# Temperature Dependence in Erosion Rates of Polyimide Under Hyperthermal Atomic Oxygen Exposures

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## Introduction

IT has been recognized that polyimide film, which is widely used in space applications, is eroded by atomic oxygen in a low Earth orbit (LEO) space environment. Because polyimide has been used as a monitoring material to evaluate atomic oxygen fluence, details of atomic oxygen reaction with polyimide should be made clear under various atomic oxygen exposure conditions.[1] One of the important factors that would influence the reactivity of atomic oxygen is a sample temperature. However, there exists no ground-based report regarding an accurate measurement on the temperature dependence of polyimide erosion under the exposure to atomic oxygen beam with a translational energy of 5 eV, which corresponds to the collisional energy of atomic oxygen and materials in LEO.

In this Note we are reporting the in situ results of temperature dependence in polyimide erosion under 5-eV atomic oxygen exposures. The effect of translational energy of the atomic oxygen beam on the temperature dependence of polyimide erosion was also examined in the ground-based experiments.

## Experiments

The laser detonation atomic oxygen source was used in this experiment.[2] The translational energy of the atomic oxygen beam was monitored by the time-of-flight measurement system. The polyimide film used in this study was a pyromellitic-dianhydride-oxydianiline polyimide supplied by Toray Industries, Inc. (Semicofine SP-510). A polyimide film with 0.1-$\mu$m thickness was prepared by spin-coating on a sensor crystal of quartz crystal microbalance (QCM). Details of the sample preparation are reported in Ref. 3. It was confirmed by x-ray photoelectron spectroscopy that the surface structure of the spin-coated polyimide film was similar to that of Kapton-H film. The mass of the polyimide film was measured every 10 s from the shift in the resonance frequency of the QCM during the atomic oxygen exposures. The sample temperature was controlled in the range of 253–353 K with an accuracy of 0.1 K by the temperature-controlled recirculating water system. Prior to the measurement, the polyimide film was exposed to atomic oxygen until the adsorbed oxygen was saturated. This is in order to avoid the nonlinear effect of mass loss, which appeared at the beginning of the atomic oxygen exposures at pristine polyimide surfaces.[4]

## Results and Discussion

Figure 1 shows the translational energy distributions of atomic oxygen beams used in the experiments. Two atomic oxygen beams were used in this experiment. As well as the atomic oxygen beam with mean translational energy of 5.0 eV, the atomic oxygen beam with translational energy of 1.1 eV was used in order to investigate the effect of translational energy in the erosion. The flux of atomic oxygen was measured from the mass gain of Ag film with a reaction probability of 0.62. This reaction probability of atomic oxygen with Ag was determined as a compensation factor through a comparison between the mass gain of Ag film and the Kapton equivalent fluence that was calculated with the established erosion yield of Kapton-H film ($3.00 \times 10^{-24}$ cm$^3$/atom). The flux of the beam was calculated to be $2.6 \times 10^{14}$ atom/cm$^2 \cdot$ s for the atomic oxygen beam with translational energy of 5.0 eV, whereas it was $2.1 \times 10^{15}$ atom/cm$^2 \cdot$ s for the 1.1-eV beam. Figure 2 shows the frequency shift of the polyimide-coated QCM during the atomic oxygen exposures at the sample temperatures $T$ from 253 to 353 K. Linear relationships between the exposure time and the frequency shift (mass loss) were clearly observed at all sample temperatures measured. The slope of the erosion rates was calculated by the least-squares fit. It was observed that the erosion rate by the 5-eV atomic oxygen beam was greater than those by the 1.1-eV beam even though atomic oxygen flux was smaller. This finding suggested that the erosion yield of atomic oxygen with polyimide depends strongly on the translational energy of the beam. Because the erosion rate $Re$ was considered to be expressed in the Arrhenius-type function, the relationship between $1/T$ and $Re$ was plotted (Arrhenius plot). The results are shown in Fig. 3. From the slope of the Arrhenius plots, the activation energies of the mass loss reaction $Ea$ were calculated to be $5.7 \times 10^{-4}$ eV for the 5.0-eV atomic oxygen beam and $4.5 \times 10^{-2}$ eV for the 1.1-eV atomic oxygen beam. Both of the activation energies are small; however, the activation energy with the

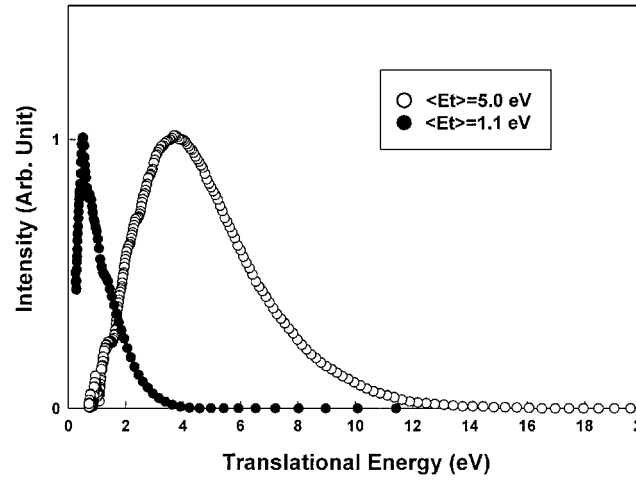


**Fig. 1 Translational energy distributions of the atomic oxygen beam used in this study. Mean translational energies of the atomic oxygen beams are 5.0 and 1.1 eV.**

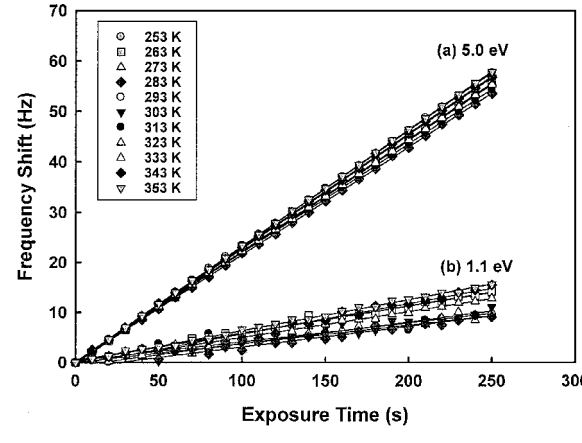


**Fig. 2 Resonance frequency shifts of the polyimide spin-coated QCM during the atomic oxygen beam exposures at sample temperatures from 253 to 353 K. Mean translational energies of the atomic oxygen beam used are 5.0 and 1.1 eV.**

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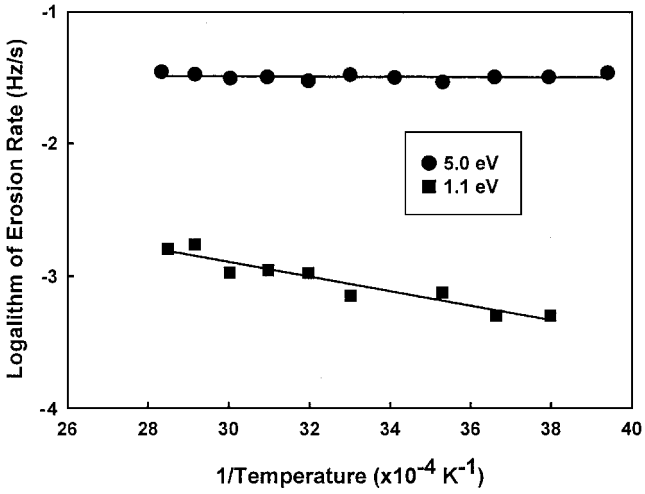


**Fig. 3 Arrhenius plots of the erosion rates during the atomic oxygen beam exposures with translational energies of 5.0 and 1.1 eV.**

5.0-eV atomic oxygen beam was two orders smaller than that with the 1.1-eV beam.

In the flight experiments aboard STS-8, polyimide films were exposed to the orbital atomic oxygen environment at 338 and 394 K. As a result, no temperature effect on the erosion depth was observed, and activation energy of the erosion was reported to be zero.[5] In contrast, ground-based studies using thermal atom systems gave the activation energies ranging from 0.13 to 0.29 eV (Refs. 6–8). In the hyperthermal atom system (translational energy of 1.5 eV) the activation energy of 0.04 eV was reported.[9] The activation energies with the 5.0- and 1.1-eV atomic oxygen beams measured in this study correspond to those observed in the flight experiments and the ground-based experiments (hyperthermal case) reported earlier. Obviously, the results of this ground-based experiment reproduced the activation energies measured both in space and in laboratories. It is thus concluded that the presence of translational energy effect on the temperature dependence in polyimide erosion, which has only been speculated on in previous reports, was experimentally verified in this study.

## Conclusions

The temperature dependence of the erosion rates of polyimide film in the hyperthermal atomic oxygen exposures was examined. It was observed that the activation energies of the polyimide erosion was $5.7 \times 10^{-4}$ eV for the 5.0-eV atomic oxygen beam, whereas it was $4.5 \times 10^{-2}$ eV for the 1.1-eV atomic oxygen beam at the temperature range between 253 and 353 K. Presence of the translational energy effects on the erosion yield and the temperature dependence of polyimide erosion were experimentally verified.

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