviation

Introduction

WITH the advance of planetary explorations, spacecraft encounter expanded thermal and particles radiation environments. Mercury exploration spacecraft will receive intense solar illumination 10 times larger than that around the Earth.[1] The fluence of protons near Mercury, over 1.0 MeV, is about 200 times that around the geosynchronous orbit at solar maximum years.[1,2] Thermal control materials covering spacecraft for Mercury explorations are, therefore, required to be stable in the severe thermal environments and to have durability against both UV ray and proton irradiation.

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Until now, the irradiation degradation of α_S and ε_N has been evaluated for many kinds of thermal control materials.[3–5] It is well known that thermal control films based on polyimides have high thermal stability and particles irradiation resistance.[3,6] Kapton-H® (E. I. du Pont de Nemours and Company), made of pyromellitic dianhydride and 4,4′-oxydianiline (ODA), and Upilex-R® (Ube Industries, Ltd.), made of biphenyltetracarboxylic dianhydride (*s*-BPDA) and ODA, are typical polyimides that have been widely used in space. In the group of polyimides, however, we can find another one, made of *s*-BPDA and *p*-phenylenediamine (PDA), named Upilex-S® by Ube Industries, Ltd.

A known feature of Upilex-S is that it has chemical thermal stability higher than that of Kapton-H and Upilex-R. The chemical thermal stability of polymer film is evaluated either by the Arrhenius plots of tensile strength half-life period or by the thermogravimetric analysis. In the former, $T_{20,000}$, defined as the temperature that corresponds to the half-life period extrapolated to 20,000 h of tensile strength in the Arrhenius plots, is used as a specifying index. $T_{20,000}$ of Kapton-H, Upilex-R, and Upilex-S[7,8] are shown in Table 1. In Table 1, we see that $T_{20,000}$ of Upilex-S is higher than that of Kapton-H and Upilex-R, with a difference of 20–25°C. The thermogravimetric analysis also indicates that Upilex-S has higher thermal stability.[8]

In this paper we present optical properties of the Upilex-S film and their degradation under UV and particle irradiation. There are few reports that measured and evaluated details about Upilex-S.[9] Here Upilex-S is called *s*-BPDA/PDA to indicate the chemical structure of the film clearly. The films we have tested are *s*-BPDA/PDA itself, *s*-BPDA/PDA coated with aluminum on the back surface [(*s*-BPDA/PDA)/Al], and *s*-BPDA/PDA coated with aluminum on the back surface and with indium tin oxide (ITO) on the front surface [ITO/(*s*-BPDA/PDA)/Al)]. The thickness of *s*-BPDA/PDA in the first and the second cases is 20 μm and in the third case is 25 μm. The thickness of the aluminum layer is 1000 Å. The films, *s*-BPDA/PDA and (*s*-BPDA/PDA)/Al, have been evaluated in laboratories, and ITO/(*s*-BPDA/PDA)/Al has been evaluated on a spacecraft orbiting around Earth. We will describe the experimental method and results and discuss the features of *s*-BPDA/PDA as a thermal control material. We will also discuss recovery of the degradation that we have observed on the film irradiated by protons.

Basic Characteristics of (*s*-BPDA/PDA)/Al

The chemical structure of the polyimide *s*-BPDA/PDA is shown in Fig. 1. When a film is used as a thermal control material,

Table 1 $T_{20,000}$ of polyimide film[7,8]

Material	Density, g/cm^3	$T_{20,000}$, °C
Kapton-H	1.42	265
Upilex-R	1.39	270
Upilex-S	1.47	290

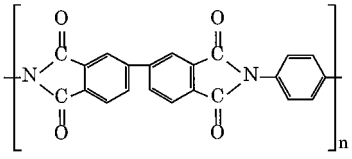

Fig. 1 Chemical structure of *s*-BPDA/PDA polyimide.

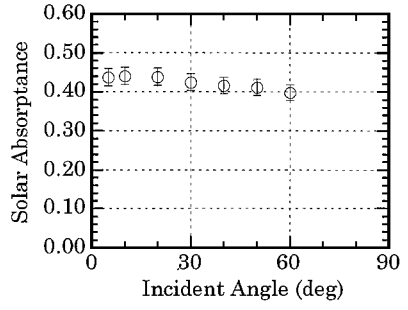

Fig. 2 Incident angle dependence of solar absorptance of (*s*-BPDA/PDA)/Al.

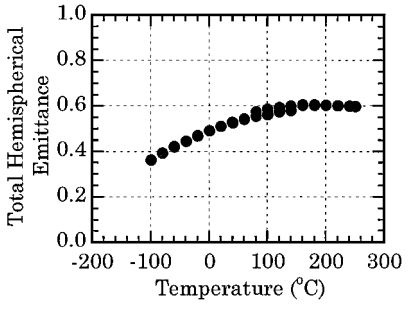

Fig. 3 Temperature dependence of total hemispherical emittance of (*s*-BPDA/PDA)/Al.

absorptance in the visible wavelength region and emittance in the infrared wavelength region are the key parameters that we should know. Therefore, we have measured the incident angle dependence of α_S and the temperature dependence of ε_H of (*s*-BPDA/PDA)/Al using a monochrometer with an integrating sphere[10] and a calorimetric method,[11] respectively.

Figure 2 is the α_S of the film obtained for the incident angles from 5 to 60 deg. The α_S has been derived from the measured spectral reflectance in wavelengths from 0.26 to 2.5 μm. The ε_H of the film has been derived as shown in Fig. 3. The ε_H has been measured for the temperatures from −100 to +250°C. Because the imide and phenyl group in polyimide films has a strong infrared absorption band in the wavelength from 5 to 10 μm, the ε_H has a peak around 180°C in its temperature dependence.

Experiments

Laboratory Test

Evaluation of Optical Properties

To measure the changes of optical properties of the film by UV and by protons and electrons irradiations has been the objective of the experiments. On the *s*-BPDA/PDA film, change of the absorption coefficient α, defined subsequently, has been investigated. When a light beam is incident vertically on a thin film, the spectral reflectance R and the spectral transmittance T of the film are given by[12]

$$R = R_0 + \frac{R_0(1-R_0)^2\{1+(k/n)^2\}\exp(-2\alpha d)}{1-R_0^2\exp(-2\alpha d)} \tag{1}$$

$$T = \frac{(1-R_0)^2\{1+(k/n)^2\}\exp(-\alpha d)}{1-R_0^2\exp(-2\alpha d)} \tag{2}$$

In Eqs. (1) and (2), R_0 and α are defined by

$$R_0 = \frac{(n-1)^2+k^2}{(n+1)^2+k^2} \tag{3}$$

$$\alpha = \frac{4\pi k}{\lambda} \tag{4}$$

From a set of measurements on R and T, we can derive a set of values on n and k using Eqs. (1) and (2). From k, we obtain the absorption coefficient α. In Eqs. (1) and (2), multiple reflections at the boundaries are taken into account. We have used the Fourier transform spectrometer (FTS-60A/896; Bio-Rad Laboratories, Inc.) to measure R and T in the wavelength region from 0.25 to 100.0 μm. In the measurement of R, the specular reflectance accessory (Graseby Specac, Ltd.) was used, and the incident angle was set at 12.5 deg, for which we confirmed that Eqs. (1) and (2) could still be applied with practically no corrections.

On the (*s*-BPDA/PDA)/Al film, changes of α_S and ε_N have been studied because the degradation of thermal control films in laboratory tests is generally evaluated by α_S at a fixed incident angle and the emittance of normal direction ε_N at room temperature. (The incident angle dependence of α_S and the temperature dependence of total hemispherical emittance ε_H are the parameters necessary to design spacecraft.) Both α_S and ε_N have been derived from the spectral reflectance measured by using the Fourier transform spectrometer (FTS-60A/896) with a variable angle reflection accessory (The Seagull™; Harrick Scientific Corporation). The wavelength region of the spectral reflectance measurements was 0.25–3.3 μm to derive α_S and 1.6–100.0 μm to derive ε_N. The incident angle of the measurement was 20 deg.

UV and Particles Exposure

We have assumed that UV radiation from the sun, terrestrial trapped particles, and solar flare protons are the main radiations affecting the properties of the film. The size of test sample is 30 × 30 mm. We have conducted the UV and low-energy electrons irradiation experiments at the Institute of Space and Astronautical Science (ISAS) using the apparatus shown in Fig. 4. The sample is mounted on a heat sink. The UV source is a 2-kW Hg–Xe short arc lamp, and its intensity can be changed from 0.2 to 14.0 times solar constant in the wavelength region of 0.2–0.5 μm. The UV source intensity is uniform within 10% all over the sample surface. The sample temperature is about 80°C. The acceleration voltage of the low-energy electron accelerator can be varied from 2.0 to 20 kV. The vacuum chamber containing the sample is kept at the pressure of 1.3×10^{-5} Pa. The irradiation beam scans the whole area of a sample. The margin of error of irradiation energy is ±0.5 keV.

The high-energy electrons irradiation tests were conducted by using a Cockcroft–Walton accelerator, with acceleration voltages from 0.5 to 2.0 MV, at the Japan Atomic Energy Research Institute.

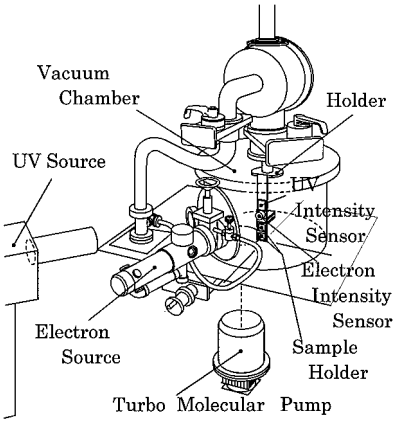

Fig. 4 Experimental apparatus.

Table 2 Radiations irradiated on *s*-BPDA/PDA film

Source	Radiation energy	Fluence
UV	8.9 SC[a]	5970 ESH
Electron	20 keV	67 μA
	0.5 MeV	1.0×10^{15} electrons/cm^2
	2.0 MeV	
Proton	0.5 MeV	5.0×10^{14} protons/cm^2
	1.0 MeV	
	2.0 MeV	

[a]Solar constant.

Table 3 Radiations irradiated on (*s*-BPDA/PDA)/Al

Source	Radiation energy	Fluence
UV	9.3 SC	8530 ESH
Proton	0.5 MeV	5.0×10^{14} protons/cm^2
	1.0 MeV	1.0×10^{13} protons/cm^2
	2.0 MeV	3.0×10^{12} protons/cm^2

The irradiation uniformity is less than 3% on the whole area of the sample. The sample is mounted on a heat sink, and its temperature is less than 50°C. The protons irradiation tests were performed by using a tandem accelerator at the Osaka National Research Institute. The generated energy of the protons is from 0.5 to 2.0 MeV. The irradiation beam scans the whole area of the sample. The sample is also mounted on a heat sink, and its temperature is under 50°C. The accuracy of irradiation energy is within 1%.

In the evaluation of the *s*-BPDA/PDA film, we exposed the film to the radiations shown in Table 2. The UV source intensity was 8.9 times solar constant, and the accumulated flux was 5970 equivalent sun hour (ESH), where ESH is equal to the product of exposed time and the UV source intensity. Thus, 5970 ESH corresponds to an exposure time of 311 days on a 500-km high circular Earth orbit. The fluence of the high-energy electrons, 1.0×10^{15} electrons/cm^2, is equivalent to that of 16 years for 0.5 MeV and of 1060 years for 2.0 MeV in the case of geosynchronous Earth orbit.[13] The fluence of proton, 5.0×10^{14} protons/cm^2, is equivalent to that of 158 years for 0.5 MeV and 15,800 years for 1.0 MeV in the case of geosynchronous Earth orbit[2] and is equivalent to 625 years for 1.0 MeV and 2500 years for 2.0 MeV around Mercury.[1,2]

In the evaluation of the (*s*-BPDA/PDA)/Al film, we exposed the film to the radiations shown in Table 3. The UV source intensity is 9.3 times solar constant, and the fluence was 8530 ESH. The electrons irradiation test was omitted because we noted from the *s*-BPDA/PDA experiment that the effect of the protons irradiation covers the effect of the electrons irradiation. The fluence of the protons is equivalent to that for about 10 years around Mercury at solar maximum years. Our irradiation test was conducted under a high dose rate compared with the expected space environment. The degree of the effect of the dose rate generally depends on the sample temperature during irradiation. In our test, the sample temperature was much less than $T_{20,000} = 290$°C of the polyimide film, and the high dose rate has no influence on the test results.

Test in Space

A test of the thermal control film ITO/(*s*-BPDA/PDA)/Al has been conducted on the Earth-orbiting satellite Akebono, which was launched by ISAS in February 1989. The Akebono's orbit is a sub-polar one with a perigee height of 300 km and an apogee height of 10,000 km. The spacecraft has frequently passed through the Van Allen radiation belts. A control using magnetic torquers has pointed the spin axis of the spacecraft toward the sun with a deviation angle less than 3.0 deg. The film has been placed on the top plate of the spacecraft, facing the sun. The α_S and the ε_H have been measured by a calorimetric method, which utilizes solar illumination and an electrical heater attached to the sample. Both α_S and ε_H are derived by giving several different levels of input power to the sample heater, with no need of parameters such as the mass and the specific heat of the sample materials.[14] The error in α_S and ε_H are estimated to be $\pm 5\%$.

Results and Discussion

Laboratory Test

s-BPDA/PDA Film

Experimental results. After the *s*-BPDA/PDA film was exposed to the radiations given in Table 2, the absorption coefficient of the samples was measured before the recovery process, described later, proceeded.

At the wavelength region of 0.4–0.7 μm, the original film has an absorption coefficient as shown in Fig. 5. In the following, we give the change of the absorption coefficient as the increase from the absorption before irradiations. When the film is affected by the radiations, its color changes from yellowish brown to dark brown. First, the UV irradiation yielded little increase in the absorption coefficient of the film, as shown in Fig. 6. The low-energy electrons yielded a noticeable increase in the absorption coefficient, though the high-energy electrons yielded little. The behavior is shown in Fig. 7. The increase by the 20-keV electrons exposure arises around 0.44 μm. The high-energy electrons have little effect. These results are consistent with the range of electrons[15] that is estimated to be 1200 μm for 0.5 MeV and 7000 μm for 2.0 MeV, which is far larger than the thickness of the film (20 μm). The estimated range of 20-keV electrons is 5 μm.

The effect of the protons irradiation was larger than that of the low-energy electrons. Figure 8 shows the increase of the absorption coefficient by the irradiation of the three energies of protons. The increase in the case of 1.0-MeV protons is highest among the three cases. The increase of absorption coefficient is observed around the 0.42-μm wavelength, and the increase reduces monotonically

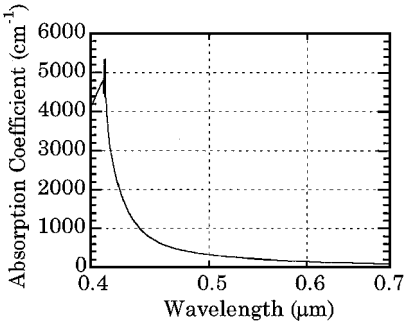

Fig. 5 Absorption coefficient of *s*-BPDA/PDA film.

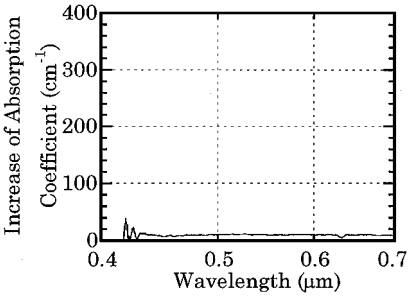

Fig. 6 Effect of UV irradiation on absorption coefficient of *s*-BPDA/PDA film.

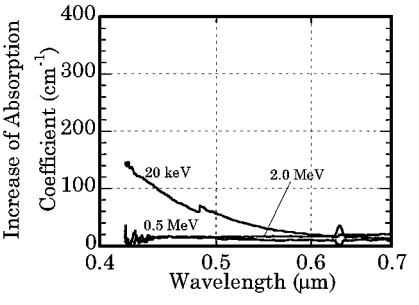

Fig. 7 Effect of electron irradiation on absorption coefficient of *s*-BPDA/PDA film.

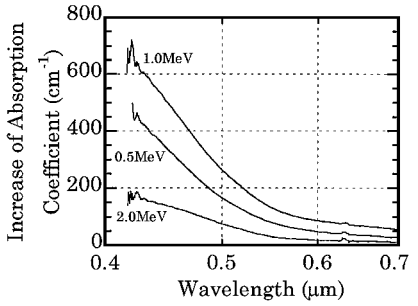

Fig. 8 Effect of proton irradiation on absorption coefficient of *s*–BPDA/PDA film.

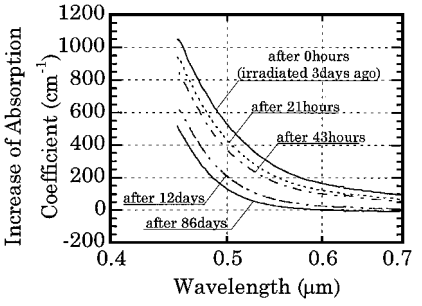

Fig. 9 Recovery of the degradation in the air and under a room light.

toward the longer wavelengths. The range, the implanted depth, of protons is calculated using the Monte Carlo code, TRIM. The TRIM code is a conventional simulation to calculate the projected range of ions. The range of 0.5-, 1.0-, and 2.0-MeV protons has been estimated to be 6.25, 17.7, and 55.4 μm, respectively. Trapping by the film is closely related to the observed degradation.

In the infrared wavelength region from 2.5 to 100.0 μm, no noticeable change in the absorption coefficient was observed after the UV, electrons, and protons irradiations.

Recovery of degradation. It is known that the degradation of the thermal control film by radiation exposure is recovered partially with time.[3] When the laboratory for the optical measurements and accelerators are far apart and much time is needed to transport the exposed film, this recovery phenomenon would obscure accurate evaluation of the degradation.

We have experimentally studied the recovery of the degradation of the film, selecting *s*–BPDA/PDA irradiated with the 1.0-MeV protons as a sample. The fluence was 1.5×10^{15} protons/cm^2. Three days after the irradiation, we started to measure the recovery of degradation. For the three days after the irradiation, the sample was stored in a vacuum and dark environment. The absorption coefficient was measured under some temporal intervals until the 86th day from the start. Figure 9 shows the result of the measurements, including at the start, after 21 h, after 43 h, after 12 days, and after 86 days. The sample was placed in the atmosphere and under a room light for these 86 days. We observe clear proceeding of the recovery in Fig. 9, and note that the recovery is substantial. After 86 days, degradation reduces to roughly one-half or one-third in the wavelength region shorter than 0.5 μm and is recovered almost completely at the wavelength longer than 0.6 μm.

We have also confirmed that the recovery depends on the storage condition of the film after the irradiation. We examined four kinds of storage conditions: combinations of in air or in vacuum and in light or in darkness. Figure 10 shows the absorption coefficient after 12 days and Fig. 11 after 86 days. Figures 10 and 11 indicate that no recovery occurs in vacuum and in darkness until the 12th day from the start, storage in air substantially influences the recovery, and storage in darkness delays the recovery slightly.

The experimental results on the protons and high-energy electron irradiation described are obtained for the sample after three days have elapsed after irradiations. Because the samples were kept in vacuum and in dark environments for these days, the presented results are to be considered as before the recovery process proceeded on the film.

Table 4 Glass transition temperature of polyimide film[8]

Material	T_g, °C
Kapton-H	428
Upilex-R	303
Upilex-S	359

Table 5 Measured α_S and ε_N of (*s*–BPDA/PDA)/Al

Absorption/emittance	Mean	σ
α_S (at 20 deg)	0.407	0.004
ε_N (at RT)	0.582	0.006

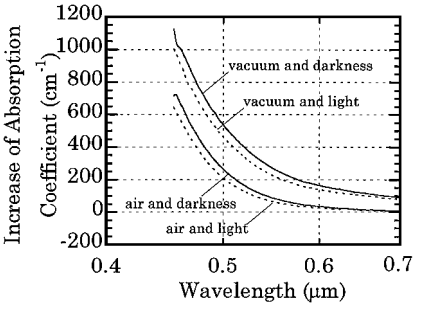

Fig. 10 Recovery under four different storage conditions after 12 days.

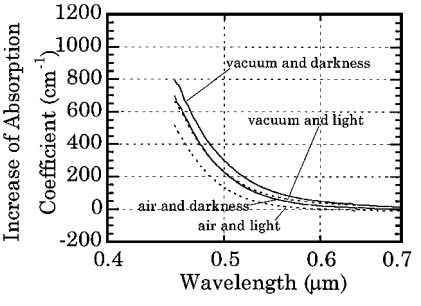

Fig. 11 Recovery under four different storage conditions after 86 days.

Cause of degradation. Because the increase of the absorption coefficient arises around 0.44 μm and the recovery of the degradation takes many days extending over 3 months, we estimate that generation of radicals including benzene nucleus in its chemical structure is the cause of the degradation. It is known that the radicals mentioned earlier exhibit an absorption band located between 0.40 and 0.50 μm (Ref. 16). In addition, it is known that the radiation-generated radicals in a polymer film are long lived because of their lack of mobility below a glass transition temperature. As a reference, we give the glass transition temperature of polyimide film[8] in Table 4. At room temperature (RT), the lifetime of the radiation-generated radicals is known to be from a few weeks to a few months.[17] The decay of the radiation-generated radicals on Kapton-H and Upilex-R was experimentally measured by other investigators, and their long lives, extending a few months, were confirmed.[18,19]

(s–BPDA/PDA)/Al Film

The film (*s*–BPDA/PDA)/Al is the material that can be actually used for the thermal control of spacecraft. We have tested *s*–BPDA/PDA film of 20 μm thickness with an aluminum coating of 1000 Å thickness on the back surface. The α_S and the ε_N of the film have been derived from the measured spectral reflectance, and the results are given in Table 5. The values in Table 5 are obtained from the measurement of 10 samples at RT. For reference, we show examples of the measured spectral reflectance of the film in Figs. 12 and 13.

Next, the (*s*–BPDA/PDA)/Al was exposed to the UV and the protons. The spectral reflectance of the irradiated samples was measured before the recovery of the degradation began as already mentioned. Table 6 summarizes α_S and ε_N before and after irradiation.

Table 6 Before and after irradiation: α_S and ε_N of (*s*-BPDA/PDA)/Al

Source	Radiation energy, MeV	α_S			ε_N		
		Before	After	$\Delta\alpha_S$	Before	After	$\Delta\varepsilon_N$
Proton	0.5	0.409	0.449	+0.040	0.585	0.569	−0.016
	1.0	0.411	0.418	+0.007	0.580	0.569	−0.011
	2.0	0.404	0.403	−0.001	0.575	0.561	−0.014
UV		0.407	0.417	+0.010	0.585	0.582	−0.003

Table 7 Before and after irradiation: α_S and ε_N of (*s*-BPDA/PDA)/Al and Upilex-R/Al

Material	α_S			ε_N		
	Before	After	$\Delta\alpha_S$	Before	After	$\Delta\varepsilon_N$
(*s*-BPDA/PDA)/Al	0.409	0.449	+0.040	0.585	0.569	−0.016
Upilex-R/Al	0.335	0.366	+0.031	0.653	0.642	−0.011

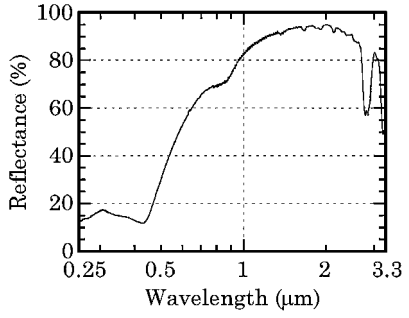

Fig. 12 Spectral reflectance of (*s*-BPDA/PDA)/Al for 0.25–3.3 μm.

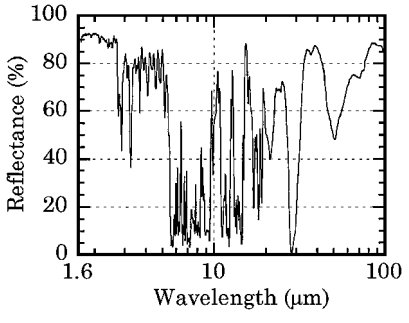

Fig. 13 Spectral reflectance of (*s*-BPDA/PDA)/Al for 1.6–100.0 μm.

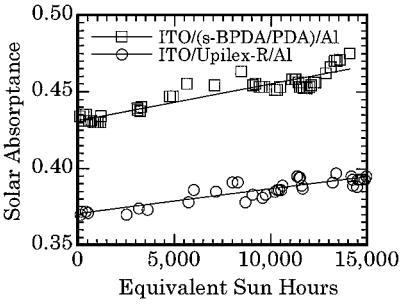

Fig. 14 Solar absorptance of ITO/(*s*-BPDA/PDA)/Al and ITO/Upilex-R/Al on the spacecraft Akebono.

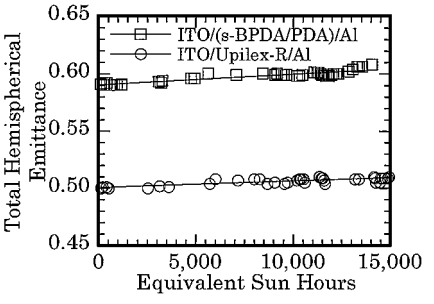

Fig. 15 Total hemispherical emittance of ITO/(*s*-BPDA/PDA)/Al and ITO/Upilex-R/Al on the spacecraft Akebono.

We note from Table 6 that the changes of α_S and ε_N are small: The largest change is a 10% increase of α_S in the case of 0.5-MeV irradiation; ε_N decreases by the protons irradiation, but less than 2%, α_S increases about 2.5% by UV; and ε_N does not change by UV. For the case of 0.5-MeV protons irradiation, at which the largest changes of α_S and ε_N occurred in the (*s*-BPDA/PDA)/Al film, we compared the degradation of (*s*-BPDA/PDA)/Al with that of the Upilex-R/Al film, which is the standard thermal control film that ISAS has used together with Kapton/Al. The comparison, given in Table 7, shows that the changes of α_S and ε_N in (*s*-BPDA/PDA)/Al is larger than that in Upilex-R/Al. The magnitude of the changes observed in (*s*-BPDA/PDA)/Al is, however, considered to be in an allowable range from a spacecraft thermal control point of view, and (*s*-BPDA/PDA)/Al is considered to be similarly applicable to thermal control of spacecraft. Considering the excellent heat resistance of *s*-BPDA/PDA, we can say that the (*s*-BPDA/PDA)/Al film will be effectively applied to the thermal control of spacecraft, especially for interior planet missions, such as Mercury explorations, where sufficient tolerance for radiations is required.

Flight Test

The films ITO/(*s*-BPDA/PDA)/Al and ITO/Upilex-R/Al were tested on the satellite Akebono for 2 years after its orbit injection. The time variations of α_S and ε_H measured with onboard equipment are shown in Figs. 14 and 15, where the time is expressed in ESH. For 14,000 ESH, α_S of ITO/(*s*-BPDA/PDA)/Al and ITO/Upilex-R/Al increased by 0.036 and 0.024, respectively, whereas ε_H of ITO/(*s*-BPDA/PDA)/Al and ITO/Upilex-R/Al increased by 0.014 and 0.009, respectively. The degradation in the ground and the flight tests has shown the same tendency, that is, the change of α_S and ε_H (or ε_N) in the thermal control film using *s*-BPDA/PDA is larger than that using Upilex-R, although both are relatively large compared to those observed in the laboratory experiments.

Conclusions

We have demonstrated that the polyimide film *s*-BPDA/PDA is appropriate for the thermal control of spacecraft aimed at Mercury explorations or explorations under similar environments, where thermal control materials demand more severe qualities for UV rays resistibility, high-energy particle resistibility, and high thermal stability. A feature of *s*-BPDA/PDA is that its chemical thermal stability is higher than that of established thermal materials, such as Kapton-H and Upilex-R. The radiations exposure experiments on the film *s*-BPDA/PDA with aluminum coating on the back surface, together with the *s*-BPDA/PDA film itself, have shown that the degradation of the film by UV, electrons, and protons irradiations is well within an allowable level for the expected space missions

(decade years around Mercury). The film was also tested on an Earth-orbiting spacecraft for 14,000 ESH.

We have also investigated the recovery of the degradation (solar absorptance increase) of the protons irradiated film. From the timescale of the recovering and the spectrum of the solar absorptance increase, we have estimated that the degradation is due to the generation of radiation-generated radicals.

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