Randy Lin, Glenn Macala, and David Skulsky, their colleagues at JPL, for many helpful discussions. The second author would also like to thank David Miller of the Massachusetts Institute of Technology Space Systems Laboratory for all of his support. Also, we would like to thank the reviewers and the Associate Editor of the *Journal of Spacecraft and Rockets* for their review of an earlier version of this Note.

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Impingement Angle Dependence of Erosion Rate of Polyimide in Atomic Oxygen Exposures

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Introduction

IT has been recognized that atomic oxygen is one of the most important factors that influences the erosion of many space materials, especially polymeric materials, in low Earth orbit. 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 due to atomic oxygen attack in low Earth orbit was reported to be 3.00×10^{-24} cm^3/atom, and, hence, polyimide film has been used as one of the reference materials to evaluate the erosion rate of other materials.[1] For the reference material, erosion rates at various exposure conditions need to be well understood. However, the existing basic knowledge on the erosion of polyimide due to atomic oxygen is not extensive enough for predicting the erosion of polyimide film under various exposure conditions. One of the major factors that influences the erosion rate of polyimide film is the impingement angle of atomic oxygen. However, an accurate measurement of the impingement angle dependence has not been reported.

In this Note, we report ground-based experimental results of the impingement angle dependence of the erosion rate of polyimide film due to exposure to a hyperthermal atomic oxygen beam. In situ mass loss measurements were made during the atomic oxygen exposure by using a quartz crystal microbalance (QCM), so that any possible disturbance influencing the reliability of the postprocess erosion measurement such as moisture absorption, contamination, or unexpected change in the beam conditions during the exposure could be eliminated.

Experiments

The laser detonation-type atomic oxygen beam source, which was originally invented by Physical Sciences, Inc. (PSI), was used in this study.[2] Details of the experimental apparatus are reported elsewhere.[3] The translational energy of the atomic oxygen beam used in this study was approximately 4.6 eV, whereas the beam flux at the sample position was measured at 3.0×10^{14} atom/cm^2/s by using a silver-coated QCM.[4] The polyimide film used in this study was the pyromelliticanhydride(PMDA)-oxydianiline (ODA) polyimide supplied by Toray Industries, Inc. (Semicofine SP-510). The polyimide film was spin coated on the QCM sensor crystal and annealed at 150°C and then at 300°C. Details of the sample preparation are reported in Ref. 5. The polyimide film, thus prepared, was examined by x-ray photoelectron spectroscopy, and it was confirmed that the surface structure was similar to that of Kapton-H®, which is a commercially available polyimide film. The erosion rate of the polyimide film was calculated from the change in the resonant frequency of the QCM during the atomic oxygen beam exposure. The frequency of the 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°C. Before the mass loss measurements, the polyimide film was exposed to atomic oxygen (6×10^{17} atoms/cm^2) to saturate the surface oxygen content of the sample. This is done to avoid the effect of nonlinear mass loss phenomenon at the beginning of atomic oxygen exposure to pristine polyimide surfaces.[6]

Results and Discussion

Figure 1 displays the frequency shift of the QCM during atomic oxygen beam exposures at impingement angles from 0 to 90 deg. The impingement angle was taken with respect to the surface normal. A good linear relationship between the frequency shift and exposure time, that is mass loss and atomic oxygen fluence, was observed at all impingement angles. The good linearity of the mass loss with fluence was also identified for larger timescales.[6] The results shown in Fig. 1 were obtained at a sample temperature of 38°C, but similar results were also observed at sample temperatures from 15 to 70°C. The slope of the mass loss rate at every impingement angle was calculated by a least-squares fit and was plotted against the impingement angle. The results are presented in Fig. 2. It is clear that the rate of frequency shift, or erosion rate, of polyimide depends on the impingement angle and that the dependence obeys a cosine law as indicated by the solid line in Fig. 2. Note that the

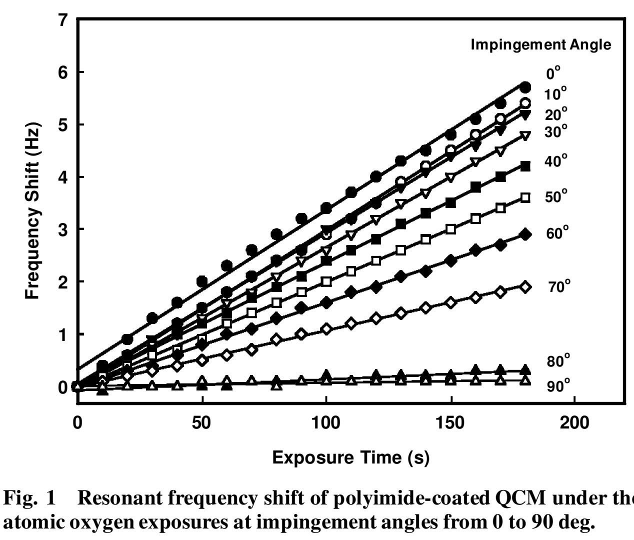

Fig. 1 Resonant frequency shift of polyimide-coated QCM under the atomic oxygen exposures at impingement angles from 0 to 90 deg.

Received 20 April 2001; revision received 2 July 2001; accepted for publication 6 July 2001.

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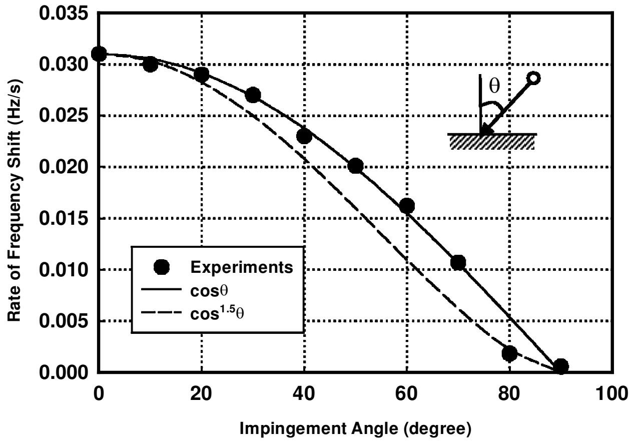

Fig. 2 Rate of frequency shift of the polyimide-coated QCM as a function of impingement angle.

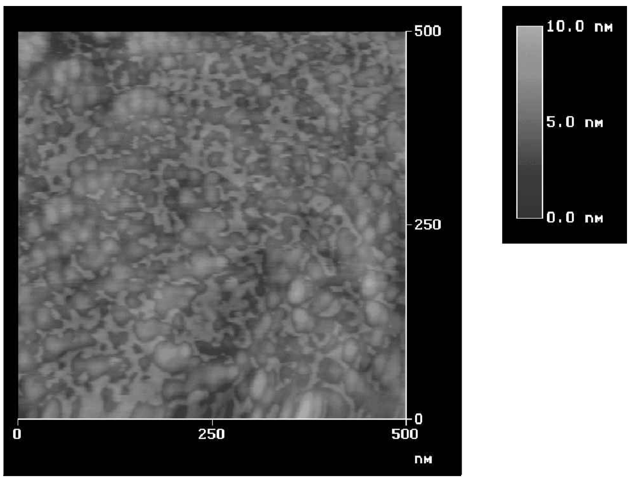

Fig. 3 Tapping mode atomic force microscope image of atomic oxygen exposed polyimide surface. atomic oxygen fluence, 8.8 × 10^{17} atoms/cm^2; scan area, 500 × 500 nm.

data point at the impingement angle of 80 deg was affected by the QCM holder, which blocked a part of the incoming atomic oxygen beam.

Banks et al. reported that the impingement angle dependence of the erosion of fluorinated ethylene propylene (FEP) Teflon® in the long-duration exposure facility (LDEF) flight experiment followed a $\cos^{1.5}\theta$ law rather than a cosine law.[7] An analysis of the flight data of Kapton-H and Mylar® onboard STS-8 concluded that the impingement angle dependence followed a $\cos^{1.5}\theta$ law.[8] However, their conclusions were based either on a small number of data points obtained by the flight experiments or on the large uncertainty of the data, which spoils the accuracy of the analysis. Furthermore, no physical explanation was provided for the $\cos^{1.5}\theta$ dependence.

The cosine law of the impingement angle dependence observed in this experiment was physically explained as follows: The effective flux of atomic oxygen at the sample surface decreases with increasing impingement angle; the effective flux of atomic oxygen is in proportion to the cosine of the impingement angle. The fact that the impingement angle dependence of the erosion rate follows a cosine law clearly indicates that the erosion rate is proportional to the effective flux of atomic oxygen, that is, the reaction yield of oxygen atom is independent of the impingement angle. Figure 3 shows the atomic force microscope image of the polyimide film that was exposed to atomic oxygen with a fluence of 8.8×10^{17} atom/cm^2. Note that all experimental data shown in Figs. 1 and 2 were obtained using the same sample, so that the atomic oxygen fluence at the sample surface reached 10^{18} atoms/cm^2, including preexposure of 6×10^{17} atoms/cm^2, when mass loss data were taken. Although the atomic oxygen fluence is relatively small compared with many in-flight experiments, viewed at the microscopic level, the surface of the polyimide was already roughened due to the atomic oxygen attack. The peak-to-valley height of the surface was larger than 10 nm, which is approximately 100 times larger than the size of a carbon atom. Therefore, on the microscopic scale, the impingement angle of oxygen atoms incident to the polyimide surface is widely distributed due to the presence of microscale roughness even though the macroscopic impingement angle is fixed. In addition, the multiple bounce effect, which is a key to the high reaction yield of atomic oxygen at the rough graphite surface,[9] also promotes the independence from impingement angle in the reaction. Therefore, the microscopic roughness and the multiple bounce effect at the polyimide surface erase the impingement angle dependence of atomic oxygen reactivity, and the macroscopic erosion phenomena of polyimide simply reflects the effective fluence of atomic oxygen, which follows the cosine law with the macroscopic impingement angle.

Conclusions

The dependence on impingement angle of atomic oxygen reaction with polyimide film was investigated. In situ mass loss measurements during atomic oxygen beam exposure clearly indicated that the erosion rate of polyimide obeys a cosine law. The physical explanation of this phenomenon was made by considering the microscopic impingement angle at the polyimide surface under the presence of the microscale roughness.

Acknowledgments

A part of the study was supported by a Grant-in-Aid for Scientific Research from the Ministry of Education, Culture, Sports, Science and Technology, Japan, and by the space utilization research promotion from the Japan Space Forum. The authors acknowledge K. Ishikawa of Kobe University for his help in experiments.

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