

Energy Dependence of Hyperthermal Oxygen Atom Erosion of a Fluorocarbon Polymer: Relevance to Space Environmental Effects

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ABSTRACT The origin of the inconsistency in the erosion phenomena of fluorocarbon polymers between a ground-based atomic-oxygen test environment and the low Earth orbital space environment has been investigated. A detailed experiment was performed in order to study the effect of the high-energy component in the atomic-oxygen beam pulses on the erosion. The experiment was carried out with the combination of a polymer-coated quartz crystal microbalance and a high-speed chopper wheel installed in a laser-detonation atomic-oxygen facility. It was clearly observed that the mass-loss rates of both polyimide and fluorocarbon polymers depend on the impact energy of atomic oxygen. However, the energy dependence is more significant on the fluorocarbon polymer compared with that on polyimide. Collisions of atomic oxygen with translational energies higher than ~ 5 eV induce a significant mass loss on the fluorocarbon polymer. These experimental findings agree with earlier measurements of the volatile products released during hyperthermal O-atom bombardment, and they are consistent with theoretical calculations on related systems. It is concluded that the difference in the collision energy distribution of atomic oxygen between laboratory and space environments is the major reason for the difference in erosion yields measured in the two environments.

KEYWORDS: hyperthermal • atomic oxygen • fluorinated polymer • space environment • low Earth orbit • polymer degradation

1. INTRODUCTION

It has been recognized that the erosion yields of fluorocarbon polymers such as fluorinated ethylene propylene copolymer (Teflon-FEP) or polytetrafluoroethylene (PTFE) in ground-based atomic-oxygen tests using a laser-detonation atomic-oxygen source are much greater than those in flight tests in low Earth orbit (LEO). Because Teflon-FEP has been widely used as a thermal control material for satellites in LEO, this inconsistency is vexing to those who wish to predict the longevity of this material based on atomic-oxygen testing using the widely accepted laser-detonation source. The origin of this problem has been attributed in general to the differences in the flux of ultraviolet radiation the laboratory and space environments and in particular to the vacuum ultraviolet (VUV) radiation from the laser-sustained oxygen plasma in the nozzle (1). Following this hypothesis, many studies have been conducted to study the synergistic effects of atomic oxygen and ultraviolet (UV) radiation in ground-based tests (2–4) and in flight experiments (5). For many years, the role of synergistic effects of atomic oxygen and UV or VUV light in the erosion of Teflon-

FEP have been debated, because it has been difficult to control the experimental variables sufficiently to confirm or rule out such effects. Recently, the groups of Tagawa and Minton have conducted experiments that clearly identify an enhanced erosion rate when ultraviolet light accompanies an atomic-oxygen exposure, but they show that this enhancement is relatively small. Tagawa and co-workers confirmed the certain role of extreme ultraviolet (EUV) radiation from the laser-sustained oxygen plasma in the enhanced erosion rate of a fluorocarbon polymer, but they demonstrated that the enhancement is additive and not synergistic (6). They also found that the erosion of the same fluorocarbon polymer by an atomic-oxygen beam and an external source of VUV radiation shows an additive effect (7). Minton and co-workers identified an enhancement in the production of volatile reaction products when VUV radiation was added to atomic-oxygen bombardment of Teflon-FEP (8), and they also found that the VUV light contributed in an additive way to the erosion yield (9). They further demonstrated a similar additive effect in the erosion of poly(methyl methacrylate) (PMMA) by atomic oxygen and VUV light. The latter result was particularly significant because PMMA is known to degrade easily in the presence of VUV light and yet the bulk of the erosion of this material was attributed to atomic oxygen. The conclusion of the studies in the Tagawa and Minton laboratories was that the high translational energies of the oxygen atoms produced in the laser-detonation source relative to the O-atom impact energies in LEO lead to higher erosion rates in the test environments than in the LEO

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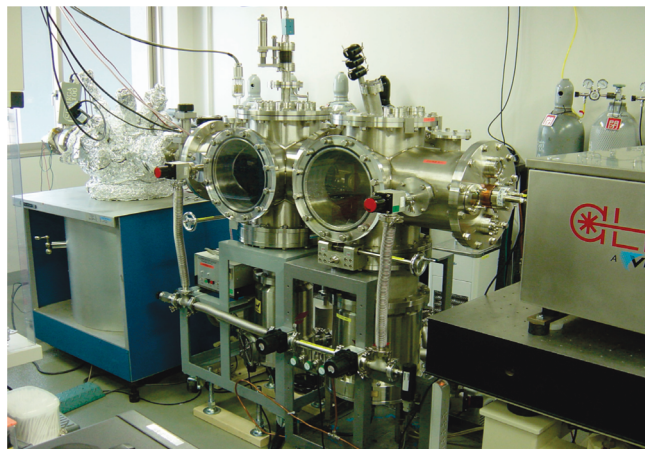


FIGURE 1. Photograph of the atomic-oxygen beam facility used in this study. Two 1500 L/s turbo molecular pumps are used to maintain a vacuum in the main chamber.

environment. In LEO, the average collision energy of atomic oxygen with ram surfaces was reported to be 5.6 eV, with a full width at half-maximum of 1.7 eV, by EOIM-3 mission (10). Note that even though the average collision energy was reported to be 5.6 eV, the average energy and full-width at half-maximum (FWHM) of atomic oxygen in circular orbit at an altitude at 400 km are calculated to be 4.5 and 1.4 eV, respectively, in the more recent literature (11). In contrast, the average translational energy of atomic oxygen produced by a laser-detonation source is typically 5 eV or higher, and the FWHM is roughly 2–5 eV. If, as was shown in the beam-surface scattering experiments of Minton (8) and in theoretical calculations by Troya (12), the O-atom reactivity with a fluorocarbon rises rapidly above 4 eV, then one might expect the laboratory exposures to yield a higher erosion yield than the LEO exposures. Although the energy dependence of O-atom reactions with Teflon-FEP was observed in the beam-surface scattering experiments of Minton (8), the energy dependence on the actual erosion yield was not proven. This is an important point to be clarified in order to improve the quantitative predictions of polymer erosion based on atomic-oxygen testing in ground-based atomic oxygen environments.

We report here a study of the energy dependence of hyperthermal atomic oxygen on the erosion rate of a fluorocarbon polymer. We compare the erosion rates of polyimide and the fluorocarbon polymer in order to study the sensitivity of the erosion of these two types of materials to O-atom impact energy and gain insight into the erosion mechanisms.

2. EXPERIMENTS AND SPECIMENS

The laser detonation atomic-oxygen beam source used in this study has been developed at Kobe University in order to study gas–surface reactions of atomic oxygen with solid materials (13–16). This source is based on an original design by Physical Sciences Inc. (PSI) (17). Figure 1 shows a photograph of the facility. The laser-detonation beam source uses a pulsed supersonic valve (PSV) and a carbon dioxide laser (10.6 μm , 5–7 J pulse⁻¹). The laser light is focused into the nozzle throat with a spherical Au mirror located 50 cm away from the nozzle. The PSV introduces pure oxygen gas (99.8%) into the nozzle and

the laser light is focused into the gas cloud in the nozzle. The energy for dissociation and acceleration are provided by the inverse Bremsstrahlung process. The hyperthermal beam, thus generated, was characterized by a time-of-flight (TOF, number density as a function of time) distribution measured by a quadrupole mass spectrometer with a scintillation detector which is installed in the beamline. Translational energies of the species in the beam were calculated using TOF distributions with the flight length of 238 cm. The laser-detonation atomic-beam source can deliver intense atomic-oxygen beam pulses with an average velocity of approximately 8 km s⁻¹.

To change the average translational energy of the atomic oxygen, a high-speed chopper system (9000 rpm) was utilized in this study. This high-speed chopper system was originally installed in order to block the ultraviolet light from oxygen plasma (18). The collision energy of atomic oxygen could be adjusted with this chopper wheel by blocking a part of the atomic oxygen beam. Figure 2 shows a block diagram and a photograph of the chopper system. Blocking of a part of the beam was made by changing the system delay. A grid with 10 mesh (transparency of 71%) is located 5 cm upstream from the sample in order to eliminate positive or negative charged particles. A 5 MHz Ag-coated quartz crystal microbalance (QCM) sensor was placed at the bottom of a rotatable manipulator in order to measure the flux of atomic oxygen (19). The typical atomic oxygen flux after slicing by the chopper wheel was estimated to be 0.7–2.0 $\times 10^{13}$ atoms cm⁻² s⁻¹ at the sample position.

The samples used in this study are a polyimide and a fluorocarbon polymer. A polyamic acid was spin-coated on a quartz crystal at 12 000 rpm for 30 s, and curing treatments at 150 °C for 1 h and 300 °C for 1 h in N₂ were carried out in order to form the polyimide structure with a thickness of approximately 0.1 μm . The spin-coated polyimide film, thus formed, has the same chemical repeat unit as Kapton-H (PMDA-ODA polyimide). In contrast, the fluorocarbon polymer was prepared by a plasma-assisted physical vapor deposition technique developed at the Technology Research Institute of Osaka Prefecture, Japan (20). The starting material of the fluorocarbon polymer used in this study was PTFE. The change in mass of the films was detected from the frequency shift of a QCM on which the films were coated. The resonance frequency of the QCM was recorded every 10 s with a frequency resolution of 0.1 Hz. All beam exposure experiments were carried out with the sample temperature at 38 °C and with the atomic oxygen beam impinging at normal incidence.

3. RESULTS AND DISCUSSION

3.1. Elimination of Ion Component.

It has been recognized that Teflon-FEP is sensitive to ion exposure. The contribution of an ionic component to the erosion of sample polymers during atomic-oxygen beam exposure was evaluated with the use of the grid located in front of the chopper wheel. An atomic oxygen beam with an average translational energy of 5.8 eV was used in this study. The relationships between the grid potential and the erosion yields of the polyimide and fluorocarbon polymers are shown in Figure 3. Note that the samples were kept at ground potential in these experiments. It is found that the reaction efficiency of polyimide (solid circles) is insensitive to the grid potential (–350 V to +350 V). For the fluorocarbon polymer (open circles), no significant change in reaction efficiency is obvious with a positive grid potential. TOF spectra of ions, which were measured by the QMS with ionizer turned off, did not vary when the grid potential was positive. This experimental

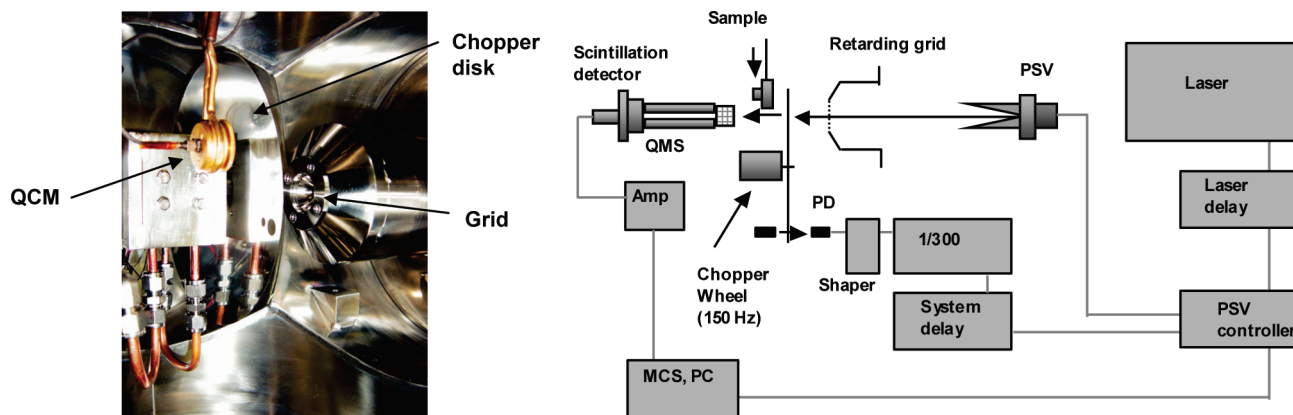


FIGURE 2. Photograph of the chopper wheel inside the laser-detonation atomic-oxygen beam source (left panel) and the system diagram (right panel). The chopper was operated at 9000 rpm under a pressure of 4×10^{-7} Torr.

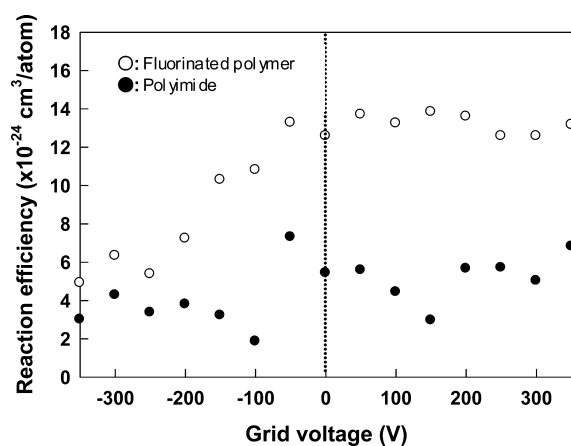


FIGURE 3. Reaction efficiency of polyimide and fluorinated polymer as a function of the grid voltage.

finding suggests that positive ions are not involved in the atomic-oxygen beam. On the other hand, the reaction efficiency of the fluorocarbon polymer decreased with increasing negative grid potential. This result suggests that the fluorocarbon polymer is much more sensitive to negatively charged particles in the beam than polyimide. The current at the sample position was measured to be 1.84×10^{-7} C pulse⁻¹ without any grid potential, which is 0.38% of the atomic oxygen flux measured by the Ag QCM if all particles are considered to be singly charged particles. The current was decreased to 0.026% with a grid potential of -350 V. From the results indicated in Figure 3, it was concluded that negatively charged species (maybe O^- or electrons) could affect the erosion of the fluorocarbon polymer but not the polyimide. This phenomenon is consistent with the soft-X-ray induced etching of Teflon-FEP, which has been attributed to secondary electrons (21). Thus, it was confirmed that the contribution of negatively charged ions or electrons in the beam could not be ignored for fluorocarbon polymers if they are involved in the beam. In the following section, all of the experiments were carried out with a grid potential of -350 V so that the effect of negatively charged particles in the beam was minimized.

3.2. Collision Energy Dependence. Flux-weighted TOF spectra, $N(t)/t$ versus t , and average collision energies of atomic-oxygen pulses sliced by the chopper wheel are

shown in Figure 4. Panels a and b in Figure 4 show the flux-weighted TOF distributions in which slow and fast parts, respectively, of the beam pulse were partially blocked by the chopper wheel. It is clearly observed that the atomic oxygen beam pulse, with an overall average energy of 5.5 eV and FWHM of 5.2 eV, is sliced by the chopper wheel operating 150 Hz (9,000 rpm). The average energy of the sliced beam is adjustable from 1.3 to 8.9 eV by changing the system delay. The polyimide- and fluorocarbon polymer-coated QCMs were installed in the beamline, and the changes in resonance frequency of the QCMs were recorded during the exposure to the sliced atomic-oxygen beam. The results are presented in Figure 5. Panels a and b in Figure 5 indicate the frequency shift of the polyimide- and fluorocarbon-coated QCMs during the atomic oxygen beam exposure, respectively. The average collision energy (E_{coll}) for each exposure condition is indicated at the right side of the graph. It is observed that the frequency shift of the polyimide-coated QCM, which is proportional to the mass-loss of the film, is negative when low-energy atomic-oxygen beams were used ($E < 4$ eV). Such a mass-increase phenomenon is believed to be the result of initial surface oxidation reactions in which oxygen is taken up by the surface while erosion is negligible (22). The lack of a negative shift of resonance frequency of the fluorocarbon-coated QCM with low-energy atomic-oxygen exposure suggests that oxidation of this surface does not occur with low O-atom energies. The frequency shifts of both the polyimide- and fluorocarbon-coated QCMs are positive with higher translational energies of the atomic-oxygen beam. The positive frequency shift of the fluorocarbon-coated QCM becomes particularly significant with O-atom translational energies near 6 eV and above. The rate of frequency shift for the fluorocarbon polymer with increasing O-atom energy is higher than that for the polyimide, indicating that the energy dependence of the reactivity is more pronounced for the fluorocarbon polymer in this energy range. The flux of the atomic oxygen beam changes with its translational energy. The flux-compensated erosion rate (E_r), which corresponds to the relative reaction efficiency of atomic oxygen, was calculated with the equation below

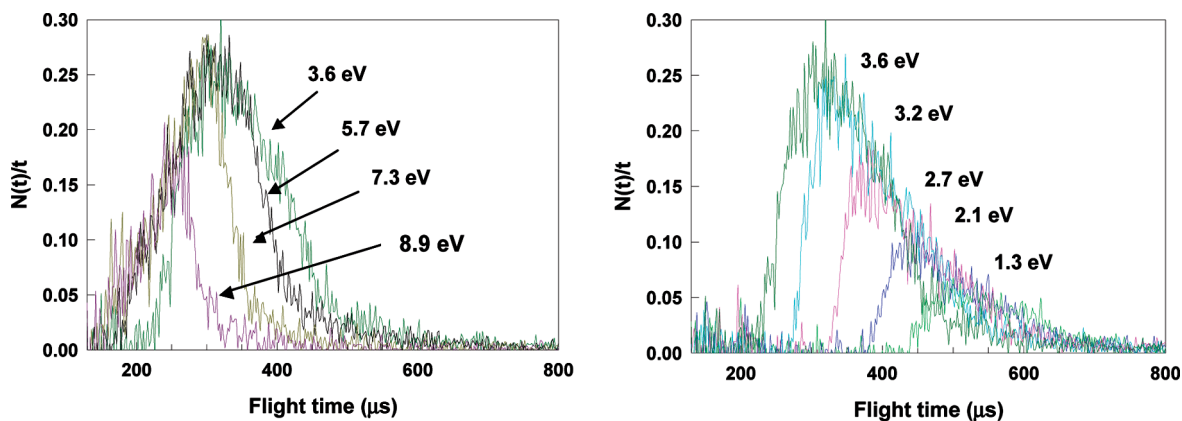


FIGURE 4. Time-of-flight spectra of atomic oxygen beam ($m/z = 16$) sliced by the high-speed chopper wheel in which the slow and fast parts of the beam pulses are blocked in (a) and (b), respectively. The same TOF spectrum ($E_{\text{coll}} = 3.6$ eV) is shown in both panels for comparison.

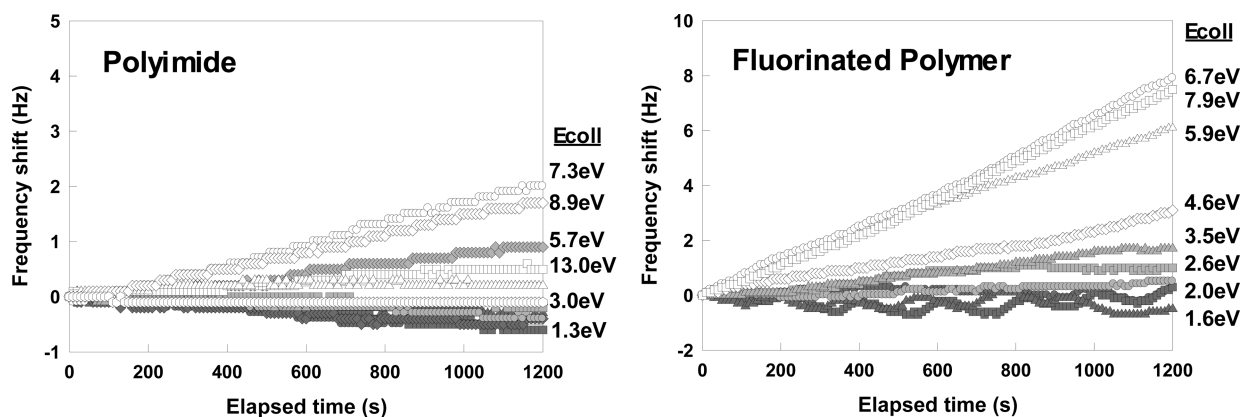


FIGURE 5. Frequency shift of (a) polyimide-coated QCM and (b) fluorocarbon polymer-coated QCM during atomic-oxygen exposures with various O-atom translational energies. Average translational energies in each exposure condition are shown in the right side of each panel.

$$Er = \frac{[\text{Slope of frequency shift of QCM}]}{[\text{Relative flux of beam}]} \quad (1)$$

The relative flux is obtained from the flux-weighted TOF distributions by using the integral of $N(t)/t$. The results are shown in panels a and b in Figure 6. It is clear that the relative reaction efficiency increases only slightly with O-atom translational energy for polyimide. However, the reaction efficiency of atomic oxygen with the fluorocarbon polymer is almost zero for O-atom energies lower than 5 eV and appears to increase rapidly for O-atom energies above 5 eV. Because the energy dependence on the reaction efficiency of the fluorocarbon polymer is much stronger than that of the polyimide, the relative erosion of the fluorocarbon polymer compared to polyimide will be enhanced when a high-energy component ($E > 5$ eV) is present in the atomic-oxygen beam. Atomic oxygen beams generated by the laser-detonation method typically have significant translational energy components above 5 eV unless they are chopped, which is rarely the case for a materials test. Given that most atomic oxygen tests essentially determine a ratio of the amount of material loss of the test specimen to that of a Kapton polyimide standard (23), a material such as a fluorocarbon polymer that has a stronger energy depen-

dence than polyimide will appear to have a higher reaction efficiency in environments where the average O-atom translational energy is higher. It is thus concluded that the apparently high reaction efficiency of a fluorinated polymer in a ground-based test with a laser-detonation source is the result of the high-energy component in the atomic-oxygen beam. A key result from this study that is generally relevant to atomic-oxygen testing is that the erosion rate of polyimide does surely depend on O-atom translational energy (although this dependence is relatively weak). As mentioned above, the atomic oxygen fluence in an atomic-oxygen test is usually determined from the mass-loss or recession of a Kapton H standard, which is assumed to have a constant reaction yield of $3.00 \times 10^{-24} \text{ cm}^3 \text{ atom}^{-1}$ (24). This reaction yield was established based on shuttle flights such that it is valid for relative velocities that are equivalent to O atoms with 4.5 eV of translational energy colliding with ram surfaces. The experimental results shown in Figure 6a indicate that the reaction yield of $3.00 \times 10^{-24} \text{ cm}^3 \text{ atom}^{-1}$ is not accurate when the O-atom energy in a test deviates from 4.5 eV.

3.3. Comparison with Earlier Results. Troya and Schatz have performed direct-dynamics classical trajectory calculations of collisions of hyperthermal ($E_{\text{coll}} = 4.5\text{--}6.0$ eV) ground-state atomic oxygen, $\text{O}(^3P)$, with hydrocarbon (CH_4

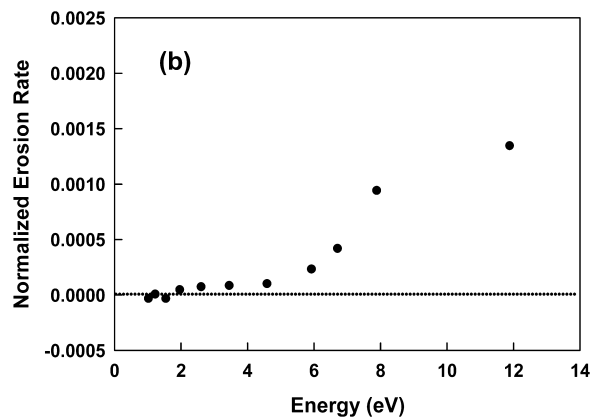
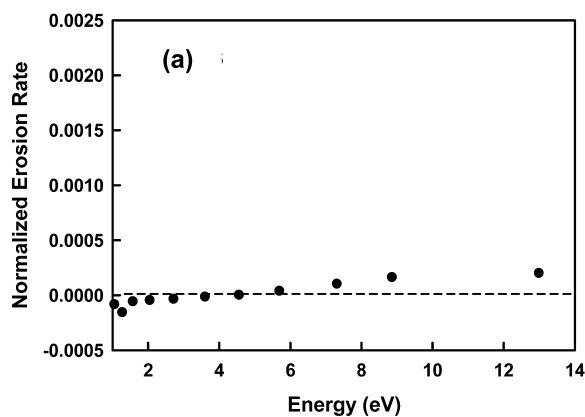


FIGURE 6. (a) Normalized erosion rate of polyimide and (b) fluorocarbon polymer as a function of atomic oxygen translational energy. The differences in atomic oxygen fluxes in the various sliced beams were compensated for by normalizing the QCM data to the relative O-atom beam flux.

and C_2H_6) and perfluorinated hydrocarbon (CF_4 and C_2F_6) molecules (12). Their computational results also suggested that F-elimination is more important than F-abstraction (which is negligible), but the cross-sections for F-elimination are also very small. The C–C bond breakage cross-sections in the $O(^3P) + C_2F_6$ reaction is larger than the F-elimination cross-section. This is opposite to the case of $O(^3P) + C_2H_6$ reaction. For $O(^3P) + C_2F_6$, they calculated that the cross-section ratio, [F-elimination]/[C–C bond breakage], is 0.12 and 0.24 at $E_{coll} = 5.5$ and 6.0 eV, respectively. The difference between C_2F_6 and C_2H_6 reactions is due to the fact that C–C bond in C_2F_6 is 0.4 eV weaker than that in C_2H_6 because of the presence of electronegative fluorine atoms. The excitation function (reaction cross-section vs E_{coll}) for C–C bond-breakage reaction was also calculated in their study. This excitation function increases more drastically than the apparent excitation function in our experiment. This difference may be the result of a variety of factors, including the energy distribution in our beam as well as the fact that mass-loss is the manifestation of many reactions. Nevertheless, our comparison between the energy dependence of polyimide erosion vs fluorocarbon polymer erosion is consistent with the theory in that the theoretical excitation function for the $O(^3P) + C_2F_6$ reaction showed a stronger dependence on E_{coll} than did the excitation function for the $O(^3P) + C_2H_6$ reaction in the $4.5 < E_{coll} < 6.0$ eV range. The new results are also consistent with the excitation function for the production of volatile CFO-containing fragments that was measured by Minton and co-workers in their beam-surface scattering experiment (8). However, the theoretical cross-section for direct C–C breakage in C_2F_6 is smaller than that in C_2H_6 , whereas we observed a higher reactivity for the fluorocarbon polymer with higher O-atom energies. This result points to a quantitative discrepancy that still exists in the experimental results obtained in this study and the difference between Kapton-equivalent erosion yields that are obtained for Teflon-FEP in hyperthermal-beam and LEO environments. Neither of these environments ever produces an erosion yield of Teflon-FEP that is higher than that of Kapton polyimide. The difference in O-atom reactivity with the fluorocarbon polymer film used in this study and Teflon-FEP may be the key to this discrepancy.

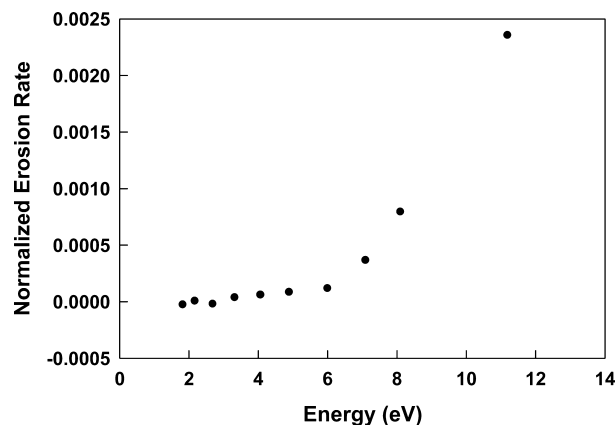


FIGURE 7. Normalized erosion rate of fluorocarbon polymer as a function of translational energy of molecular oxygen. The differences in molecular oxygen fluxes in the various sliced beams were compensated for by normalizing the QCM data to the relative O_2 beam flux, determined from time-of-flight distributions collected at a mass-to-charge ratio of $m/z = 32$.

Troya and co-workers also reported a classical-trajectory study of the dynamics of hyperthermal (5–12 eV) collisions between $Ar + C_2F_6$ and $Ar + C_2H_6$ systems (25). They found that C_2F_6 is more susceptible to collision-induced dissociation (CID) than C_2H_6 because of the lower frequencies in the heavier molecule. Three to six times more energy can be transferred to C_2F_6 than to C_2H_6 from incoming Ar atoms. Their computational results suggested a possible role of CID in the C–C breakage of fluorinated polymers.

The hyperthermal oxygen beam used in this study always contains a non-negligible fraction of molecular oxygen. Thus, in addition to reactions of atomic oxygen, molecular oxygen might also react, for example, by adding to a radical site and producing a peroxy radical, or it could contribute to C–C bond breakage through CID. Note that O_2 in the beam has roughly the same velocity as O, so the O_2 translational energy is twice as high. We cannot separate the role of CID by O_2 from reaction by O (or O_2) in our current experiments. As seen in Figure 7, when the normalized erosion rate of the fluorocarbon polymer is plotted against the translational energy of O_2 in the beam, we still observe a strong energy dependence. However, this correlation does not necessarily imply causality. In an earlier study, Minton and co-workers were able to

separate the effect of CID and chemical reaction by conducting experiments on the erosion of Teflon-FEP by a hyperthermal Ar beam (with 8 and 10 eV of translational energy) as well as a hyperthermal O/O₂ beam. They concluded that the contribution of CID to the erosion of Teflon-FEP by the hyperthermal O/O₂ beam was insignificant compared to direct reactions involving O (and possibly O₂). This conclusion is likely to be relevant to the work reported here.

In conclusion, direct reactions by high-energy collisions of atomic oxygen are considered to be the major contributors to accelerated erosion of fluorocarbon polymers in ground-based atomic-oxygen simulation facilities. This conclusion is valid when negatively charged particles are excluded from the exposure environment and when the flux of heavy particles (e.g., O₂) with similar velocities to the O atom is comparable to or less than the flux of O atoms. To achieve quantitatively predictive simulation results for atomic-oxygen-induced etching of fluorocarbon polymers in space, we must carefully control all environmental factors in the test environment in order to match the environment expected in space. The particular sensitivity of fluorocarbon polymers to the various environmental factors makes it more important to control the test environment for this material than for hydrocarbon-based polymers.

CONCLUSIONS

To study the differences in erosion efficiencies of fluorocarbon polymers in low Earth orbit and in ground-based simulation facilities, the dependence of fluorocarbon polymer erosion on O-atom translational energy was studied in the absence of the possible complicating effect of charged particles. For comparison, a similar study was conducted on a Kapton-like polyimide. In situ measurements of the erosion rates were performed with a quartz crystal microbalance. A high-speed chopper wheel was used in conjunction with a laser-detonation atomic-oxygen source to select relatively narrow translational energy distributions of O atoms in the range 1.3–8.9 eV. It was clearly observed that the mass-loss rates of both polyimide and fluorocarbon polymer depend on the collision energy of atomic oxygen. However, the effect was more significant with the fluorocarbon polymer. Translational energies higher than ~5 eV led to a significant mass loss on the fluorocarbon polymer. This experimental finding agrees with earlier theoretical calculations and with beam-surface scattering data on volatile-product formation. It was thus confirmed that the increase in the translational energy of impinging oxygen atoms resulted in an increase in the mass-loss rate on a fluorocarbon polymer relative to that of a polyimide. The results of this study suggest the importance of the translational energy and composition of the beam if quantitative predictions about space durability are to be made on the basis of ground-based simulations, particularly for fluorocarbon polymers. Furthermore, this study points out that the use of a constant erosion yield of $3.00 \times 10^{-24} \text{ cm}^3 \text{ atom}^{-1}$ for a Kapton fluence standard is only approximately valid and should only be considered to be accurate for fluences of tests with O-atom translational energies of 4.5 eV.

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