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Comparison of Polyethylene and Polyimide as a Fluence Monitor of Atomic Oxygen

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Synergistic effect on atomic oxygen-induced erosion of polyethylene and polyimide with 172 nm monochromatic ultraviolet exposure was compared. The erosion of these materials was measured by a quartz crystal microbalance under 5 eV simultaneous atomic oxygen and ultraviolet exposures. The effect of simultaneous ultraviolet exposure on the atomic oxygen-induced erosion was observed at the ultraviolet intensity of $0.5–2.6 \times 10^{-15}$ mJ/atom for polyethylene. In contrast, the similar effect was observed in polyimide at the relative ultraviolet intensities one order greater than that of polyethylene. It was found that the effect of 172 nm ultraviolet was different in polyimide and polyethylene, i.e., photoinduced erosion was observed only for polyethylene. It was concluded that polyimide is less sensitive with simultaneous ultraviolet exposure during atomic oxygen bombardment, and thus evaluated as a better material for measuring atomic oxygen fluence.

I. Introduction

It has been recognized that atomic oxygen is one of the most important hazards to the spacecraft polymeric materials in low Earth orbit (LEO). In-orbit exposure tests have been carried out regarding atomic oxygen effect on materials, and polyimide has been used for measuring the atomic oxygen fluence in such in-orbit tests. The fluence of atomic oxygen in LEO has been calculated from the polyimide erosion with the established reaction efficiency of 3.0×10^{-24} cm^3/atom (Kapton equivalent fluence) [1]. As for the reference material to measure atomic oxygen fluence, the erosion yield of polyimide has to be stable against the other environmental factors such as temperature, ultraviolet, electron, ions, radiation, and so on.

However, the synergistic effect of other environmental factors on the atomic oxygen-induced erosion of polyimide is under investigation. For example, the erosion yield of polyimide in the temperature range between -30 to $+100$°C is constant and the activation energy is as low as 10^{-3} eV [2]. However, increase in the erosion yield of polyimide at the temperature above 120°C was reported [3]. It is, therefore, important to maintain the temperature below 100°C when measuring atomic oxygen fluence with polyimide. On the other hand, the exposure condition to avoid synergistic effect with ultraviolet has not been established. In LEO, no synergistic effect was reported [4], however, it is not applicable to all exposure conditions. In contrast, a 30% increase in erosion by simultaneous ultraviolet exposure has been reported in ground-based experiments [5]. Yokota et al. [6] have recently investigated a quantitative analysis of polyimide erosion in the simultaneous exposure of atomic oxygen and ultraviolet. They found that the synergistic effect of atomic oxygen and ultraviolet (monochromatic) shows intensity dependence, i.e., the synergistic effect of atomic oxygen and ultraviolet was obvious in polyimide when the relative intensity of ultraviolet exceeds 10^{-14} mJ/atom. In their experiment, a two-step reaction was suggested in the mass-loss reaction of polyimide; gasification reaction following oxidation reaction. In these reaction steps, hyperthermal (5 eV) atomic oxygen is responsible for both steps, however, ultraviolet exposure is responsible only for the gasification reaction. The reaction dynamics of atomic oxygen-induced mass-loss reaction of polyimide under the presence of ultraviolet is complicated.

Polyimide seems not to be an ideal material to measure atomic oxygen fluence. Grossman et al. [7] discussed this point based on their experimental results, but their atomic oxygen source was a thermal atomic oxygen source so that effect of translational energy was not taken into consideration. In the atomic oxygen testing protocol [1], polyethylene is also listed as a monitoring material for atomic oxygen fluence because of its simplest chemical structure of all polymers. Because of the simplicity of its chemical structure, polyethylene is a good material to study atomic oxygen-induced reaction with ultraviolet. In a previous paper, we found that the synergistic effect of atomic oxygen and ultraviolet was obvious in polyethylene [8].

In this paper, polyethylene and polyimide were compared as an atomic oxygen fluence monitoring material under the bombardment of hyperthermal atomic oxygen and 172 nm ultraviolet radiation. The effect of simultaneous ultraviolet exposure on the atomic oxygen-induced erosion of polyethylene and polyimide was discussed based on the ground-based experimental results.

II. Experimental Details

The samples used in this experiment were low-density polyethylene (LDPE) and pyromellitic dianhydride-oxydianiline (PMDA-ODA) polyimide films. Both of the films were spin-coated on quartz crystal microbalance (QCM) sensor crystals. The polyethylene solution containing 0.3 g of LDPE (average molecular weight of 6500) in 40 ml xylene was prepared for polyethylene film. The polyimide, which was supplied by Toray Industries, Inc., Semicofine SP-510 was used. A precursor of PMDA-ODA polyimide was spin-coated on a QCM sensor crystal and then annealed at 150°C for 1 h and at 300°C for 1 h. Details of the sample preparation are reported in [6]. The polyimide film, thus prepared, showed an x-ray photoelectron spectrum similar to that of Kapton-H film, which is a commercially available polyimide film.

A space environment simulation facility at Kobe University was used in this study. The schematic drawing of the facility is shown in Fig. 1. This facility was equipped with a laser detonation atomic oxygen beam source, which was originally designed by Physical Sciences, Inc. as a hyperthermal atomic oxygen source [6]. The atomic oxygen beam was produced using a carbon dioxide laser (wavelength 10.6 μm, output power 5–7 J/pulse). The translational energy of the atomic oxygen was approximately 5 eV. The flux of the atomic oxygen beam was calculated to be 2×10^{14} atoms/cm^2/s at the sample position. These values are almost equivalent to those in

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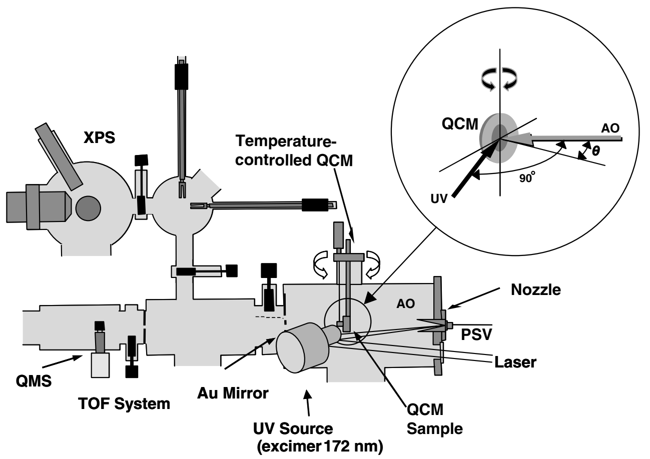


Fig. 1 Schematic drawing of space environmental simulation facility using laser detonation atomic oxygen beam source.

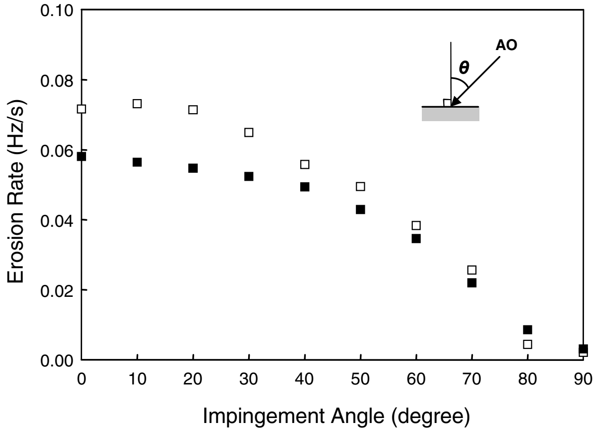
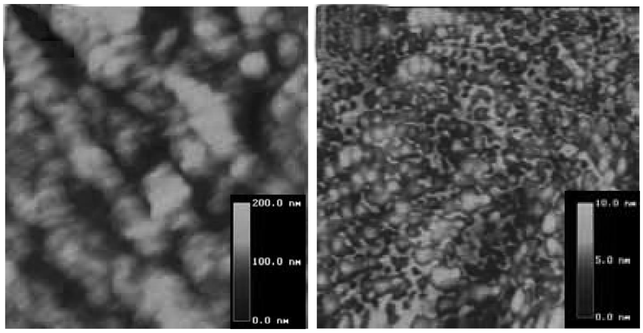


Fig. 2 Erosion rates of polyethylene and polyimide during atomic oxygen exposures measured from resonant frequency of QCM.



a) Polyethylene **b) Polyimide**

Fig. 3 Atomic force microscopic images of the atomic oxygen-exposed surfaces. Scan area, 500 × 500 nm.

LEO at the altitude of 200–300 km. An excimer light source with a wavelength of 172 nm was used as an ultraviolet source in this study [6]. This ultraviolet source was attached to the atomic oxygen source chamber. The ultraviolet radiation was exposed to the sample through an evacuated light guide to avoid absorption of vacuum ultraviolet by air (wavelength of <200 nm). Axes of the atomic oxygen beam and the ultraviolet crossed 90 deg and the sample was rotatable with the axis perpendicular both to atomic oxygen and ultraviolet (see the insert in Fig. 1). In this configuration, the effective atomic oxygen and ultraviolet fluxes can be changed by rotating the sample [6]. The maximum atomic oxygen flux and ultraviolet flux at the sample position were $6.0\text{–}14.0 \times 10^{14}$ $\text{atoms/cm}^2\text{/s}$ and $0.55\ \text{mW/cm}^2$, respectively. The erosion rate of the polymer film was calculated from the resonant frequency of QCM during the atomic oxygen beam and/or ultraviolet exposures. The sample temperature was kept at 311 K during the experiment by the temperature-controlled circulating water system. Before the mass-loss measurements, the polyethylene and polyimide films were exposed to atomic oxygen to saturate the surface oxygen content of the sample. This was to avoid the nonlinear effect in mass loss, which appears at the beginning of the atomic oxygen exposures at pristine polymer surfaces [9].

III. Results and Discussion

A. Atomic Oxygen-Induced Polymer Erosion

In the experiment reported herein, the relative intensity of atomic oxygen and ultraviolet was adjusted by rotating the QCM sample along with the axis, which is perpendicular to the axes both to atomic oxygen and ultraviolet. However, in this experimental configuration, the incident angle of atomic oxygen at polymer surface is changed when relative atomic oxygen/ultraviolet intensity is adjusted. Therefore, it is necessary to investigate the effect of incident angle of atomic oxygen and ultraviolet on the polymer erosion before the quantitative analysis of synergistic effect of atomic oxygen and ultraviolet.

From the previous result regarding the incident angle dependence of atomic oxygen on the erosion of polyimide-coated QCM, it was observed that the erosion rate is linear with time in every incident angle and the erosion rate decreased with increasing incident angle by a cosine distribution [10]. Figure 2 compares the incident angle dependence of atomic oxygen-induced erosion of polyethylene (solid square) and polyimide (open square) [8]. The experimental result on the incident angle dependence of atomic oxygen-induced erosion of polyethylene showed a similar tendency with those reported on polyimide film. However, it is not a simple cosine distribution ($\cos^{0.87}\theta$) compared with polyimide ($\cos^{1.0}\theta$). The fact that the incident angle dependence of the erosion rate follows closely to a cosine law indicates that the erosion rates of polyethylene and polyimide are both basically proportional to the effective flux of atomic oxygen. It was thus concluded that the reaction yield of atomic oxygen with these polymers is identical to the incident angle.

Figure 3 shows the atomic force microscopy images of polyethylene and polyimide films exposed to atomic oxygen. The atomic oxygen fluences are 3.0×10^{18} atoms/cm^2 for polyethylene and 8.8×10^{17} atoms/cm^2 for polyimide. It was obvious that the surfaces of the polymers were roughened by atomic oxygen exposure. Therefore, an actual incident angle of oxygen atom to a surface moiety is not directly related to macroscopic incident angle because the surface is not atomically flat due to the presence of microscale roughness. Thus, the macroscopic erosion rate of polymer simply reflects the effective fluence of atomic oxygen which follows cosine law with the macroscopic incident angle.

Also, it should be mentioned that the tilting of the sample reduces not only the effective flux of atomic oxygen, but also the effective incident energy (a normal component of translational energy). Lowering the incident angle from 0 to 60 deg reduces the effective incident energy from 5.0 to 1.25 eV; it is proportional to the cosine square of the incident angle. However, the activation energies of the gasification reaction of polyimide and polyethylene in this incident energy range are as low as 10^{-3} eV which are much lower than the effective translational energy [2]. Moreover, the presence of the surface microroughness of the film, which affects the microscale incident angle of atomic oxygen with surface, erases the effective incident energy dependence in the experimental data. Thus, it was considered that the reduction of effective incident energy due to the change in incident angle does not affect the reaction yield of atomic oxygen with polymers.

B. Ultraviolet-Induced Polymer Erosion

The effect of incident angle of ultraviolet radiation on polyethylene and polyimide erosion was also studied. Figure 4 shows the frequency shift of the polyimide-coated QCM after

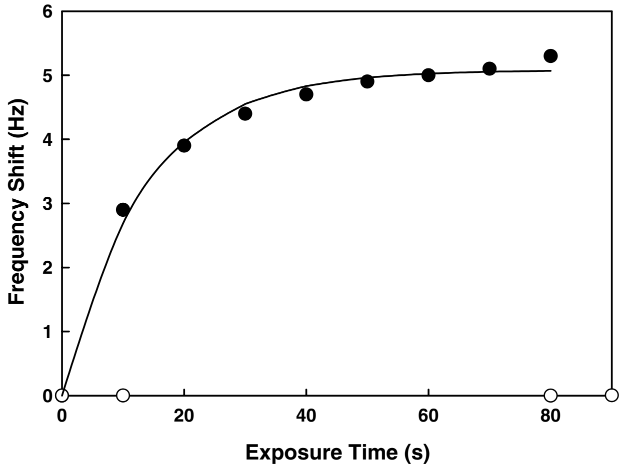


Fig. 4 Frequency shift of the polyimide-QCM during ultraviolet exposures.

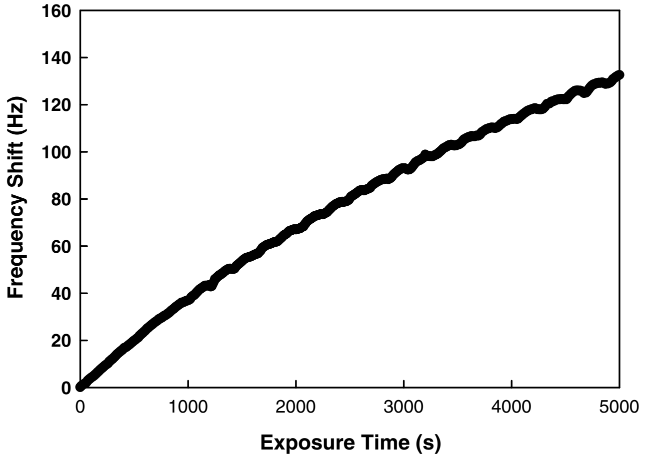


Fig. 5 Frequency shift of the polyethylene-QCM during ultraviolet exposures.

ultraviolet exposures at normal incidence (solid circle). The flux of 172 nm ultraviolet radiation was 4.5 mW/cm^2 at normal incidence. Note that the sample surface was exposed to atomic oxygen (2.3×10^{18} atoms/cm^2) before ultraviolet irradiation to avoid the nonlinear phenomena. As shown in Fig. 4, resonant frequency of the QCM was increased when ultraviolet starts irradiating due to the desorption of adsorbed gaseous molecules. However, the slope of the curve gradually decreases with time. It has been analyzed that the curve can be fit in the form of

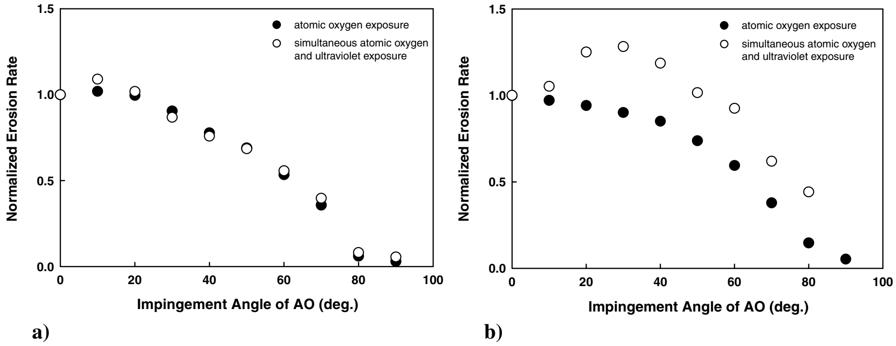
$$\Delta f = A[1 - \exp(-ct)] \tag{1}$$

where Δf is the frequency shift of the QCM, A and c are the constant, and t is the exposure time of ultraviolet radiation [6]. The fact that the experimental data was fit by Eq. (1) suggests that the surface oxidizing species (carbon dioxide) are desorbed by ultraviolet exposure (photoinduced desorption). This is confirmed by the fact that no mass loss was detected by the ultraviolet exposure without the preoxidation process (open circle in Fig. 4). The incident angle dependence of ultraviolet radiation was also examined. By increasing the incident angle of ultraviolet radiation, desorption rate of the oxidized species is decreased. This is explained by the decrease in effective photon flux of ultraviolet radiation.

In contrast, polyethylene showed a different tendency of mass change with ultraviolet exposure. Figure 5 shows a typical mass change of polyethylene under 172 nm ultraviolet exposure (4.5 mW/cm^2). The polyethylene surface was exposed to atomic oxygen (5.9×10^{17} atoms/cm^2) before ultraviolet irradiation. Mass of the polyethylene shows a decrease with exposure time. Unlike polyimide, the saturation of mass loss is not obvious in polyethylene. The absolute mass lost by the ultraviolet exposure was much greater than that of polyimide. This mass-loss property suggests that not only the adsorbed gaseous molecules, but also the polyethylene structure, is decomposed by the 172 nm ultraviolet exposure.

C. Synergistic Effect of Atomic Oxygen and Ultraviolet

Atomic oxygen and ultraviolet beams irradiated polymer-coated QCM simultaneously and the erosion rate was recorded by the resonant frequency of QCM. The maximum atomic oxygen flux and ultraviolet flux at the sample position were 6.8×10^{14} atoms/cm^2/s and 0.55 mW/cm^2, respectively. Figures 6a and 6b show the erosion rates of polyimide and polyethylene under simultaneous atomic oxygen and ultraviolet exposure conditions as a function of incident angle of atomic oxygen. The abscissa is the incident angle of atomic oxygen, i.e., the incident angle of 0 deg means 100% atomic oxygen and 0% ultraviolet, and the incident angle of 90 deg means 0% atomic oxygen and 100% ultraviolet. The longitude is the normalized erosion rate. If no ultraviolet effect was observed, the data points with ultraviolet exposure (open circle) should follow the cosine function. This is the case for polyimide (Fig. 6a). In contrast, the erosion rates with ultraviolet exposure (open circle) do not follow the cosine function and are 30–100% greater than those without ultraviolet exposures (solid circle) in the case of polyethylene (Fig. 6b). It was clear that simultaneous ultraviolet exposure affects the atomic oxygen-induced erosion of polyethylene. The relationship between the relative intensities of ultraviolet per atomic oxygen (UV/AO ratio) and the normalized erosion rate was replotted in Fig. 7. The erosion rate of atomic oxygen-induced polyethylene was enhanced 30–80% at the relatively low UV intensities at the UV/AO ratio of $0.5\text{–}2.6 \times 10^{-15}$ mJ/atom, where polyimide does not show any enhancement of mass loss with ultraviolet exposure. As reported elsewhere [6], the synergistic effect of atomic oxygen and 172 nm ultraviolet on polyimide was obvious when ultraviolet intensity is higher than 1×10^{-14} mJ/atom. However, it was not obvious in the UV/AO ratio lower than 10^{-15} mJ/atom, as demonstrated in Fig. 7. In contrast, polyethylene shows a clear synergistic effect of ultraviolet irradiation even in the UV/AO ratio lower than 10^{-15} mJ/atom. An ultraviolet intensity in this study was compared



a) **b)**

Fig. 6 Normalized erosion rates of a) polyimide and b) polyethylene.

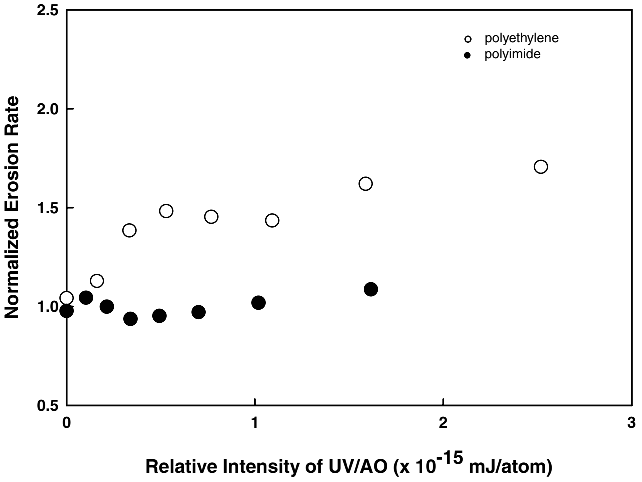


Fig. 7 Relationship between the relative intensity of UV/AO and the normalized erosion rate.

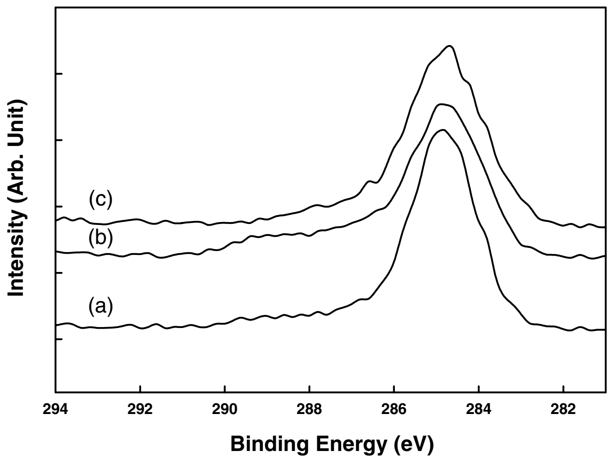


Fig. 8 C1s XPS spectra of atomic oxygen-exposed polyethylene before and after ultraviolet exposure.

with an LEO environment. The ultraviolet intensity between 175.4 and 190.5 nm ($\Delta\lambda = 14.9$ nm) in LEO is reported to be 3.1×10^{-3} mW/cm^2 [11]. On the other hand, the excimer lamp used in this experiment has an emission spectrum with a center wavelength of 172 nm and full width at half maximum of 14 nm. The ultraviolet intensity of this lamp is estimated to be 0.55 mW/cm^2 at the sample position. This is two orders greater than that included in an actual solar radiation when the effect of neighboring wave length is ignored. In contrast, atomic oxygen flux in this study is 6.8×10^{14} atoms/cm^2/s, which is one order greater than that in the altitude of the International Space Station (ISS) (5.6×10^{13} atoms/cm^2/s). The UV/AO ratio in ISS orbit is evaluated to be in the range of 10^{-16} mJ/atom, where the erosion rate of polyethylene increases by ultraviolet as shown in Fig. 7. It is, therefore, concluded that a polyethylene in ISS orbit would show synergistic effect of atomic oxygen and ultraviolet on erosion, whereas, in contrast, it would not be obvious on polyimide.

D. Reaction Dynamics of Atomic Oxygen and Ultraviolet with Polyethylene and Polyimide

X-ray photoelectron spectroscopy (XPS) was used for analyzing the polyethylene surfaces before and after ultraviolet exposures. The experiment was carried out with an atomic oxygen fluence of 3.0×10^{18} atoms/cm^2. Figure 8 indicates the C1s core level XPS spectra of atomic oxygen-exposed polyethylene before and after ultraviolet exposure (as shown: a) pristine, b) after atomic oxygen exposure, and c) after ultraviolet exposure following atomic oxygen exposure). As clearly observed in Fig. 8, atomic oxygen-exposed polyethylene showed a high-energy shoulder at 288.5 eV besides the main peak at 284.8 eV. This shoulder is contributed to by a carboxyl group [12]. It is obvious that the high-energy peak disappeared after ultraviolet irradiation. Similar results were obtained from polyimide samples. Table 1 shows the composition of the polyethylene and polyimide surfaces analyzed by XPS. After atomic oxygen exposure, the oxygen composition increased to 27.2% for polyethylene and to 33.3% for polyimide. The high-energy shoulder disappeared or decreased after ultraviolet exposure, and the surface oxygen composition was recovered to the normal value (27.2 → 10.3% for polyethylene and 33.3 → 26.6% for polyimide).

Table 1 Surface composition of polyethylene and polyimide before/after atomic oxygen and ultraviolet exposures

	Polyethylene		Polyimide		
Samples	Atomic percent (%)		Atomic percent (%)		
	C	O	C	O	N
Pristine	92.8	7.2	75.5	17.8	6.7
AO exposed[a,b]	72.8	27.2	61.2	33.3	5.5
AO → UV exposed[c,d]	89.7	10.3	66.9	26.6	6.5
UV exposed[c,d]	92.5	7.5	75.6	16.6	7.8

[a] Polyethylene, AO fluence 3.0×10^{18} atoms/cm^2
[b] Polyimide, AO fluence 9.6×10^{17} atoms/cm^2
[c] Polyethylene, UV intensity 0.45 mW/cm^2 for 40 min.
[d] Polyimide, UV intensity 0.6 mW/cm^2 for 4 hrs.

The experimental results using QCM and XPS are summarized as follows: 1) hyperthermal atomic oxygen exposure oxidizes the polymer surfaces and leads to the desorption of volatile oxidized products; 2) 172 nm ultraviolet exposure promotes the decomposition and desorption of surface functional groups, but it does not affect the mass of polyimide if the surface was not oxidized; and 3) 172 nm ultraviolet exposure affects the mass of the polyethylene even without atomic oxygen exposure.

These findings lead to the following conclusions: hyperthermal atomic oxygen reacts both to polyimide and polyethylene. These reactions are basically the similar oxidization processes. In contrast, the effect of 172 nm ultraviolet is different in polyimide and polyethylene. Ultraviolet of 172 nm is effective only for the desorption process of the reactive products from the polyimide surface, i.e., the polyimide structure itself is not decomposed by 172 nm ultraviolet alone. On the other hand, 172 nm ultraviolet radiation decomposes polyethylene. This is due to the fact that 172 nm ultraviolet can be directory absorbed by C–C bonds, which are the main polyethylene backbone structure. This photoinduced decomposition pathway is one of the reasons for the synergistic effect on polyethylene shown in Fig. 6b as pointed out by the previous study [8]. Beside these atomic oxygen-induced and photoinduced scission of the polyethylene backbone, a combined process of atomic oxygen and ultraviolet may contribute as the third reaction pathway. Once atomic oxygen reacts to the polyethylene backbone, a ketone group is formed. Ketone is known as a photon-adsorbing site and Norrish type 1 or type 2 photochemical reactions scissor the backbone of the polyethylene [13]. Because of the backbone scission, the ketone group would form carbonyl or carboxyl surface functional groups. They are removed from the backbone by a photoinduced reaction with forming volatile CO and CO_2. The third reaction pathway may contribute to the enhancement of the erosion in the exposure condition when both atomic oxygen and ultraviolet exist, i.e., incident angles between 20 and 60 deg in Fig. 6b which shows the largest erosion rate in all exposure conditions tested.

IV. Conclusions

Synergistic effects on atomic oxygen-induced erosion of polyimide and polyethylene with 172 nm ultraviolet were investigated using QCM. To change the relative intensity of atomic oxygen and ultraviolet, and the polymer sample on QCM was rotated with an axis perpendicular both to the axes of atomic oxygen and ultraviolet. It was found that no ultraviolet-induced erosion was observed for polyimide without atomic oxygen exposure. In contrast, a clear mass loss was obvious when polyethylene was exposed to 172 nm ultraviolet even without atomic oxygen. It was observed that the atomic oxygen-induced erosion of polyethylene increased 30–80% by a simultaneous ultraviolet exposure in the UV/AO ratio of 10^{-15} mJ/atom. The increase in mass loss was considered due to the photoinduced decomposition of the carboxyl group and C–C backbone of polyethylene. Because the synergistic effect for polyethylene was obvious at the ultraviolet intensities one order lower than that for polyimide, it was concluded that polyimide is a better material as a witness sample for measuring atomic oxygen fluence in LEO from the viewpoint of the synergistic effect with vacuum ultraviolet.

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