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Erosion of Kapton® H by Hyperthermal Atomic Oxygen

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Organic polymers are subject to erosion from ambient atomic oxygen in low Earth orbit. The linearity of the O-atom fluence dependence of Kapton® H erosion and the dependence of Kapton H erosion yield on surface temperature have been investigated. Sample exposures were performed with a pulsed beam containing hyperthermal O atoms that were generated with a laser detonation source. After exposure, samples were removed from the chamber in which the exposures were performed, and postexposure analyses were performed: etch depth (profilometry) and surface topography (atomic force microscopy). A systematic set of exposures, which eroded room-temperature Kapton H from 1.4 to 25 μm, showed that the erosion yield of Kapton H is linearly dependent on O-atom fluence. This result helps validate the use of Kapton H mass loss (or erosion depth) as a linear measure of the O-atom fluence of a materials exposure. The erosion of Kapton H was strongly temperature dependent. At lower temperatures (<100°C), the erosion yield appeared to be independent of sample temperature. However, above 100°C, the erosion yield exhibited an Arrhenius-like temperature dependence, with an apparent activation energy of 0.31 eV. These observations suggest that O-atom-induced erosion of Kapton H proceeds through direct, nonthermal, gas-surface reactions and through reactions that depend on surface temperature.

I. Introduction

IN the low-Earth-orbital environment, spacecraft materials are subject to erosion and oxidation from ambient atomic oxygen in the ground O(^3P) electronic state. The high relative velocity between a spacecraft and the ambient oxygen atoms results in O-atom reactivity that may be orders of magnitude higher than that of thermal (300 K) O(^3P) atoms. Organic polymers are especially susceptible to erosion from reactions with oxygen atoms in low Earth orbit, yet they are commonly used on spacecraft as thermal control and structural materials. The phenomenology of polymer erosion has been investigated extensively in space- and ground-based exposure environments.[1–3] A Kapton® H standard witness sample is commonly used to identify the effective O-atom fluence of an exposure.[3,4] Kapton H is assumed to erode linearly with O-atom fluence and, within the accuracy of this assumption, this material serves as a convenient reference material for determining the fluence of a test. The detailed mechanisms of polymer erosion resulting from reactions of hyperthermal atomic oxygen are still poorly understood because controlled O-atom environments are difficult to achieve and the mechanisms are complex. Even while research into the mechanisms by which organic polymers degrade under hyperthermal O-atom attack continues to reveal the rich chemistry that occurs in the extreme environment of low Earth orbit,[3,5–7] new insight into the erosion mechanisms may nevertheless be gained from erosion phenomenology.

The dependence of the erosion yield on sample temperature can provide general information about the erosion mechanisms. If the rate-limiting processes occur in thermal equilibrium with the surface, then a temperature-dependent study of the erosion rate will yield an effective activation energy for the rate-limiting processes. In contrast, if the rate-limiting processes do not occur in thermal equilibrium with the surface, then these processes would not be expected to depend on surface temperature, but rather on the collision energy between incident O atoms and the surface. Koontz et al.[8] have studied the temperature dependence of the erosion of Kapton H when this material was exposed to different thermal O-containing environments, and an Arrhenius form, $R_e = A\exp(E_a/RT)$, was used to describe the dependence of Kapton erosion yield R_e on the surface temperature T. Activation energies E_a of less than 0.29 eV were obtained. However, activation energies derived from temperature-dependent sample exposures in low Earth orbit are considerably smaller.[9] In fact, no temperature dependence was observed for Kapton erosion in low Earth orbit.[10–12] Yokota et al.[13] recently reported a very careful study of the temperature-dependent reactivity of a Kapton-like polyimide on exposure to two different O-atom beams, one containing O atoms with an average translational energy of 5.0 eV and the other containing O atoms with an average translational energy of 1.1 eV. By varying the sample temperature from 253 to 353 K and measuring mass loss in situ with a quartz crystal microbalance, they derived small activation energies of 5.7×10^{-4} eV for the 5.0-eV exposure and 4.5×10^{-2} eV for the 1.1-eV exposure. The low or nonexistent activation energies (or minimal temperature dependence) observed for hyperthermal O-atom exposures and the substantial activation energies observed for thermal O-atom exposures suggest that high-energy O-atom surface collisions, characteristic of those occurring on spacecraft surfaces at low-Earth-orbital altitudes, overcome any reaction barriers that would limit the erosion rate when O-atoms bombard a surface at low incident energies. Because the erosion yield of a polymer under hyperthermal O-atom attack does not appear to depend on surface temperature, it may be concluded that the erosion yield is determined by gas-surface reactions that do not occur in thermal equilibrium with the surface.

The experiments on the temperature dependence of erosion have, thus far, not systematically probed the temperature dependence of polymer erosion at temperatures above the highest temperature used by Yokota et al.,[13] that is, 353 K, and the studies by Yokota et al. used relatively low fluence exposures ($<10^{18}$ O atoms $\cdot$ cm^{-2}), so that the surfaces were fairly smooth during the exposures. After a significant fluence of directed hyperthermal O atoms ($>10^{19}$ O atoms $\cdot$ cm^{-2}), polymer surfaces become very rough.[3] Multiple collisions on such rough surfaces can drive incident O atoms toward thermal equilibrium with the surface. Therefore, reactions in thermal equilibrium with the surface might be more important for higher fluence exposures that result in many micrometers of surface erosion.

We have conducted a study of the dependence of the erosion yield of Kapton H on surface temperature over the temperature range from 298 to 573 K. The surface roughness of the eroded samples was characterized by atomic force microscopy. Accompanying every exposure for this study and for other (unrelated) studies in our laboratory was a Kapton H reference specimen that was held at

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296 K. Analysis of the erosion depth of all of these reference samples as a function of exposure duration has provided a measure of the linearity of the dependence of the Kapton H erosion yield on O-atom fluence.

II. Experimental Details

The sample exposures were conducted in the source region of a molecular beam apparatus.[3,5] A schematic diagram of the experimental arrangement is shown in Fig. 1. The pulsed, hyperthermal, O-containing beam was produced with a laser detonation source that is similar to the original laser-detonation source designed by Physical Sciences, Inc. (see Ref. 14). The source produces neutral beams (ion content $\ll 1\%$) containing 60–70% O atoms and 30–40% O_2 molecules traveling at velocities in the vicinity of 8 km · s^{-1}. The average O-atom translational energy used in the experiments reported here was ~5 eV, and the energy width (full width at one-half maximum) was ~2.5 eV. Characterization of the beam has been performed to determine that the hyperthermal O atoms are in their ground electronic state, $O(^3P)$ (Ref. 15). In a similar study, it was concluded that the O_2 molecules in the beam are in the ground $O_2(^3\Sigma_g^-)$ state.[16] These results establish that the exposure environment used in the laboratory subjects materials to ground-state O and O_2. The laser detonation source also produces a broad spectrum of light, extending, presumably, from the infrared to the vacuum ultraviolet (VUV); however, the spectrum and the intensity at the position of the samples is not known.

The samples were exposed to the hyperthermal beam at a distance of 40 cm from the apex of the conical nozzle source. A small portion of the O-atom beam passed through several apertures and regions of differential pumping before entering a mass spectrometer, which was used to characterize the ratio of O to O_2 in the beam and the velocity distributions of these species. The Kapton-equivalent O-atom fluence of an exposure was determined by measuring the erosion depth of a Kapton H reference sample that is assumed to have an erosion yield of 3.00×10^{-24} cm^3· O-atom^{-1}. The Kapton-equivalent fluence measurements led to the conclusion that the average O-atom flux of one beam pulse at the samples was $\sim 1.67 \times 10^{15}$ O atoms · cm^{-2} · pulse^{-1}.

The samples were placed in a sample mount (Fig. 2) such that the beam impingement angle was a few degrees off normal. The Kapton H reference sample is placed in the thermally isolated position 5. The center axis of the hyperthermal beam passes approximately 3 mm to the right of the sample mount at a level equal to the centers of sample positions 4, 5, and 6. Although sample position 6 is the closest to the beam axis, the beam is large enough to expose all of the samples at an almost equal flux. The entire mount was exposed to the beam at a repetition rate of 2 pulses · s^{-1}. Kapton H samples 1.22 cm in diameter and 125 μm thick were placed in the sample mount, with a stainless-steel wire mesh placed over them. The mesh allowed the erosion depth between the exposed and unexposed areas of the samples to be measured. The sample mount contains nine locations for samples. The reference sample was thermally isolated from the rest of the sample mount and was maintained at 296 K. The temperature of the samples at the other eight positions can be elevated from room temperature to 573 K by resistive heaters embedded in the sample mount. The specific temperatures used were 298, 333, 373, 423, 498, and 573 K.

Kapton H samples were always placed in positions 4 and 7 of the sample mount. To ensure uniform temperature of these thermally insulating samples, thin films (125 μm thick) of Kapton H were bonded to silicon substrates with a thermally conductive, silver-filled polyimide adhesive (Ablebond 70-1). This adhesive was cured in vacuum, with a maximum curing temperature of 573 K. When mounted in the sample mount, the back sides of the silicon substrates were in intimate contact with metal surfaces that were at the equilibrium temperature of the heated mount.

All samples used for studying the temperature dependence of the erosion yield were exposed to 100,000 pulses of the hyperthermal beam. Many additional exposures (for other purposes) were carried out over a 2-year period, and the Kapton H reference samples for all of the exposures during this period were used to study the dependence of Kapton H erosion depth on exposure duration. After exposure, samples were removed from the chamber and examined by profilometry and atomic force microscopy (AFM). The wire mesh allowed the measurement of many steps on exposed samples with the use of a Dektak-3 (Veeco Metrology Group) surface profiler. The average erosion depth and corresponding standard deviation for a given sample were calculated from measurements of 40–45 different step heights. AFM measurements of surface roughness of the

Fig. 2 Sample mount and sample position numbering scheme.

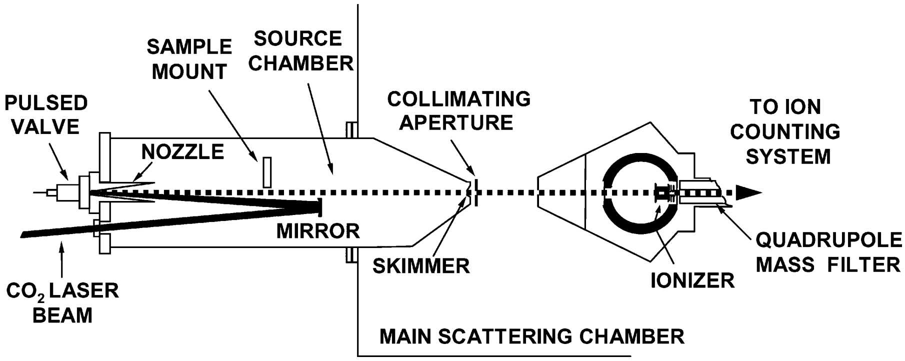


Fig. 1 Schematic diagram of beam source, sample mount location, main scattering chamber, and mass spectrometer detector.

exposed regions of sample surfaces were obtained with a Nanoscope IIIa Tapping Mode atomic force microscope (Digital Instruments).

III. Results and Analysis

The erosion depth of the Kapton H reference samples is plotted in Fig. 3 as a function of exposure duration, which varied from 28,000 pulses ($\sim$4 h) to 250,000 pulses ($\sim$35 h). These exposure durations correspond to Kapton-equivalent O-atom fluences of 3.68×10^{19} and 4.11×10^{20} atoms $\cdot$ cm^{-2}. These data were collected over a 2-year period, totaling 2,800,227 beam pulses and $\sim$389 h of exposures. There is variation in the per-pulse flux from exposure to exposure, but within the fluence uncertainties of the various exposures, the erosion depth appears to increase linearly with the O-atom fluence (exposure duration). The linear regression fit to the data goes to zero at a fluence greater than zero, which is consistent with earlier reports of an induction period before erosion commences.[17] However, the uncertainty in the fit would allow for the line to pass through the origin.

The erosion depths of Kapton H are plotted as a function of sample temperature in Fig. 4. Clearly, the erosion depth has a strong temperature dependence, especially at temperatures greater than 400 K. The erosion depths of the variable-temperature samples were divided by the erosion depths of the respective Kapton H reference samples, and the ratios are plotted as a function of temperature in Fig. 5. These data were fitted very well by an Arrhenius-like equation that includes a constant term, erosion ratio $= A \exp(-E_a/RT) + C$. The values of the constants A, E_a, and C are 1137, 0.31 eV, and 0.9114, respectively. The constant C must be added to the Arrhenius term because the erosion ratio tends toward a constant value of approximately one at low temperatures.

AFM images of unexposed and exposed Kapton H surfaces are shown in Fig. 6. All images were collected with the same area, 2.5×2.5 μm, and they are shown with a z scale of 500 nm. RMS surface roughnesses were calculated with the Nanoscope IIIa software and are shown in Fig. 7. Before exposure, the Kapton samples had smooth surfaces, with rms surface roughnesses of $\sim$7 nm. The exposed surfaces exhibited the familiar "shag-carpet" topography.[3] Surfaces that were eroded at the lowest temperature exhibited the largest roughness, and, as the sample temperature was increased, the roughness decreased significantly, reaching a minimum and then increasing again. The roughness measurements are consistent with the visual changes in surface topography (Fig. 6).

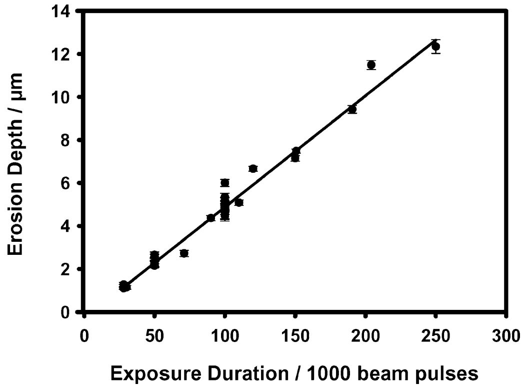


Fig. 3 Erosion of Kapton H as a function of exposure duration; error bars represent $\pm 1\sigma$, where σ is the standard deviation of 40–45 step-height measurements taken on individual sample.

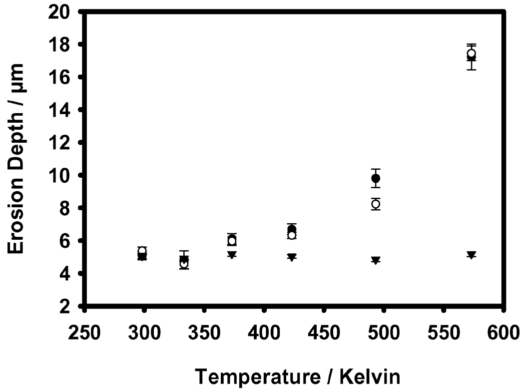


Fig. 4 Erosion depth of Kapton H as a function of temperature: ●, sample position 7; ○, sample position 4; and ▲, Kapton H reference sample (maintained at 296 K).

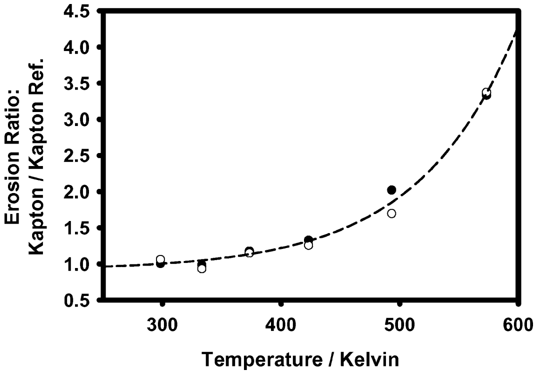
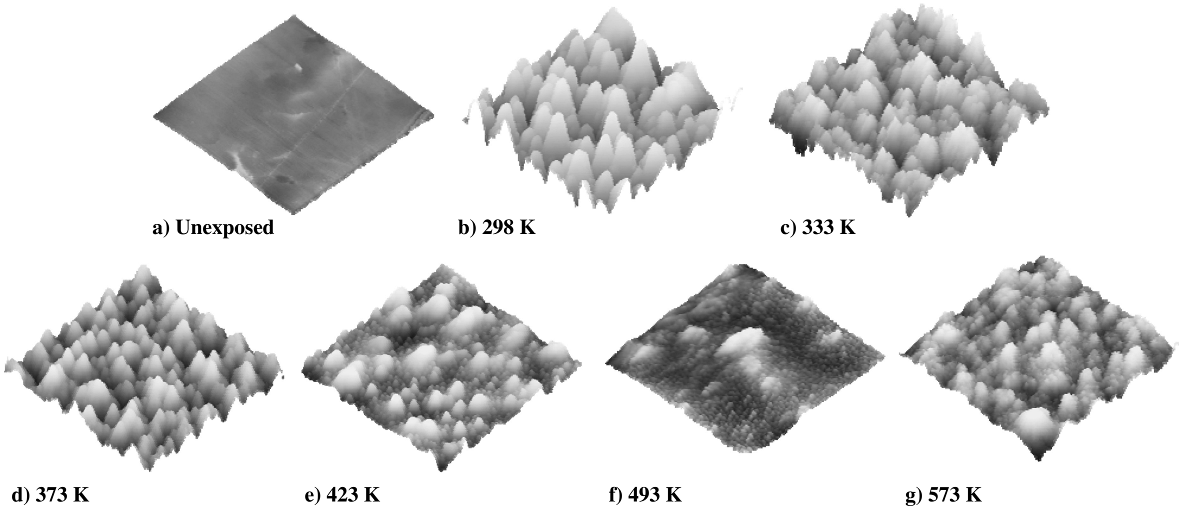


Fig. 5 Ratio of erosion depth of variable-temperature Kapton H samples to erosion depth of Kapton H reference sample as function of temperature: ●, sample position 7; ○, sample position 4; and - - - -, best fit through all points with two-term Arrhenius-like function (see text).

IV. Discussion

Kapton H is often used as a standard reference material to determine the (Kapton-equivalent) O-atom fluence of an exposure. This fluence is interpreted as being equivalent to a dose of normal-incidence, 5 eV O(^3P), atoms that would erode a Kapton H surface a specified amount, that is, the erosion depth of the Kapton H witness sample of an exposue. Although rarely discussed, the flux of the Kapton equivalent dose must be assumed to be low enough to avoid effects from gas-phase collisions near the surface. A safe upper limit to avoid such effects would be a continuous exposure at a flux of 10^{17} O atoms cm^{-2} $\cdot$ s^{-1} or pulsed exposure (of $\sim$10 μs duration) where the peak flux is less than $\sim 10^{21}$ atoms cm^{-2} $\cdot$ s^{-1} (Ref. 18). To calculate the Kapton-equivalent O-atom fluence, the value of 3.00×10^{-24} cm^3 $\cdot$ O-atom^{-1} is taken to be the erosion yield of Kapton H when it is eroded by 5-eV O(^3P) atoms at normal incidence. Note that Kapton-equivalent fluences are often reported for a variety of exposure environments, including environments that contain O atoms with translational energies much less than 5 eV and environments that are not well characterized, such as oxygen or air plasmas. Presumably, the validity of the reported Kapton-equivalent fluence improves as the exposure environment approaches that of a continuous stream of 5 eV O(^3P) striking a surface at normal incidence, with fluxes not greater than those just given. The beam source used in the studies reported here contains mostly O(^3P) atoms, and the impingement angle on the surface is near normal. Also, the beam is pulsed, not continuous. When a pulse width at the sample surface of $\sim$10 μs and a per-pulse flux of 1.67×10^{15} O atoms $\cdot$ cm^{-2} $\cdot$ pulse^{-1}, are assumed, the peak flux is $\sim 1.67 \times 10^{20}$ O atoms $\cdot$ cm^{-2} $\cdot$ pulse^{-1}. Despite this high peak flux, the number of atoms plus molecules in the pulse is not sufficiently high that gas-phase collision effects are very important above the sample surface.[18] Therefore, the O_2 and VUV light produced as byproducts in the source are expected to be the most significant deviations from the ideal exposure situation. O_2 could be involved in collisional processes[6,7] and in reactions with radical sites generated at the surface,[3] and VUV-induced photochemistry might generate reactive radical sites that would enhance the reactivity of O or O_2. Although molecular oxygen and VUV light might work in synergy with atomic oxygen to erode Kapton H, the conclusions drawn here will remain robust as long as the erosion mechanisms are dominated by the reaction of hyperthermal O(^3P), which is the dominant and most reactive component of the beam.



a) Unexposed b) 298 K c) 333 K

d) 373 K e) 423 K f) 493 K g) 573 K

Fig. 6 Representative AFM images of a) unexposed and b)–g) exposed Kapton H collected from samples exposed at sample position 4; all images are 2.5 × 2.5 μm and have z scale of 500 nm.

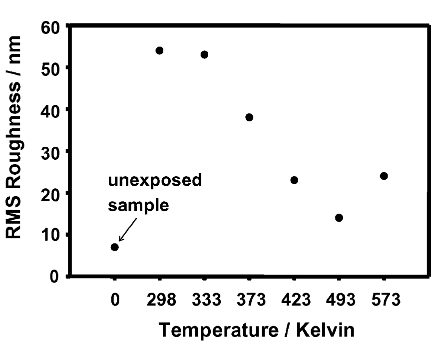


Fig. 7 RMS surface roughness as a function of sample exposure temperature; for each value, roughnesses from four 2.5 × 2.5 μm areas were averaged.

In spite of the possible effects of O_2 and VUV light, the main uncertainty in the study of the dependence of Kapton H erosion yield on fluence likely comes from the variation in the atomic-oxygen flux for exposures conducted on different days. The data in Fig. 3 provide a view of this variation, particularly in the erosion depths measured for exposure durations of 100,000 beam pulses. Even though we attempted to operate the beam reproducibly, the flux apparently varied by roughly $\pm 10\%$. This level of reproducibility of the beam flux represents the current state of the art for our source. Because we did not have an independent measure of the flux for the different exposures, the relative fluence for the data reported in Fig. 3 is taken to be given by the number of beam pulses. Within the uncertainty in the fluence, our data show that the erosion yield of Kapton H varies linearly with fluence. Although it is possible that the erosion yield may be slightly nonlinear with fluence, this nonlinearity must be small. Therefore, our data support the assumption that the erosion yield of Kapton H varies linearly with 5-eV $O(^3P)$ fluence. This result further validates the use of Kapton H mass loss or erosion depth as a linear measure of the O-atom fluence of a materials exposure.

The erosion depth of Kapton H showed a significant dependence on surface temperature. As the surface temperature was increased from room temperature to 573 K, the erosion depth increased by a factor of 3.3. This observation may seem to contradict the result by Yokota et al.,[13] which showed that the erosion yield of a Kapton-like polymer that was exposed to the O-containing beam from a laser-detonation source was almost independent of temperature. However, they studied the mass-loss rate over a narrower temperature range. They concluded that the erosion rate of the polyimide had an Arrhenius temperature dependence, with an activation energy of 5.7×10^{-4} eV for the temperature range from 253 to 353 K. This measurement employed a quartz crystal microbalance, and mass-loss data were obtained during the initial erosion period of the polymer. We also observed a negligible temperature dependence in a similar temperature range, from 298 to 373 K. In fact, our results agree with those of Yokota et al. in this temperature range, within the uncertainty of our data. The obvious temperature dependence that we observed became apparent at temperatures above those used by Yokota et al.

The two-term fit to the data in Fig. 5 suggests that the loss of material from a Kapton H surface is the result of temperature-dependent and temperature-independent mechanisms. The constant term C results from a mechanism that is temperature independent. This mechanism likely involves the direct reaction of O atoms with the surface on a timescale too short for thermal equilibrium to be achieved. Direct reactions of 5-eV O atoms with the surface should be able to overcome even significant reaction barriers, and so reaction would proceed independently of surface temperature and yield an apparent activation energy of zero. Many hydrocarbon reactions with $O(^3P)$ have hydrogen abstraction barriers in the range 0.2–0.5 eV (Ref. 19), whereas other reaction pathways involving, for example, hydrogen elimination and C–C bond-breaking may have barriers as high as 2 eV (Refs. 20 and 21). A variety of reactions may occur, given the available collision energy. Our results do not permit a conclusion about the nature of the temperature-independent mechanism. Based on analogous reactions of hyperthermal $O(^3P)$ atoms with gas-phase alkanes,[15,19–21] any energetically allowed reaction should be considered possible.

A temperature-dependent reaction process becomes important as the sample temperature increases. An Arrhenius functional form describes well the temperature dependence of Kapton H erosion depth at the higher temperatures used. This dependence suggests a mechanism that may take place in thermal equilibrium with the surface, implying that for this mechanism to occur, O atoms must transfer their energy to the surface and become trapped (either through physisorption or through reaction) before the rate-limiting erosion process(es) proceed(s). The trapping probability increases with surface roughness, which allows for multiple bounces at the surface that drive incident atoms toward thermal equilibrium and increase the likelihood that a reaction will occur. In the studies reported here, and in any situation where macroscopic amounts of material are removed by highly directional O atoms, the surface reaches a steady-state roughness. Therefore, the conclusion of a mechanism that depends on thermal accommodation (or trapping) of incident O atoms is consistent with the steady-state erosion of rough surfaces. Trapping would be expected to be less probable on smooth surfaces, possibly reducing the importance of the thermal mechanism and, thus, the temperature dependence of erosion.

The temperature dependence of the Kapton H erosion yield is undoubtedly linked to the dependence of surface roughness on temperature, but the relationship is not clear. At lower temperatures, at which the temperature-independent mechanism dominates, a very rough surface is produced. However, as the temperature is increased and the temperature-dependent mechanism starts to dominate, the eroded surface does not become as rough. The thermal mechanism retains no memory of the incident beam direction, and so O atoms that become trapped may react with equal probability anywhere on the surface. In addition, the reactivity of trapped atoms increases at higher temperatures. Both of these factors may contribute to the smoothing of the surface, but why the roughness reaches a minimum and begins to rise again with temperature is difficult to explain. The ultimate dependence of surface roughness on temperature represents a complicated interplay between the temperature-dependent and temperature-independent mechanisms.

V. Conclusions

The erosion depth of Kapton H as a function of exposure duration in a hyperthermal O-atom beam was investigated for a broad range of exposure durations covering almost two orders of magnitude. Within the reproducibility of the exposure flux provided by our laser-detonation source, the erosion depth of Kapton H was found to be linearly dependent on O-atom fluence. This result is consistent with the use of Kapton H as a linear erosion standard for materials testing in atomic oxygen environments.

The dependence of Kapton H erosion by hyperthermal atomic oxygen on surface temperature was studied by directing a hyperthermal O-containing beam at Kapton H samples held at various temperatures and then conducting postexposure analyses of these samples by profilometry and by AFM. Although the detailed erosion mechanisms are not known, it appears that a temperature-independent process dominates the erosion at temperatures lower than ~400 K and that both this temperature-independent process and an additional temperature-dependent process contribute to the erosion at temperatures above 400 K. At the highest temperature used (573 K), the erosion yield resulting from the temperature-dependent process was 3.3 times that resulting from the temperature-independent process. The temperature-independent process is believed to involve direct reactions of hyperthermal O atoms with the surface, whereas the temperature-dependent process is believed to proceed after incident O atoms have become trapped on the surface, perhaps through physisorption or reaction. The activation energy for the temperature-dependent process was determined to be 0.31 eV, which is similar to the barrier for an H-atom abstraction reaction: $O(^3P) + RH \rightarrow OH + R$.

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