Synergistic Study on Atomic Oxygen-Induced Erosion of Polyethylene with Vacuum Ultraviolet

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Ground-based experimental results on atomic oxygen-induced erosion of polyethylene with simultaneous vacuum ultraviolet (VUV) are reported. In situ mass-loss measurement was made using a quartz crystal microbalance with a mass resolution of 2 ng. A laser detonation atomic oxygen beam source was used to simulate collision energy of atomic oxygen with materials in low Earth orbit. To change relative intensity of atomic oxygen and VUV, the sample was rotated with an axis perpendicular both to the axes of atomic oxygen and VUV. It was observed that the erosion rate of polyethylene was increased 30–100% by a simultaneous VUV exposure depending on the incident angle of atomic oxygen. No contribution of temperature increase was confirmed in the polyethylene erosion. The experimental results suggested that two reaction pathways exist in the simultaneous atomic oxygen and VUV exposure conditions. It is suggested that the synergistic effect on polyethylene erosion is emphasized in the exposure condition where relative atomic oxygen flux is low against VUV flux.

Nomenclature

Ea = activation energy of gasification reaction, eV

T = sample temperature, K

 Δf = frequency shift of quartz crystal microbalance, Hz

= incident angle of atomic oxygen with respect to

surface normal, deg

 $90-\theta$ = incident angle of vacuum ultraviolet with respect to surface normal, deg

Introduction

T has been recognized that atomic oxygen is one of the most important factors that influences the erosion of polymeric materials in a low Earth orbit (LEO) space environment. Although a number of polymeric materials are utilized in space systems, polyimide is one of the most widely used polymeric materials in spacecraft applications. The erosion rate of polyimide film as a result of atomic oxygen attack in low Earth orbit has been established to be 3.00×10^{-24} cm³/atom, and, hence, polyimide film has been used as one of the reference materials to evaluate the atomic oxygen fluence. Polyethylene also is a candidate as a reference material because of its simple structure. For reference materials, erosion properties of polyethylene at various exposure conditions need to be well understood. However, the existing basic knowledge of the erosion of polyethylene under atomic oxygen exposure is not sufficient for predicting the erosion depth under various exposure conditions.

One of the major factors that influence the atomic oxygen-induced erosion rate of polymer film is a simultaneous ultraviolet (UV) exposure. Some preliminary studies are conducted to clarify the synergistic effect during a space flight or in a laboratory. However, both studies have difficulties; passive space flight experiments cannot control experimental details; and, in contrast, laboratory tests cannot simulate the collision energy of atomic oxygen with materials. It has been reported that synergistic effect was not observed in the

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EOIM-3 flight experiment.² On the other hand, 30% of increase in erosion rate by UV exposure was reported in a ground-based thermal atomic oxygen system.³ This discrepancy probably came from the differences in experimental condition such as 1) collision energy of atomic oxygen, 2) sample temperature, 3) relative intensity of UV and atomic oxygen, and so on. To clarify the detail of synergistic effect on atomic oxygen-induced material erosion under UV exposure, contribution of each factor should be distinguished. However, it is not easy to distinguish these factors in an atomic oxygen simulation experiment. For example, a thermal atom system cannot simulate collision energy of atomic oxygen; a conventional UV source heats the sample; or acceleration factors of atomic oxygen and UV fluxes are not easy to adjust. A combination of hyperthermal atomic oxygen beam and monochromatic UV sources might be beneficial to distinguish these factors.

In this paper, we are reporting on the ground-based experimental results on synergistic effect of atomic oxygen and vacuumultraviolet (VUV) exposure on polyethylene. A laser detonation hyperthermal atomic oxygen source and an excimer light source with a wavelength of 172 nm were used to distinguish the preceding experimental factors. Contribution of a possible temperature increase during VUV exposure and the effect of incident angle of atomic oxygen and VUV to control their fluxes are also studied.

Experiments

The laser detonation atomic oxygen beam source, which was originally invented by Physical Sciences, Inc., was used in this study (Fig. 1; Ref. 4). Details of the experimental apparatus are reported elsewhere.⁵ The translational energy of the atomic oxygen beam used in this study was approximately 5.1 eV, whereas the beam flux at the sample position was measured at 6.5×10^{14} atoms/cm²/s by using a silver-coated quartz crystal microbalance (QCM).⁶ A polyethylene film was prepared by a spin coating on QCM sensor crystals. A solution that contains 0.3 g of low-density polyethylene (average molecular weight of 6500) in 40 ml of solvent was utilized for the formation of thin polyethylene film using the spin-coating technique. The erosion rate of polyethylene film was calculated from the change in the resonant frequency of the QCM during the atomic oxygen beam and/or VUV exposures. The frequency of QCM was measured every 10 s with a frequency resolution of 0.1 Hz, which corresponds to a mass resolution of 2 ng. The temperature of the film was controlled with an accuracy of 0.1 K by the temperaturecontrolled recirculating water system.

The incident angle dependence was measured at the sample temperature of 311 K, whereas the temperature dependence was observed in the sample temperature range between 253 and 353 K

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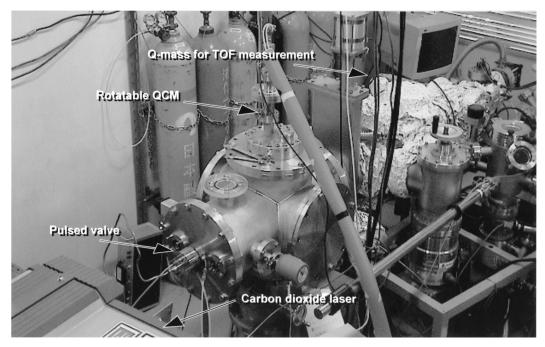


Fig. 1 Photograph of the laser detonation atomic oxygen source used in this study.

with the incident angle of 0 deg (normal incidence). The incident angle of atomic oxygen was changed by rotating QCM. A VUV source used in this experiment was an excimer light source with a wavelength of 172 nm. Axes of atomic oxygen beam and VUV were crossed 90 deg, and the sample was rotatable with the axis perpendicular both to atomic oxygen and VUV in order to change the relative intensities of atomic oxygen and VUV. VUV light was also generated at the high-temperature plasma in atomic oxygen source. This VUV was not filtered out such that atomic oxygen-induced erosion in this study includes the effect of VUV from the atomic oxygen source. Thus, the effect of additional VUV from the 172-nm VUV source was distinguished and analyzed in this study. Before the measurements, the polyethylene film was exposed to atomic oxygen (6×10^{17} atoms/cm²) to saturate the surface oxygen content of the sample. This is what occurred in order to avoid the effect of nonlinear mass-loss phenomenon observed at the beginning of the atomic oxygen exposure of pristine polymer surfaces.⁷

Results and Discussion

Effect of Sample Temperature

Ultraviolet exposures to the polymeric materials often increase sample temperature and can influence the erosion of polymers. In a synergistic testing on atomic oxygen and VUV, an effect of sample temperature, which is affected by VUV exposures, should be distinguished with that of photochemical effect. To evaluate the effect of temperature increase in the synergistic testing of atomic oxygen and VUV on polymers, temperature dependence of atomic oxygeninduced erosion of polyethylene was investigated without VUV exposure. Figure 2 shows the frequency shift of the polyethylenecoated QCM during the 5.1-eV atomic oxygen beam exposure at sample temperatures from 253 to 353 K. As clearly indicated, a linear relationship between exposure time and frequency shift, which corresponds to mass loss, was observed in every temperature tested. The slope of each data plot was calculated by the least-square method. Because the frequency shift of QCM Δf , which corresponds to mass loss of the film, is expressed in the Arrhenius-type function, the relationship between 1/T and Δf was plotted. The results are shown in Fig. 3. From the slope of the Arrhenius plots in Fig. 3, the activation energy of mass loss reaction Ea was calculated to be 6.1×10^{-3} eV for the 5.1-eV atomic oxygen beam. The same experiment was also carried out with a polyimide-coated QCM, and the activation energy of 5.7×10^{-4} eV

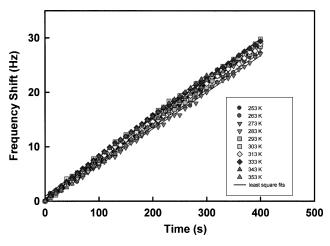


Fig. 2 Resonance frequency shifts of polyethylene-coated QCM under 5.1-eV atomic oxygen beam exposure at sample temperatures from 253 to 353 K.

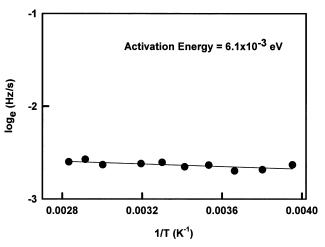


Fig. 3 Arrhenius plot of the erosion rates of polyethylene film with 5.1-eV atomic oxygen beam.

was measured. 8 It was thus concluded that the activation energies of gasification reactions of hydrocarbons with a 5-eV atomic oxygen in LEO are in the order of 10^{-3} – 10^{-4} eV. These small activation energies are responsible for the temperature-independent erosion property of polymeric materials flown on space shuttle missions.⁹ In contrast, some ground-based experiments using thermal atom system reported relatively large activation energies ranging 0.13-0.29 eV (Refs. 10-12). The inconsistency of the activation energies between space and ground-based experiments was considered to be caused by the translational energy of impinging atoms. To clarify the origin of the discrepancy, the activation energy of gasification reaction of polyimide with a 1.1-eV atomic oxygen beam was measured. As a result, the activation energy of 4.7×10^{-2} eV was obtained for polyimide.⁸ This is an activation energy two orders greater than that with a 5.1-eV atomic oxygen beam. The activation energy of 4.7×10^{-2} eV under the 1.1-eV atomic oxygen beam exposure was consistent with the experimental results using 1.5-eV atomic oxygen beam reported by Cross et al.13 Therefore, the gap reported between flight and ground-based results was bridged in this

From the experimental results on the temperature dependence of atomic oxygen-induced erosion reported in this section, it was confirmed that the sample temperature hardly influences the erosion rate of polyethylene in the range between 253–353 K in the hyperthermal atomic oxygen exposures. The maximum surface temperature during VUV exposure in this study is estimated to be 340 K even without the active temperature control. Thus, it is concluded that the temperature increase during VUV exposures did not affect the atomic oxygen-induced erosion results observed in this study.

Atomic Oxygen Flux Control

For a quantitative study on atomic oxygen-induced erosion of polymers, control of atomic oxygen flux is important. It is, however, not easy for controlling atomic oxygen flux in a ground-based facility. In this study we attempted to control the effective atomic oxygen flux at the sample surface by tilting the sample with respect to the atomic oxygen beam axis. Figure 4 shows the frequency shift of the polyethylene-coated QCM during the atomic oxygen beam exposures at the incident angle of atomic oxygen from 0 to 90 deg. The incident angle was taken with respect to the surface normal. A good linear relationship between the frequency shift and the exposure time, that is, mass loss and atomic oxygen fluence, was observed at all incident angles. The slope of the mass loss rate at every incident angle was calculated by the least-square fit and plotted against the incident angle. The results are presented in Fig. 5. It is clear that the rate of the frequency shift, or erosion rate, of polyethylene depends on the incident angle, and it follows a $\cos^{0.87}\theta$ dependence as indicated by the solid line in Fig. 5. The data points at the incident angle of 80 deg in Fig. 5 are affected by the QCM holder, which

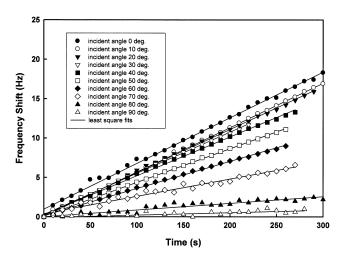


Fig. 4 Resonant frequency shift of polyethylene-coated QCM under the 5.1-eV atomic oxygen exposures at incident angles from 0 to 90 deg.

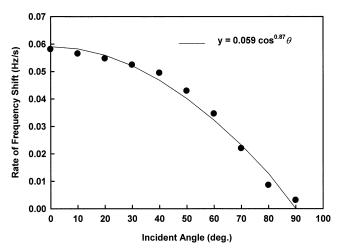


Fig. 5 Rate of frequency shift of the polyethylene-coated QCM as a function of incident angle of atomic oxygen.

blocks a part of the incoming atomic oxygen beam. The $\cos^{0.87}\theta$ dependence is close to the $\cos^{1.0}\theta$ dependence, which was observed in the case of polyimide.¹⁴

In space, Banks and coworkers reported that the incident angle dependence of the erosion of fluorinated ethylene propylene (FEP) Teflon® in the Long Duration Exposure Facility flight experiment followed the $\cos^{1.5}\theta$ law rather than a cosine law. ¹⁵ An analysis of the flight data of Kapton-H and Mylar aboard STS-8 concluded that the incident angle dependence also followed the $\cos^{1.5}\theta$ law. ¹⁶ However, their conclusions were based on a small number of data points obtained from flight experiments, which incorporated a wide range of error spoiling the accuracy of the analysis. Furthermore, no physical explanation was provided for the $\cos^{1.5}\theta$ dependence.

The cosine law of the incident angle dependence observed in this study was physically explained by the presence of microscale roughness and the multiple bounce effect at the polymer surface as described elsewhere.14 Thus, the macroscopic erosion phenomena of polyimide simply reflects the effective fluence of atomic oxygen, which follows the cosine law with the macroscopic incident angle. The tilting of the sample reduces not only the effective flux of atomic oxygen but also the effective impingement energy. Lowering the incident angle from 0 to 60 deg leads to the reduction of effective impingement energy from 5.0 to 1.25 eV; it is proportional to the cosine square of the incident angle. The activation energies of gasification reaction of polymers in this impinging energy range are expected to be as low as 10^{-3} eV as described in the preceding section. It was thus concluded that the effect of activation energies in the incident angle dependence of erosion rates could be negligibly small. From the experimental results shown here, it is concluded that the flux control of atomic oxygen is possible by tilting the sample with respect to the beam axis.

Synergistic Effect with VUV Exposure

A quantitative analysis on the synergistic effect of atomic oxygen and VUV was investigated by the crossed atom and VUV beam configuration shown in Fig. 6. A 172-nm VUV source was installed perpendicular to the atomic oxygen beam line, and the sample was rotatable with the rotating axis perpendicular both to the atomic oxygen and VUV beam axes. In this configuration, the effective atomic oxygen and VUV fluxes can be changed by rotating the sample as indicated in the preceding section. The maximum atomic oxygen flux and VUV flux at the sample position were $6.5 \times$ 10¹⁴ atoms/cm²/s and 0.55 mW/cm², respectively. Figure 7 shows the rate of frequency shift of polyethylene-coated QCM as a function of incident angle of atomic oxygen under simultaneous atomic oxygen and VUV exposures. Note that the incident angle of atomic oxygen is represented by θ , whereas that of VUV is 90– θ . The angular dependence of atomic oxygen-induced erosion without VUV exposure follows the $\cos^{0.87}\theta$ dependence as described earlier. In

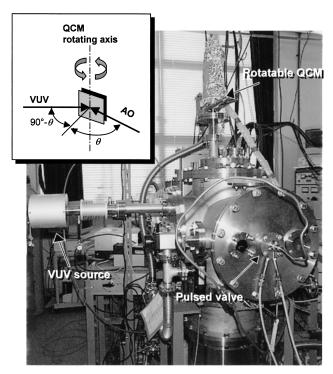


Fig. 6 Experimental setup for the synergistic effect experiment on the atomic oxygen and VUV exposures.

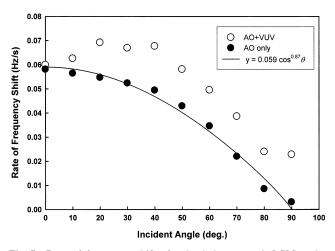


Fig. 7 Rate of frequency shift of polyethylene-coated QCM under atomic oxygen beam exposure with (\bigcirc) and without (\bullet) simultaneous VUV exposure.

contrast, it was observed that the rate of frequency shifts of polyethylene film under atomic oxygen and VUV exposure (open circle) increased with incident angle and then started decreasing. At the incident angles smaller than 10 deg, VUV was partially blocked by the QCM holder, and the VUV effect on erosion rate was not clear. In contrast, for incident angles between 20 and 70 deg the erosion rates with VUV exposure are 30 to 100% greater than those without VUV exposure at all incident angles, which followed $\cos^{0.69}\theta$ dependence. This finding indicated that the mass-loss reaction during the simultaneous exposures of atomic oxygen and VUV was basically rate limited by the atomic oxygen flux.

The erosion rates of polyethylene by atomic oxygen beam exposure (solid circle in Fig. 7) were subtracted from those in simultaneous exposures of atomic oxygen and VUV (open circle). The results are shown in Fig. 8. As shown in Fig. 8, the residual is independent of the incident angle at the incident angles greater than 20 deg, where VUV is fully exposed to the sample surface. The open circles in Fig. 8 indicate the erosion rates of polyethylene only by VUV

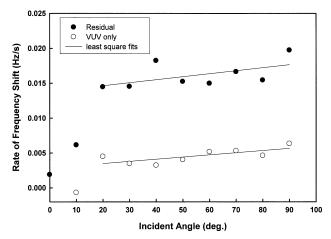


Fig. 8 Rate of frequency shift of polyethylene-coated QCM under VUV exposure (\bigcirc) and the contribution of VUV in the synergistic effect (\bullet) . Note that the abscissa represents the incident angle of atomic oxygen.

and not by atomic oxygen exposure. Again, the erosion rates are constant with the incident angle of VUV. A similar result was also observed with polyimide films on a QCM sensor crystal. From these experimental findings it is considered that the VUV flux geometrically follows cosine distribution when the sample is rotated, but the effect of VUV is not altered by its incident angle. These results are caused probably by the penetration depth of VUV is greater than the film thickness and VUV could reach the polymer/gold interface and be scattered. The surface and interfacial roughness of polyethylene can play a critical role to cancel the angular dependence of VUV. Besides the independency of the incident angles, it is obvious that the two data plots in Fig. 8 are not consistent, that is, the residuals are three times greater than the erosion by VUV only. Even though the structure of low-density polyethylene does not absorb the VUV with 172 nm, it is identified as the presence of atomic oxygen-enhanced VUV-induced erosion of polyethylene. Oxidized radicals such as carbonyl group formed by the atomic oxygen-induced oxidation of polyethylene can have a critical role on synergistic effect of atomic oxygen and VUV at 172 nm.

From the experimental results shown in Figs. 7 and 8, it is suggested that two independent erosion pathways exist in a simultaneous atomic oxygen and VUV exposure condition; atomic oxygeninduced erosion that follows $\cos^{0.87}\theta$ dependence with incident angle and VUV-induced erosion that is independent of incident angle of VUV. The erosion rate of VUV-induced erosion pathway is accelerated three times under the presence of atomic oxygen in the experimental condition. Because the absolute reaction rate is greater in atomic oxygen-induced erosion, overall erosion was observed to be rate limited by atomic oxygen. Thus, the synergistic effect was not obvious in a high-flux atomic oxygen condition such as the flight experiment onboard EOIM-3 (Ref. 2). However, the effect of VUV-induced erosion becomes to be emphasized in a low-flux atomic oxygen environment in LEO, that is, higher altitudes. Thus, a synergistic testing on material erosion onboard the space station (altitude: 480 km) might give different results from that onboard the shuttle transportation system (altitude: 200 km).

Conclusions

The synergistic effect on atomic oxygen-induced erosion of polyethylene with 172-nm VUV was investigated. To change the relative intensity of atomic oxygen and VUV, the sample was rotated with an axis perpendicular to the axes of both atomic oxygen and VUV. It was observed that the erosion rate of polyethylene increased 30–100% by a simultaneous VUV exposure depending on the relative intensity of VUV and atomic oxygen. The synergistic effect of atomic oxygen-induced erosion with VUV was three times larger than that by VUV only. These erosion properties suggested that two independent erosion pathways exist in a simultaneous atomic

oxygen and VUV exposure condition: the atomic oxygen-induced erosion and the VUV-induced erosion, which is accelerated three times under the presence of atomic oxygen. Contribution by the temperature increase of a specimen as a result of a VUV exposure was confirmed to be negligible in a hyperthermal atomic oxygen exposure.

Acknowledgments

A part of the study was supported by the Grant-in-Aid for scientific research from the Ministry of Education, Sports, Culture, Science and Technology, Japan, Contracts 13750842 and 14350511. The authors acknowledge T. Kida and S. Seikyu of Kobe University for their help with the experiments.

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