ATOMIC OXYGEN-INDUCED EROSION OF POLYMERIC MATERIALS UNDER SURFACE CHARGING CONDITION

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Abstract

Polyimide is one of the most widely used polymers as spacecraft materials. Polyimide has also been used as a reference material to measure atomic oxygen fluence in LEO. Erosion properties of polyimide by hyperthermal impact of atomic oxygen under various synergistic exposure conditions need to be well understood for an accurate fluence measurement. In this study, the effect of surface charging on the atomic oxygen-induced erosion of polyimide was studied. The erosion rates of polyimide were not altered by bias voltages up to $\pm 1000 \text{ V}$ under 4.8 eV atomic oxygen exposure condition. In contract, 20 % increase in erosion rate was observed by 1.1 eV atomic oxygen beam exposure. It was confirmed that the effect of surface charging on the erosion of polyimide depends on the translational energy of impinging atomic oxygen.

1. Introduction

Low Earth Orbit (LEO) is a complicated environmental field where the various space environmental factors interact with materials such as microgravity, thermal cycling, plasma environment (ions and electrons), ultraviolet, radiation, high-energy charged particles, space debris and neutral gases. Space environmental effect seems to become more and more important for long-term low-risk missions. One of the difficulties in studying space environmental effect is its synergistic effect. For example, a synergistic effect of 172 nm vacuum ultraviolet on atomic oxygen-interaction with polyimide enhances erosion rate more than 300% depending on the ultraviolet intensity even though ultraviolet itself does not give any mass-loss on polyimide [1]. It was important to recognize that surface reaction, including gasification reaction by hyperthermal atomic oxygen impact in LEO, is sensitive to the surface properties which is influenced by the other environmental factors beside atomic oxygen.

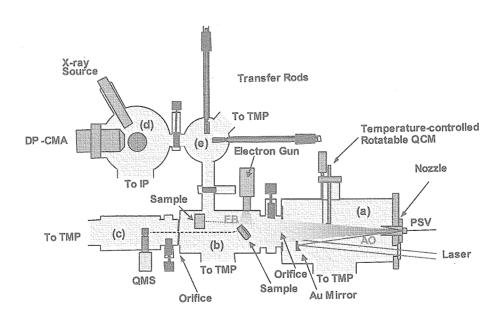


Figure 1. A schematic drawing of the space environmental simulation facility using a laser detonation atomic oxygen beam source. (a): Atomic oxygen source chamber, (b): reaction chamber, (c): Time of Flight (TOF) chamber, (d): XPS chamber, and (e): preparation chamber. Electron gun was attached to the reaction chamber.

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Insulating surfaces of spacecraft are sometimes charged up to several kV by plasma environment and photoemission by solar ultraviolet in LEO. Spacecraft charging/discharging has been recognized as a serious problem on electronic systems aboard spacecraft. It sometimes causes seriously damages to spacecraft system due to discharge. Not only such an acute event, but spacecraft charging may influence various effects on spacecraft materials, i.e., surface charging may affect the atomic oxygen-induced erosion of materials. The only literature reported was the effect of electron beam irradiation to the atomic oxygen erosion of polysulfone [2]. It was found that electron beam irradiation or bias voltages applied to the back-plate of polysulfone increased the signal of reactive products (CO and CO₂) from the target, which suggested the increase in mass-loss of polysulfone. Since polyimide has been used as a standard material as a witness sample for material exposure tests in LEO, it is necessity to clarify the effect of surface charging to the erosion property of polyimide under atomic oxygen exposure.

In this study, we examined the effect of surface charging on the atomic oxygen-induced erosion of polyimide. A laser detonation atomic oxygen beam source at Kobe University, which delivers 5 eV atomic oxygen beam was used for simulating high-energy collision of atomic oxygen in LEO space environment. A quartz crystal microbalance (QCM) technique, which was established to study synergistic effect of atomic oxygen and ultraviolet on polymer erosion [3], was applied to measure the erosion rate of polyimide under surface-charging conditions. Effect of bias voltage on the atomic oxygen-induced erosion of polyimide and of surface properties were analyzed and discussed.

2. Experiments

The space environment simulation facility at Kobe University was used in this study. The laser detonation atomic oxygen beam source, which was originally invented by Physical Sciences Incorporation (PSI), was equipped in this facility. The electron gun for Reflection High Energy Electron Diffraction (RHEED) was attached to this apparatus as an electron beam source. The schematic drawing of the facility is shown in Figure 1. Details of the experimental apparatus are reported elsewhere [4]. Translational energy of the atomic oxygen beam used in this study was approximately 4.8 eV, while the beam flux at the sample position was measured to be 2.4 x 10¹⁴ atoms/cm²/s by using a silver-coated QCM. The acceleration voltage of electron beam was 7 keV and filament current was 2.2 A. Axes of the atomic oxygen beam and the electron beam was crossed 90 degrees and the sample was set at the cross point of atom and electron beams in order to expose simultaneously (see Figure. 1). The polyimide film used in this study was the pyromelliticdianhydrid-oxydianiline (PMDA-ODA) type polyimide supplied by Toray Industries Inc. (Semicofine SP-510). The polyimide film was spin-coated on a QCM sensor crystal, and annealed at 150 °C for 1hour and at 300 °C for 1hour. Details of the sample preparation are reported in the reference 5. The erosion rate of the polyimide film was calculated from the change in the resonant frequency of the QCM during the electron beam and atomic oxygen beam exposure. The sample temperature was kept 38 °C during the experiment by the temperature-controlled recalculating water system.

3 Result and Discussion

3.1 Effect of electron beam exposure

It is necessary to confirm whether or not the electron beam exposure alone affects the erosion rate of polyimide before the simultaneous exposure test of atomic oxygen and electron beam. A 7 keV electron beam was exposed to the sample. Two measurements were carried out under different conditions; one was in the case that the sample holder was grounded, and the other was floated. The results are shown in Figure 2. The change in frequency of polyimide-coated QCM, i.e., mass loss, was not observed under both experimental conditions. This experimental result clearly indicates that 7 keV electron beam does not affect the erosion rate of polyimide.

3.2 Synergistic effects of atomic oxygen and electron beam

The effect of electron beam exposure on the atomic oxygen-induced erosion rate was examined. The experimental results are shown in Figure 3. The slopes of the erosion rates were calculated by the least-square fit. When electron beam was exposed during atomic oxygen exposure, the mass loss rate (white circle and triangle)

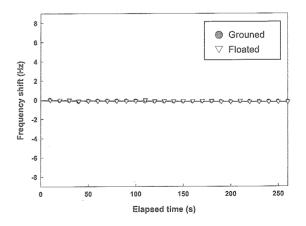


Figure 2. Change in frequency of polyimide-QCM during electron beam exposure. Electron energy: 7 keV, Filament current: 2.2 A.

slightly decreased (4 %) compared to that of atomic oxygen exposure alone (black circle). This suggests that the 7 keV electron beam would decrease the atomic oxygen-induced erosion rate of polyimide. experimental result is consistent with that reported by Dimov which 4 keV low energy electron beam enhanced stabilization of C-O bond [6]. It is assumed that unstable C-O bond created by the atomic oxygen bombardment become stabilized by the electron beam exposure and the erosion of polyimide is prevented. However, the contrary effect of electron beam irradiation in the atomic oxygen erosion of polysulfone was reported by King and Wilson [2]. They found that electron beam irradiation to polysulfone increased the signal of reactive products from the target (CO and CO₂). experimental result suggested that the mass-loss rate of polysulfone is increased by the simultaneous electronbeam exposure. This discrepancy may be due to the difference in material response against electron beam, so that the effect of bias voltages to polyimide was investigated.

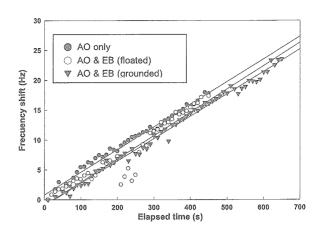


Figure 3. Frequency shift of the polyimide-QCM under simultaneous exposure of atomic oxygen and electron beams. Atomic oxygen energy: 4.8 eV, flux: 2.4 x 10¹⁴ atoms/cm²/s, Electron beam energy: 7 keV.

3.3 Effect of surface charging on the erosion

The effect of surface charging on the atomic oxygen-induced polyimide was investigated by application of bias voltage. The bias voltage of ± 1000 V was applied to the sample holder during atomic oxygen exposure. Two atomic oxygen beams were used in this experiment in order to investigate the effect of translational energy in the erosion; 4.8 eV and 1.1 eV. Flux of atomic oxygen beam was measured to be 13 x 10^{14} atom/cm²/s for the 4.8 eV beam, whereas 8 x 10^{14} atom/cm²/s for the 1.1 eV beam. Figure 4 shows the frequency shift of the polyimide-coated QCM during 4.8 eV atomic oxygen beam exposures. It was observed that the erosion rate by 4.8 eV atomic oxygen beam was not greatly influenced by the bias voltages of 1000V (both positive and negative). In contrary, the erosion rates of polyimide increased 20 % by sample charging (both ± 1000 V and ± 1000 V) in the case of 1.1 eV atomic beam oxygen exposures as shown in Figure 5. This finding suggested that the reaction yield of atomic oxygen with polyimide depends on the translational energy of atomic oxygen beam.

3.4 Sample surface condition by surface charging

The change in surface chemical condition by surface charging was examined using Fourier Transform Infrared Spectrometer (FT-IR). The ±1000 V bias voltage were applied to the aluminum backplate at the sample stage in FT-IR. Reflection FT-IR measurements were carried out during the application of bias voltages. The

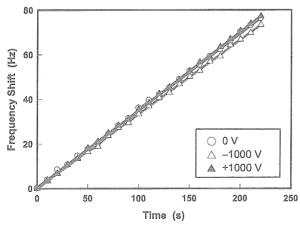


Figure 4. The atomic oxygen-induced erosion rate of polyimide with/without bias voltages. Translational energy of atomic oxygen: 4.8 eV, Flux: 13×10^{14} atoms/cm²/s, Bias voltage: ± 1000 V.

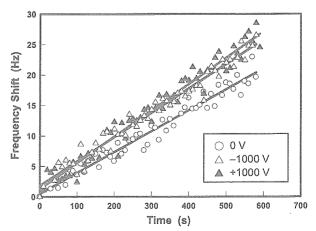


Figure 5. The atomic oxygen-induced erosion rate of polyimide with/without bias voltages. Translational energy of atomic oxygen: 1.1 eV, Flux: 8 x 10¹⁴ atoms/cm²/s, Bias voltage: ±1000 V.

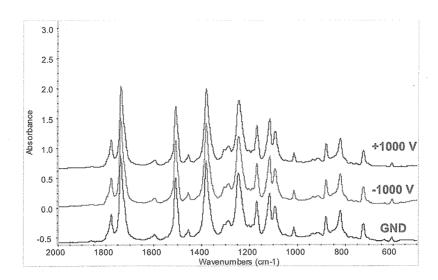


Figure 6. FT-IR spectra of polyimide film under bias voltages.

results were shown in Figure 6. It was confirmed that the FT-IR spectra of polyimide under the presence of bias voltages to $\pm 1000 \text{ V}$ does not influence the FT-IR spectra, which suggests the molecular chain of polyimide was not affected by the bias voltages.

The contact angle of the polyimide film surface was also measured in order to investigate the change of surface condition by the presence of bias voltages. The sample used in this experiment was pristine polyimide film without atomic oxygen exposure. To avoid any danger during the measurement, the measurements were carried out immediately after turn-off the applied voltages. Without bias voltages, the contact angle of polyimide was 77.2°. This value is a typical advancing contact angle of water on a polyimide. However, the advancing contact angle decreased to 60.9° shortly after -1000 V was applied. It even decreased to 57.4° when +1000 V was applied. The contact angle gradually recovered to the primitive condition after turning-off the bias voltages. This result indicates that the surface free energy of polyimide film became high with bias voltages. The change in surface free energy of polyimide is caused by the rotation and bending of the molecular chain of polyimide. The relationship between erosion rate by atomic oxygen and the surface tension, i.e., surface free energy, of the polymeric materials has been reported by Kleiman [7]. It was reported that the erosion yield of polymers becomes greater when the surface free energy is high. Change in surface free energy in polyimide due to surface charging leads to increase the atomic oxygen-induced erosion rate.

4. Conclusion

The effect of electron beam exposure and surface charging on atomic oxygen-induced polyimide was investigated in the ground-based experiment. A 7 keV electron beam exposure alone does not affect the mass loss of polyimide. However the simultaneous exposure of atomic oxygen beam and 7 keV electron beam slightly decreased the erosion rate of polyimide. This may be due to the stabilization of C-O bond by low energy electron beam exposure. The effect of surface charging on the erosion rate of polyimide was clearly observed when 1.1 eV atomic oxygen was exposed. The erosion rate increased 20 % compared by biasing at 1000V (positive and negative). In LEO, atomic oxygen lost its translational energy after reflecting at the spacecraft surface, and may attack to the polymeric materials in lower energies. In such low translational energy collisions, polyimide erosion by atomic oxygen might be affected by surface charging condition.

Acknowledgements

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