



Temperature dependence study of FEP Teflon and Kapton H erosion in a simulated LEO
atomic-oxygen environment
by Deanna Marie Buczala

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in
Chemistry
Montana State University
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Abstract:

Polymer degradation occurs as a result of exposure to atomic oxygen in low Earth orbit (LEO). The damaging effects of the space environment on materials have prompted the simulation of LEO to study polymer erosion. The laboratory setting provides a method of evaluating material erosion by atomic oxygen attack in an affordable and time-efficient manner. This study investigates the temperature dependence of the erosion of Kapton H and FEP Teflon when exposed to hyperthermal 5 eV atomic oxygen beam. This study extends earlier investigations of the temperature dependence of Kapton H erosion and helps unveil the reaction mechanisms that lead to erosion of Kapton H and FEP Teflon in LEO. The investigations reported in thesis relied on the use of Kapton H to calibrate the atomic oxygen exposures, so an additional study was performed to verify the linear dependence of Kapton H erosion. Surface characterizations of the eroded samples in this study were performed by surface profilometry and atomic force microscopy.

The Kapton H erosion yield grew exponentially with temperature in the range 298 K to 573 K. The temperature dependence of Kapton H erosion could be explained by temperature-dependent and -independent mechanisms. The temperature-independent mechanism dominates at lower temperatures and involves the direct reaction of O atoms with the polymer surface, with an activation energy that is easily overcome by the O-atom collision energy with the surface. The temperature-dependent mechanism becomes important at higher temperatures. Apparently, at high surface temperatures, material is removed through reactions that occur in thermal equilibrium with the surface and have an effective activation energy of 0.31 eV.

From 298 K to 423 K the erosion yield of FEP Teflon grew exponentially. The nonlinear Arrhenius behavior suggested a two-term mechanism analogous to that used to describe the temperature dependent erosion of Kapton H. At temperatures lower than 373 K the temperature-independent mechanism dominates the material erosion and at higher temperatures, a mechanism becomes important that occurs in thermal equilibrium with the surface and has an activation energy of 0.24 eV.

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A thesis submitted in partial fulfillment
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This thesis has been read by each member of the thesis committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

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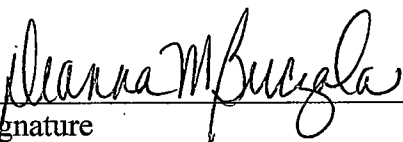
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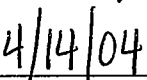
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This thesis is dedicated in the memory of my dad.

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ABSTRACT

Polymer degradation occurs as a result of exposure to atomic oxygen in low Earth orbit (LEO). The damaging effects of the space environment on materials have prompted the simulation of LEO to study polymer erosion. The laboratory setting provides a method of evaluating material erosion by atomic oxygen attack in an affordable and time-efficient manner.

This study investigates the temperature dependence of the erosion of Kapton H and FEP Teflon when exposed to a hyperthermal 5 eV atomic oxygen beam. This study extends earlier investigations of the temperature dependence of Kapton H erosion and helps unveil the reaction mechanisms that lead to erosion of Kapton H and FEP Teflon in LEO. The investigations reported in thesis relied on the use of Kapton H to calibrate the atomic oxygen exposures, so an additional study was performed to verify the linear dependence of Kapton H erosion. Surface characterizations of the eroded samples in this study were performed by surface profilometry and atomic force microscopy.

The Kapton H erosion yield grew exponentially with temperature in the range 298 K to 573 K. The temperature dependence of Kapton H erosion could be explained by temperature-dependent and -independent mechanisms. The temperature-independent mechanism dominates at lower temperatures and involves the direct reaction of O atoms with the polymer surface, with an activation energy that is easily overcome by the O-atom collision energy with the surface. The temperature-dependent mechanism becomes important at higher temperatures. Apparently, at high surface temperatures, material is removed through reactions that occur in thermal equilibrium with the surface and have an effective activation energy of 0.31 eV.

From 298 K to 423 K the erosion yield of FEP Teflon grew exponentially. The non-linear Arrhenius behavior suggested a two-term mechanism analogous to that used to describe the temperature dependent erosion of Kapton H. At temperatures lower than 373 K the temperature-independent mechanism dominates the material erosion and at higher temperatures, a mechanism becomes important that occurs in thermal equilibrium with the surface and has an activation energy of 0.24 eV.

CHAPTER 1

INTRODUCTION

The International Space Station (ISS) is a monumental scientific and engineering achievement; however, it is plagued with the problem of material deterioration as it maintains orbit above the Earth. This part of the Earth's atmosphere, from 200-700 km, is commonly referred to as low Earth orbit (LEO).¹ Since the early 1980's, it has been determined through both space- and laboratory-based experiments that materials degrade after long term exposure to the LEO environment.

The most abundant species in LEO is atomic oxygen. Atomic oxygen is formed when molecular oxygen is photodissociated by vacuum ultraviolet radiation.²⁻⁶ The mean free path at LEO altitudes is large enough that recombination to form O₂ or O₃ is negligible.⁷⁻⁹ In the steady state, molecular oxygen is generally less than one-tenth of the atomic oxygen density.¹⁰ Figure 1 illustrates the number density of various neutral species at LEO altitudes. The O-atom number density at a typical shuttle altitude of 300 km is $\sim 10^9$ cm⁻³. The number density of atomic oxygen is low, but when combined with the relative velocity between orbiting spacecraft and the ambient atmosphere (~ 7.4 km s⁻¹), the oxygen atoms bombard the ram surface of a spacecraft at a rate of $\sim 10^{15}$ O atoms cm⁻² s⁻¹. This corresponds to O atoms with a mean translational energy of 4.5 eV striking the leading edge of a satellite.^{1,5} The kinetic temperature of roughly 1000 K for the ambient atmosphere results in

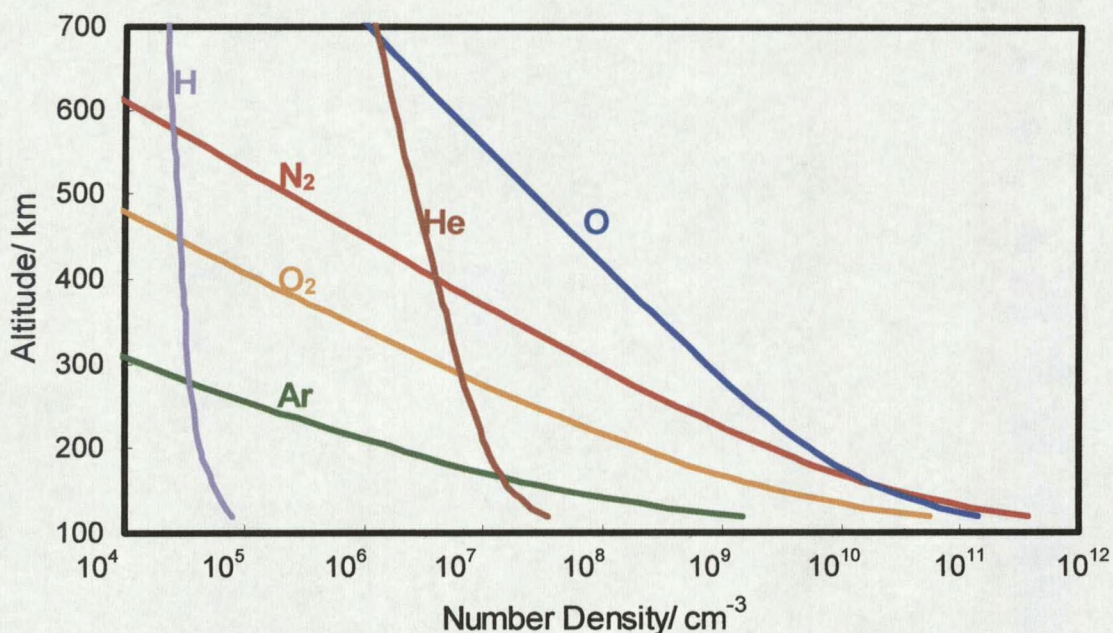


Figure 1. Number density of atomic and molecular species at low Earth Orbital altitudes.

an energy spread (full width half maximum) of ~ 2.5 eV in the collisions.¹¹

Material exposures *in situ* have the benefit of faithfully representing the LEO environment. Unfortunately, there are many disadvantages to space exposures due to the difficulty of maintaining control over all parameters. A major problem with in-flight exposure is the severe contamination that can result from mishandling of samples, material out-gassing,¹² and long periods between pre- and post-flight analysis. In addition, there may be other damaging space effects, including solar ultraviolet radiation, solar flare x-rays, electron and proton radiation, and temperature effects.¹³

Due to the limited availability and high cost of space exposures, the laboratory setting provides a method of evaluating material erosion by atomic oxygen attack in an

affordable and time-efficient manner.³ An ideal laboratory exposure would provide a controlled, faithful reproduction of the LEO environment; it would have a flux greater than 10^{15} atoms $\text{cm}^{-2} \text{s}^{-1}$, a mean collision energy of approximately 5 eV $\text{O}(^3P)$ atoms, and no impurities present (*e.g.*, VUV light, excited species, and ions).¹⁴ The laser-detonation source in this laboratory generates a high purity, ground state atomic/molecular oxygen beam with an O-atom translation energy of ~ 5 eV and a flux of $>10^{15}$ atoms $\text{cm}^{-2} \text{s}^{-1}$ (40 cm from the source).¹² The use of a hyperthermal atom beam source is beneficial to the study of polymer surfaces.⁴ Although, the laboratory is unable to simulate LEO completely, by gaining an understanding of the erosion mechanism of polymers in the controlled laboratory environment, it should then be possible to predict the durability of a material in a variety of environments.

Post-flight analysis of materials from early space flights identified the damaging effects of LEO on polymer surfaces.¹⁴ Many polymer films have desired mechanical and optical properties (solar absorptance and thermal emittance); in addition, they are easily fabricated and installed.¹³ The interest in these materials is due to their many applications on spacecraft: structural materials, space robots, manipulator arms, solar arrays, thermal blankets, and second-surface mirrors.^{12,15} Unfortunately, these materials experience degradation in optical and mechanical properties when exposed to LEO for extended periods of time, *i.e.*, weight loss, change in physical properties, loss of surface gloss, premature aging, and a reduction of thermal properties.^{1,8,9,13}

After several Space Shuttle operations, multiple studies found that the erosion of

polymeric material was caused by $O(^3P)$, enhanced by the presence of VUV radiation.^{6,11,16,17} For example, the Long Duration Exposure Facility (LDEF, 5.8 years in space) and the Hubble Space Telescope (HST, 3.6 years in space) have provided evidence of space degradation.¹⁸ Fluoroethylene propylene (FEP) Teflon and Kapton H polymers recovered from the leading edge of LDEF demonstrated significant erosion, with rough, sharp peaks that pointed in the direction of the atomic oxygen flow. When shuttle mission STS-41(LDEF) returned to Earth in January 1990, the Kapton H surfaces on a multilayer insulation blanket were oxidized and experienced weight loss due to surface erosion.¹⁸ The effect of incident angle on erosion was observed but not measured. FEP located on the trailing edge of LDEF received mostly VUV and little atomic oxygen bombardment. The morphology observed was a hard brittle layer not detected on the leading edge materials.¹⁹ The FEP multilayer insulation recovered from HST showed evidence of severe embrittlement along solar facing surfaces.¹⁷ The embrittled layer was not observed on polymers exposed primarily to atomic oxygen.¹⁹ The damaging effects of the space environment on materials has prompted the simulation of LEO to study polymer erosion.

Kapton H (pyromellitic dianhydride-oxydianiline (PMDA-ODA) polyimide) is one of the most studied polymers. The structure of Kapton is shown in figure 2. This polyimide polymer has been extensively studied because of its flexible and lightweight structure, temperature stability, excellent insulation properties, UV stability, and IR transparency.¹⁰ It is a structural material used for solar array blankets due to its mechanical strength and electrical properties. Kapton H is the standard polymer reference used by the space effects commu-

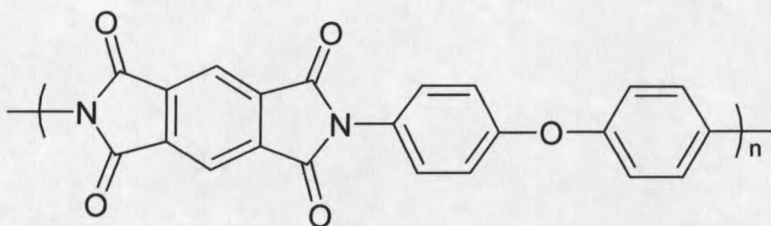


Figure 2. Structure of Kapton H (pyromellitic dianhydride-oxydianiline (PMDA-ODA) polyimide).

nity to determine the atomic oxygen fluence of an exposure test. The accepted O-atom-induced erosion yield of Kapton H is $3.00 \times 10^{-24} \text{ cm}^3 \text{ atom}^{-1}$.

The change in surface morphology after exposure to the LEO environment results in the development of a rough "shag-carpet." It was noted that needle-like structures form on the surface when exposed to hyperthermal rather than thermal oxygen atoms. Since thermal oxygen atoms did not alter the surface topography, this suggests that translational energy and/or direction of O-atom attack may be a factor influencing polyimide erosion.²⁰ Tagawa *et al.*²¹ studied the synergistic effects of atomic oxygen and VUV light on Kapton-like polymer surfaces. They reported that the erosion yield was similar to LEO when both atomic oxygen and VUV light were present. When atomic oxygen alone bombarded the surface, the erosion yield was significantly lower than the accepted value. The effect of VUV light alone was not studied. Volatile species were measured using a residual gas analyzer (RGA) detecting the release of CO and CO₂ products. The CO₂ product increased when both atomic oxygen and VUV were present. The increase in the gasification to CO₂ suggests the possible presence of a synergistic effect. Despite the amount of knowledge gained about the erosion of Kapton, the mechanism is still unknown.

Recently, Tagawa *et al.*²² studied the temperature dependence of Kapton-like polyimide erosion when the polymer was exposed to a 5 eV atomic oxygen beam similar to the beam in our laboratory. The polyimide film was formed when a polyamic amide acid was spin coated onto a quartz crystal microbalance (QCM) and cured at high-temperatures. X-ray photoelectron spectroscopy (XPS) confirmed the structure was similar to Kapton H. The mass of the film was measured every 10 seconds from shifts in the resonance frequency of the quartz oscillator during the exposure. The exposure measurements were obtained during the initial erosion of the polymer, with a fluence range of 2.6×10^{14} to 6.5×10^{16} atoms cm^{-2} . The sample temperature range was 253 K to $353 \text{ K} \pm 0.1 \text{ K}$ with a flux of 2.6×10^{14} atom $\text{cm}^{-1} \text{ s}^{-1}$. A plot of the natural log of the erosion yield ($\ln R_p$) versus inverse temperature (T^{-1}) for this limited temperature range gave a linear Arrhenius plot, with an activation energy (E_a) of $5.7 \times 10^{-4} \text{ eV}$ ($0.055 \text{ kJ mol}^{-1}$).

Fluoroethylene propylene (FEP) Teflon is a commonly used polymer due to its relatively high resistance to oxygen atoms; therefore, in space it has not been protected.¹⁸ The atomic oxygen effect on FEP erosion is small compared to other organic molecules that consist of carbon, hydrogen, and oxygen.²³ The structure of FEP is shown in figure 3. When coated with silver or aluminum backing, it has a high thermal emittance and reflection of incident solar energy, which are desirable properties for thermal insulating materials and flexible solar reflectors.^{1,9,24} It is commonly used for thermal control on exterior spacecraft surfaces, the top layer in multi-layer insulation, or a second-surface mirror on radiator panels. When ground and space exposure data are compared, both indicate a small degra-

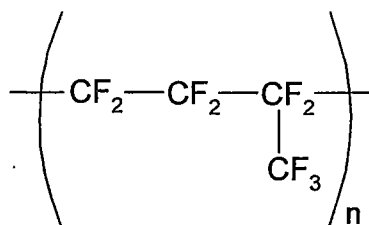


Figure 3. The structure of Fluoroethylene propylene (FEP) Teflon.

dation of thermal properties.¹⁸ FEP Teflon is prone to erosion, cracking, and subsequent mechanical failure in LEO.^{9,18} In space, factors that may contribute to FEP Teflon degradation are solar radiation, atomic oxygen, debris and micrometeoroid impacts, and thermal cycling, in addition to the stress applied by its configuration.¹⁸

This study investigates the temperature dependence of the erosion of Kapton H and FEP Teflon when these materials are exposed to a hyperthermal atomic oxygen beam. This study extends earlier investigations of the temperature dependence of Kapton H erosion and helps unveil the reaction mechanism responsible for surface erosion. The effect of temperature on FEP Teflon erosion is studied to understand the chemistry occurring on the polymer's surface. Surface characterization of the eroded samples in the present study was performed by surface profilometry and atomic force microscopy.

Chapter 2 describes the experimental details of the molecular beam apparatus, the sample geometry for each exposure, the sample preparation, and surface characterization methods.

Chapters 3 and 4 describe the results and discussion for the Kapton H erosion and temperature study. Chapter 3 presents the results and analysis of the experimental data of

Kapton H, while chapter 4 focuses on the discussion of the results of the Kapton H erosion and temperature study.

Chapters 5 and 6 present the results and discussion of the temperature study of FEP Teflon. Chapter 5 presents the results and analysis of the FEP Teflon erosion experiment and Chapter 6 focuses on the discussion of the effects of temperature on the erosion of FEP Teflon.

CHAPTER 2

EXPERIMENTAL METHODS

Experimental Configurations

The sample exposures were conducted with a molecular beam apparatus coupled to a laser detonation source, based on the original design of Physical Science Inc.²⁵⁻²⁷ A schematic diagram of the instrument is illustrated in Figure 4. The crossed molecular beam apparatus is a welded stainless steel box pumped down to pressures $<10^{-6}$ Torr by diffusion and cryo pumps mounted to the bottom by gate valves.²⁸ A surge of O_2 gas is introduced into the conical nozzle by a piezo-electric pulsed beam valve.^{26,27} As the gas expands into the conical nozzle, a ~ 7 J pulse $^{-1}$ CO_2 TEA laser beam operating at 2 Hz is focused by a

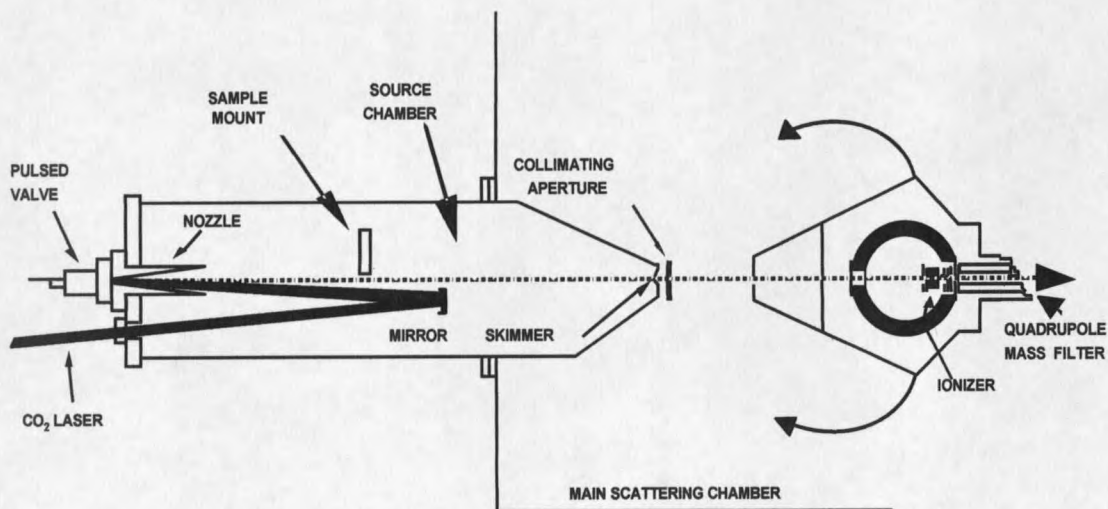


Figure 4. A schematic diagram of the beam source, sample mount location, main scattering chamber, and rotatable mass spectrometer detector.

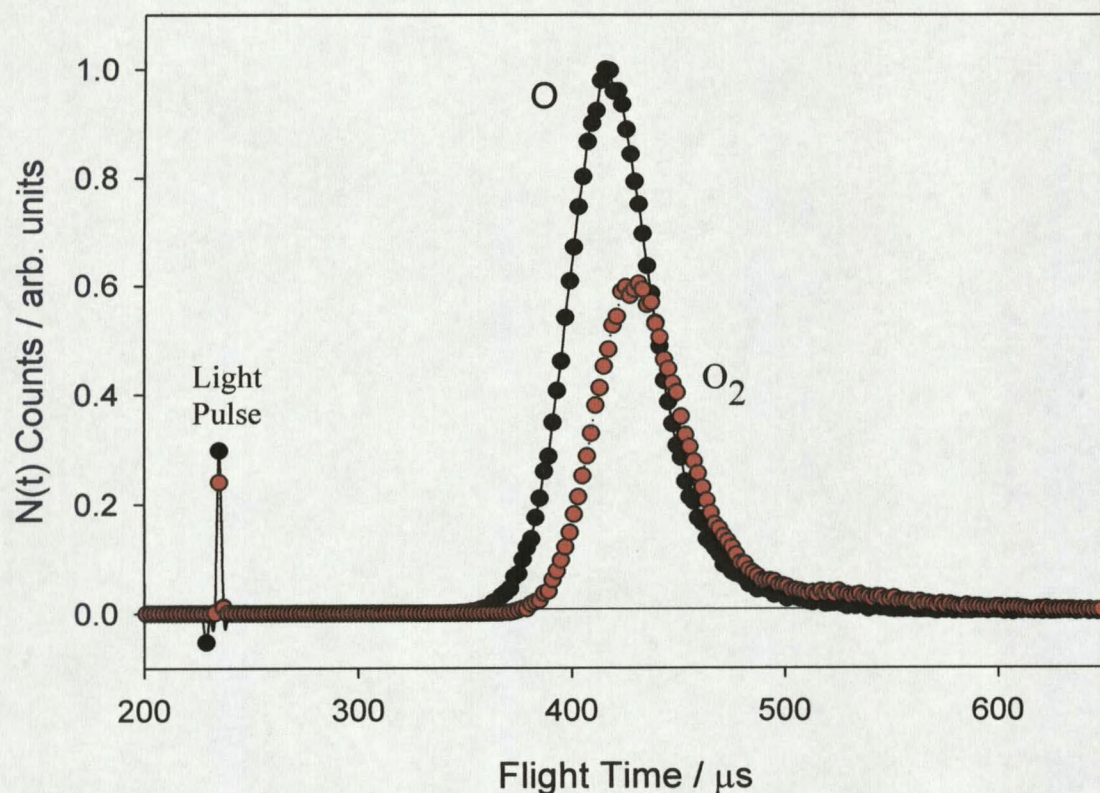


Figure 5. Time-of-flight (TOF) distribution of the atomic oxygen and molecular oxygen of the beam at the detector.

concave gold mirror 50 cm away into the nozzle where a plasma is formed with temperatures greater than 20,000 K. The conical nozzle has an interior angle of 20° and it establishes the direction of the beam which consists primarily of fast neutral O atoms and O_2 molecules with a ratio of 6:4, respectively, and a small ion content ($\ll 1\%$). The characterization of the O-atom beam was done to determine the electronic state of the oxygen atoms by Garton *et al.*²⁹ who showed that the O atoms are in the ground $O(^3P)$ state. In another similar study, these same researchers concluded that the molecular oxygen in the hyperthermal beam is in the ground $O_2(^3\Sigma_g^-)$ state.³⁰ These results are important because they establish that the exposure environment used in the laboratory subjects materials to ground state

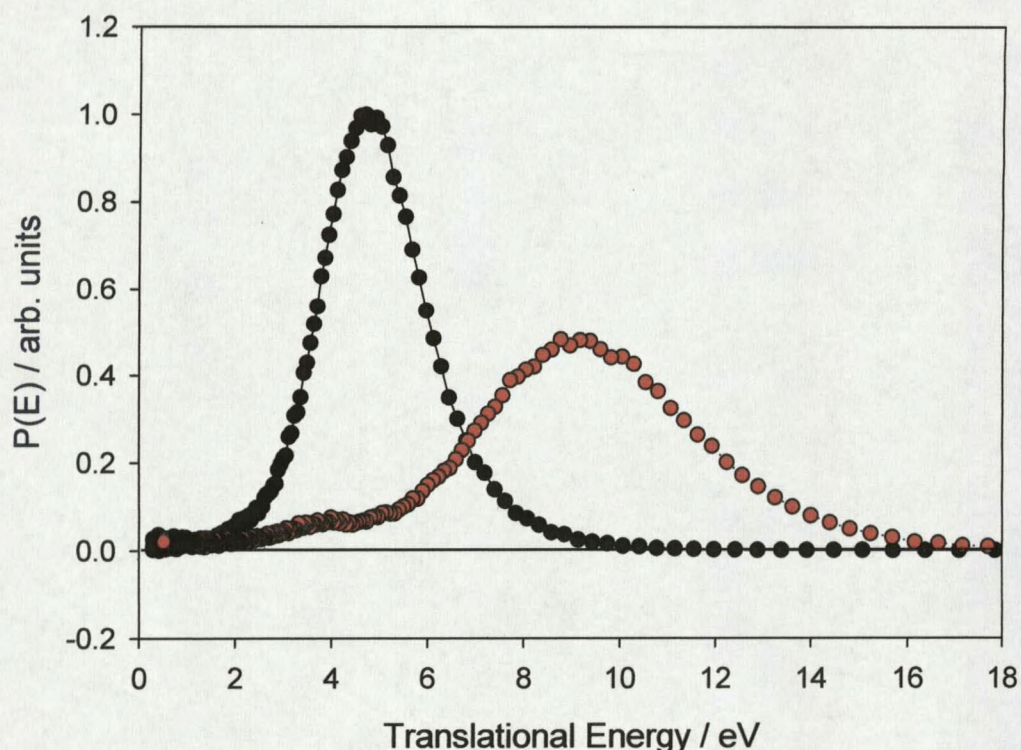


Figure 6. Translational energy distribution of the atomic oxygen and molecular oxygen of the beam.

O and O₂, as do space exposures. The average kinetic energy of the O atoms in the beam ranged from 4-6 eV with an energy distribution (FWHM) of ~2.5 eV.

The samples were exposed in the source region. A small portion of the O-atom beam passed through a skimming aperture before entering the main chamber.²⁷ The O-atom beam was then measured by a rotatable quadrupole mass spectrometer detector. The detector consists of a Brink-type electron bombardment ionizer, a quadrupole mass filter, and a Daly ion counter.²⁵ When the detector is positioned along the atomic oxygen beam axis, the O/O₂ time-of-flight can be measured. Figure 5 shows TOF distributions of O/O₂ distribution; 61% of the beam is atomic oxygen. The translational energy of the O-atom beam is determined by measuring the TOF distribution of the beam as it travels from the

conical nozzle to the detector (132.66 cm). Each pulse of the beam contains an average of 1.75×10^{15} O atoms with a mean translational energy of 5 eV and a flux of 1.6×10^{15} atoms $\text{cm}^{-2} \text{s}^{-1}$.^{1,9} The flux is measured at the sample mount and is calculated by measuring the erosion depth of a Kapton H standard that is assumed to have an erosion yield of 3.00×10^{-24} $\text{cm}^3 \text{atom}^{-2}$. The average beam velocity is 8 km s^{-1} . Figure 6 shows the translational energy distribution of atomic and molecular oxygen in the beam.

Sample Mount

The polymers are placed in a sample mount located 40 cm from the conical nozzle, a few degrees off the hyperthermal beam axis. The mount is exposed to the entire beam at a rate of two pulses per second.²² Polymer samples 0.48 inch in diameter and 0.005 inch thick were placed in the sample mount, with a stainless steel wire mesh placed over them. The wire mesh covers only a portion of the sample, allowing the erosion depth between the exposed and unexposed areas of the sample to be measured. The wire mesh is approximately 100 μm thick, and wire width of 1.22 cm and an open square area of 0.25 mm^2 . The sample holder contains nine locations for the positioning of materials. The sample mount is shown in Figure 7. A Kapton H reference standard is placed in position 5. The reference sample is thermally isolated from the rest of the sample mount and is maintained at 296 K. The temperature of the samples at the other eight positions can be elevated from 298 K to 573 K by resistive heaters embedded in the sample mount.

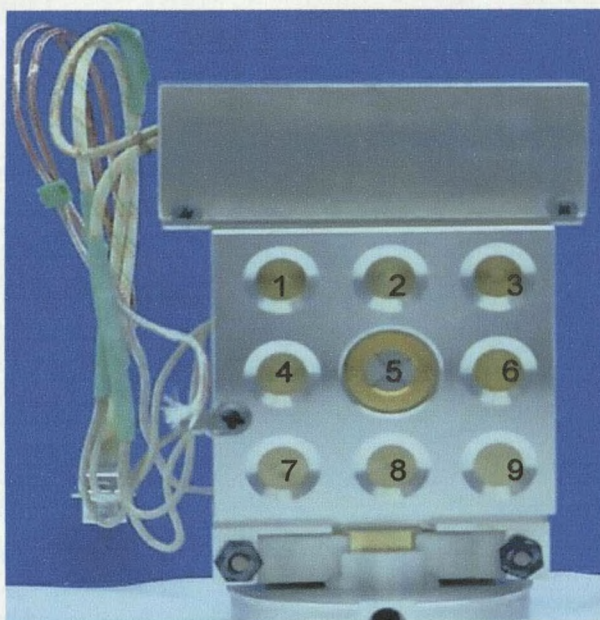


Figure 7. Sample mount and sample position numbering scheme

Polymer Surface Preparation

Each sample was cleaned with a 3:1 mixture of trichloroethylene and ethanol, respectively, and then air dried in a clean hood. A Kapton H reference standard was placed in position 5 of the mount for each exposure. Other Kapton H samples, which were exposed at various temperatures, were bonded to silicon wafers to ensure good temperature control of the polymer films. The adhesive was a silver-filled polyimide (Ablebond® 71-1). It was dried for four days under a watch glass and heat cured under vacuum for 30 minutes at 323 K, after which the curing temperature was slowly ramped up at a rate of 5 K min⁻¹ until it reached 473 K, where it remained for 30 minutes. The temperature was then increased at

the same rate to 573 K and held for 30 minutes. The FEP Teflon samples were prepared for adhesion to silicon wafers by applying Tetraetch® to one side, cleaning with hot nanopure water, and allowing the sample to dry in the clean hood. The purpose of Tetraetch® is to chemically alter one side of FEP Teflon to aid in adhesion. Ablefilm 5025® was then used to bond the Teflon coupon to the silicon wafer. The samples were heat-cured in air at 323 K for 30 minutes, then the temperature was slowly ramped at a rate of 1 K min^{-1} , finally curing at 423 K for one hour. It is important to heat cure FEP Teflon in air, because under vacuum the adhesive bonding properties may be affected. The polymer films, bonded to silicon substrates, are ready for exposure once the curing process is completed. Some samples remained unexposed and served as control samples.

Maintaining uniform surface temperature of a polymer sample has been a problem in past temperature dependence studies. This occurs because Kapton H and FEP Teflon are both good insulators and by placing them in a mount secured only around the outside, uniform temperature was not possible. However, by bonding polymer films to a silicon wafer, uniform temperature can be maintained over the entire surface. This approach was tested by exposing two Kapton samples at 573 K for 100,000 pulses. One sample was attached to a silicon wafer and one was not. When compared to the Kapton H reference standard, which was maintained at 296 K, the erosion depths were different between the surfaces. The Kapton film that was bonded to a silicon wafer eroded $14.5\text{ }\mu\text{m}$, while a free-standing Kapton film mounted in the heated sample mount eroded $\sim 11.7\text{ }\mu\text{m}$. The reference sample in a mount held at 296 K eroded only $4.6\text{ }\mu\text{m}$. The reason for the discrepancy

between the two heated samples is Kapton on silicon had an even distribution of temperature; however, the unsecured sample temperature could only be elevated around the edge where it was held in the sample mount. This unbonded film evidently never reached the temperature of the mount, as the bonded sample did.

Surface Characterizing Methods

Material Loss Measurements

Kapton H (pyromellitic dianhydride-oxydianiline (PMDA-ODA) polyimide) is a standard polymer reference used by the space effects community. It has an accepted erosion yield of $3.00 \times 10^{-24} \text{ cm}^3 \text{ atom}^{-1}$ caused by 5 eV $\text{O}(^3P)$ atoms.^{3,31} Kapton is used as a witness specimen to determine the effective O-atom fluence of an exposure. The erosion yield, R_e , is the average volume of polymer lost due to interactions with one oxygen atom impinging on its surface.^{16,19} The material erosion yield is calculated by dividing the erosion depth by the fluence (total number of incident O-atoms):

$$R_e = \frac{\text{measured erosion depth}}{\text{fluence}} \quad (\text{cm}^3 \text{ atom}^{-1})$$

The Kapton-equivalent fluence of an experiment can be thought of as the integrated O-atom flux. The fluence is found by measuring the erosion depth and dividing it by Kapton's accepted erosion yield. Typical exposure fluences are on the order of $10^{20} \text{ atoms cm}^{-2}$. The experimental flux is determined by dividing the fluence by the exposure duration. Flux is approximately $1.5 \times 10^{15} \text{ O atoms cm}^{-2} \text{ s}^{-1}$ at the sample mount. The fluence and flux in our

laboratory are on the same order of magnitude as in the LEO environment. The Kapton reference standard, when held at room temperature, is believed to have a linear relationship between the erosion depth and fluence.

A wire etched mesh placed over test samples allows for direct measurement of exposed and unexposed surfaces on the same sample with the use of profilometry. The DEKTAK³ (Veeco Metrology Group, Santa Barbara, CA) is an instrument that measures step height from 100 Å to 50 µm.³² The instrument uses a diamond-like stylus to make measurements by electromechanically moving the sample. When a scan is in progress, the stage moves the sample, causing the stylus to drag along the surface. Any height variation causes the stylus to move vertically. There is an electrical signal produced corresponding to the stylus movement. The analog signal proportional to the position change is converted into a digital format. The digital signal from a scan is stored in the computer's memory for display, manipulation, measurement, and print.³² All sample scan lengths varied from 2 to 4 microns on slow to medium speeds, with average step heights calculated from 40-45 different measurements for each sample. Figure 8 shows the arrangement of the sample and etched mesh with respect to the hyperthermal beam, and figure 9 shows representative photographs and profiles of FEP and Kapton H.

Surface Topography

Atomic force microscopy (AFM) can measure the change in surface topography that results from atomic oxygen attack.³³ In general, the AFM obtains data from the interac-

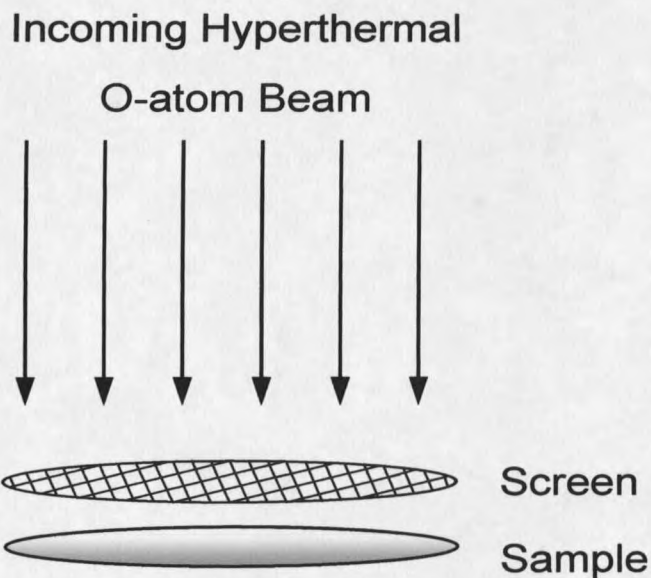


Figure 8. Schematic diagram of hyperthermal beam bombarding the sample surface through the etched mesh screen.

tion of a probe tip with the atoms on the sample as the tip scans over the surface. This information is then used to construct an image of the surface topography.³⁴ AFM studies were performed using a Nanoscope IIIa Tapping Mode-AFM (TM-AFM) (Digital Instruments, Santa Barbara, CA). Due to its non-destructive nature, TM-AFM has become a standard method used to study surface changes. With TM-AFM, the oscillating probe only contacts the surface for a short period of time.³⁵ A cantilever tip, oscillating vertically with an amplitude range of 20 to 100 nm, "taps" the surface during scanning; a feedback loop maintains constant oscillating amplitude to keep the tip-to-sample interaction constant. The movement of the cantilever is detected by a laser. When the laser bounces off the cantilever position, the light reflected onto the photo-detector shifts, providing a signal that is con-

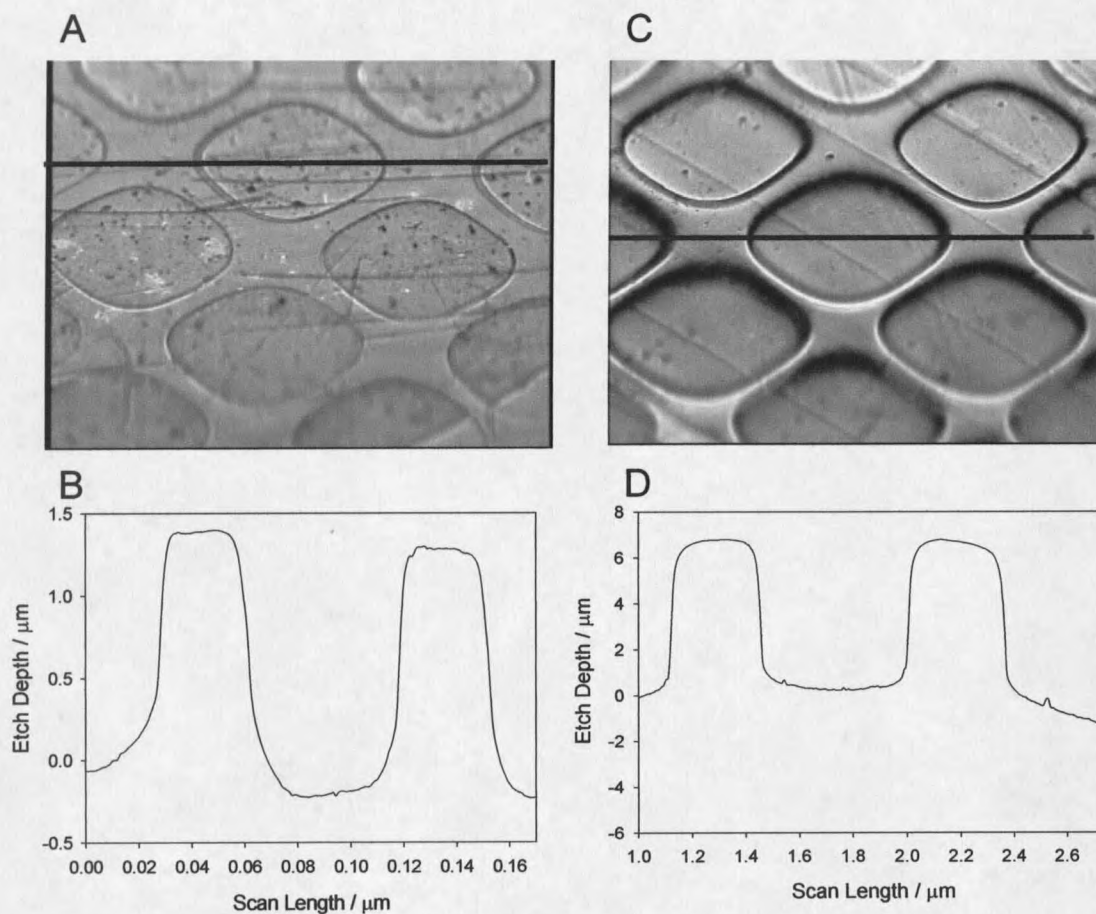


Figure 9. A) Photograph of FEP Teflon surface that was etched at 423 K by 100,000 O-atom beam pulses, B) Dektak³ profile of this exposed of FEP Teflon surface, C) photograph of Kapton H surface that was exposed at 423 K by 100,000 O-atom beam pulses, D) Dektak³ profile of Kapton H surface.

verted into a voltage.³⁴ The voltage signal from the photodiode is transformed into a topographical image of the sample surface.³⁶ A schematic of TM-AFM is illustrated in figure 10.

Surface roughness parameters are used to quantify changes in the surface topography. The root mean square of the surface roughness (R_q) measured by the Nanoscope IIIa

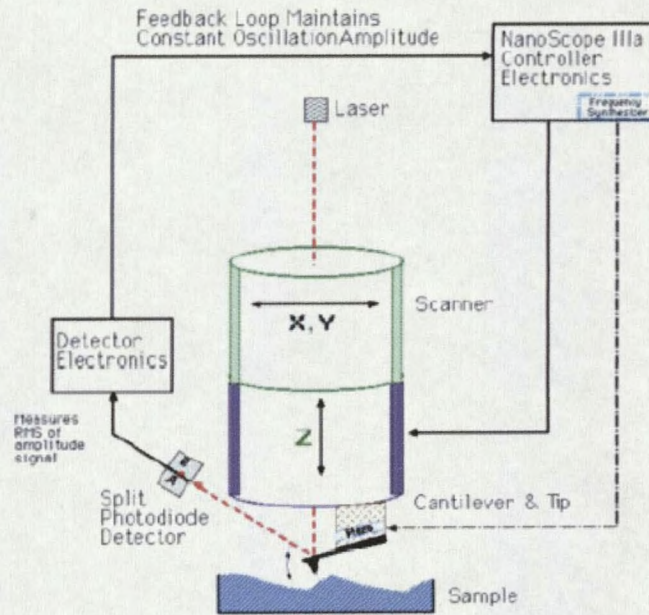


Figure 10. Schematic diagram of the major components of an AFM showing the feedback loop for TappingMode™ operation. (Reprinted from Digital Instruments AFM: Complementary Technique for High Resolution Surface Investigations).

software is the standard deviation of the Z values within the given area and is calculated by^{34,37}:

$$R_q = \sqrt{\int \int_a (Z(x, y))^2 dx dy}$$

The mean surface roughness (R_a) is the arithmetic mean value relative to the center plane of the surface and is calculated by:

$$R_a = \int \int_a |Z(x, y) dx dy|$$

The R_q and R_a values of the surface roughness are similar. R_q is the root mean square of the change in height prior to being integrated over the peaks and valleys of the surface. Therefore, peaks and valleys that have similar heights and depths are indistinguishable from one

another. R_a quantifies the magnitude of the surface heights but is not affected by the distribution of height changes. Unfortunately, both R_q and R_a can be ambiguous because surfaces composed of features of different sizes and shapes produce similar roughness measurements. However, R_q and R_a provide useful numbers to characterize relative roughness of similar surfaces.

CHAPTER 3

RESULTS OF KAPTON STUDY

Erosion Study of Kapton H Reference

The linearity of Kapton H erosion at room temperature was studied. The erosion depth of the Kapton H reference is plotted in figure 11 as a function of the exposure duration, which ranged between 28,000 pulses (~4 hours) and 250,000 pulses (~35 hours). The fluence of the O-atom beam increased from 3.68×10^{19} to 4.11×10^{20} O atom cm^{-2} , and the average flux was 1.67×10^{15} O-atom cm^{-2} pulse $^{-1}$. These data were collected over a two year period, totaling 2,800,227 beam pulses and ~389 hours of exposures. Within the fluence uncertainties of the various exposures, erosion appears to increase linearly with the exposure duration. Therefore, a linear regression of $y = -0.31 + 0.052x$ provided the best fit for all data points in figure 11. It is interesting to note that the erosion depth does not have a y-intercept equal to zero. This may suggest that there is an induction period before erosion starts to occur. In addition to the long-term study of the fluence dependence of Kapton erosion, a more focused study was conducted during a three-week period when the O-atom beam was particularly stable. The data from this period are shown as red points in figure 11. These data were fit slightly better with a polynomial $y = 1.14 \times 10^{-4}x^2 + 0.03x + 0.22$ than with a simple linear regression, suggesting that there might be a tiny non-linearity in the dependence of Kapton erosion depth on O-atom fluence.

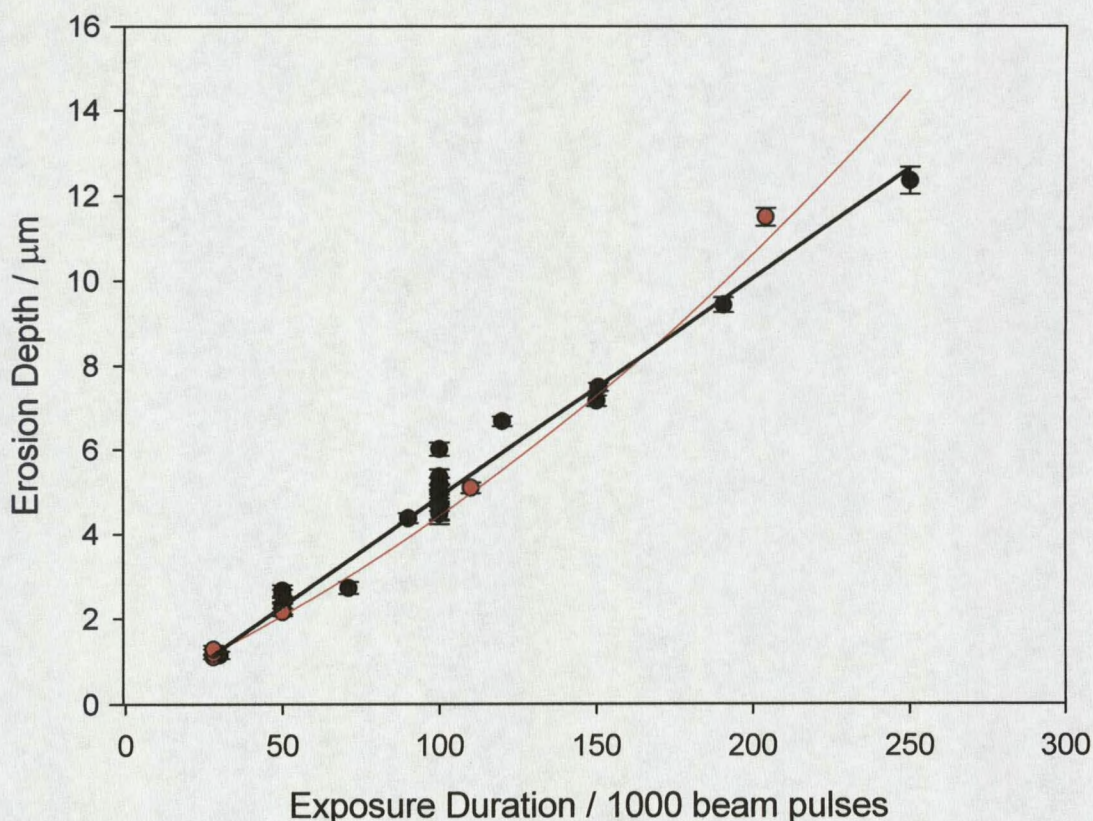


Figure 11. Erosion of Kapton H as a function of exposure duration. The black points represent all data collected and the red points represent data collected over a three week period during which the O-atom beam was particularly stable.

Temperature Dependence of Kapton H Erosion

The steady state temperature dependence of Kapton H erosion was studied in the range 298 K to 573 K. At each temperature used, Kapton H samples were placed in positions #4 and #7 of the sample mount and exposed to 100,000 pulses of the hyperthermal beam. The specific temperatures used were 298 K, 333 K, 373 K, 423 K, 498 K, and 573 K, and the average flux of the beam for each exposure was 1.67×10^{15} O-atom cm^{-2} pulse $^{-1}$. For every exposure, a Kapton H reference sample was located in position #5 of the mount and

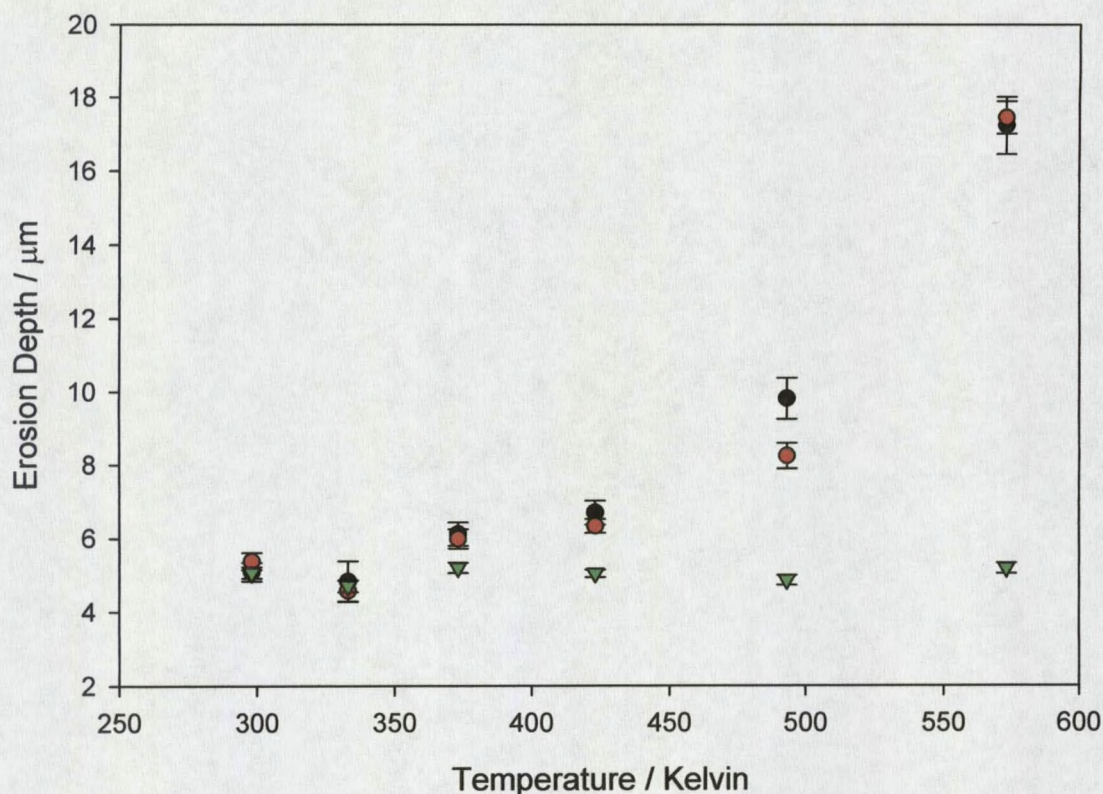


Figure 12. Erosion depth of Kapton H as a function of temperature. The black points represent position #7, the red points represent position #4, and the green triangles represent the Kapton H reference sample (maintained at 298 K).

maintained at a constant temperature (298 K). The erosion depths are plotted as a function of temperature in figure 12. When comparing the erosion of the Kapton reference sample to that of the elevated temperature samples, it is apparent that Kapton's erosion increases with temperature. The erosion depths of the elevated temperature samples were divided by the erosion depths of the corresponding Kapton H reference samples. This ratio is plotted as a function of inverse temperature in figure 13a. In figure 13b, the upper and lower limits of the Kapton erosion ratio were established utilizing the standard deviation of the erosion

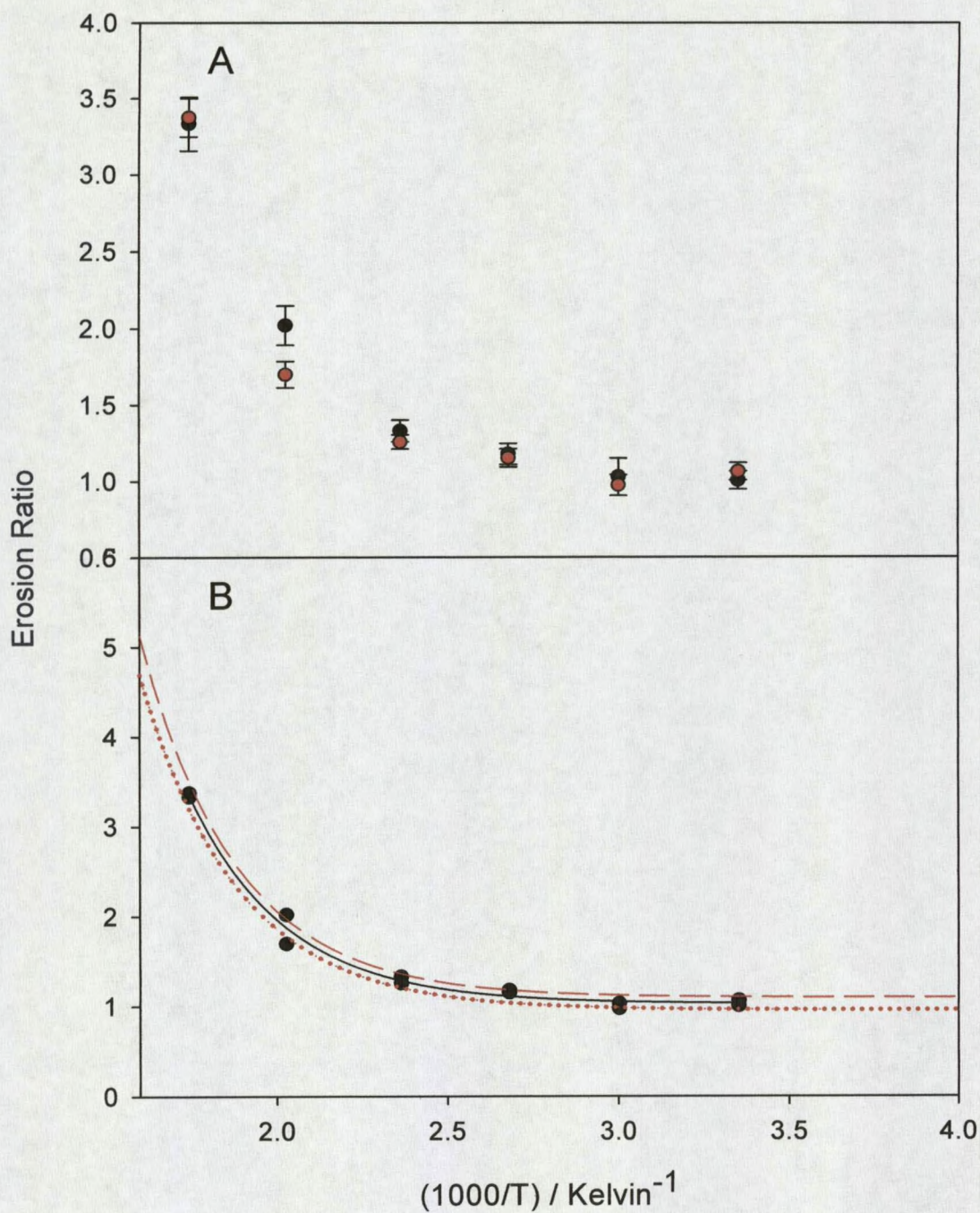


Figure 13. A) The Kapton erosion ratio as a function of inverse temperature. The black points represent position #7, and the red points represent position #4. B) The upper and lower limits of the Kapton erosion ratio. The black line represents a curve fit to the experimental data, the red long dash line represents the upper limit, and the red short dash line represents the lower limits.

ratio. An equation that fits the experimental data very well is $y = 1137\exp(-3.6x) + 1.03$, with the generic equation form of $y = A\exp(-Bx) + C$. The variation in the upper and lower limits for the B term is ± 0.014 . This functional form resembles an Arrhenius term with a constant added. An Arrhenius term has the form $k = A\exp(-E_a/RT)$, where k is the reaction rate constant (proportional to our erosion ratio), A is a pre-exponential factor, E_a is the activation energy, R is the gas constant, and T is the equilibrium temperature at which the reaction occurs. The constant must be added to the Arrhenius term, because the erosion ratio reaches a nearly constant value of one at the lowest temperature used. The success of the two-term function in fitting the data suggests that two basic mechanisms contribute to

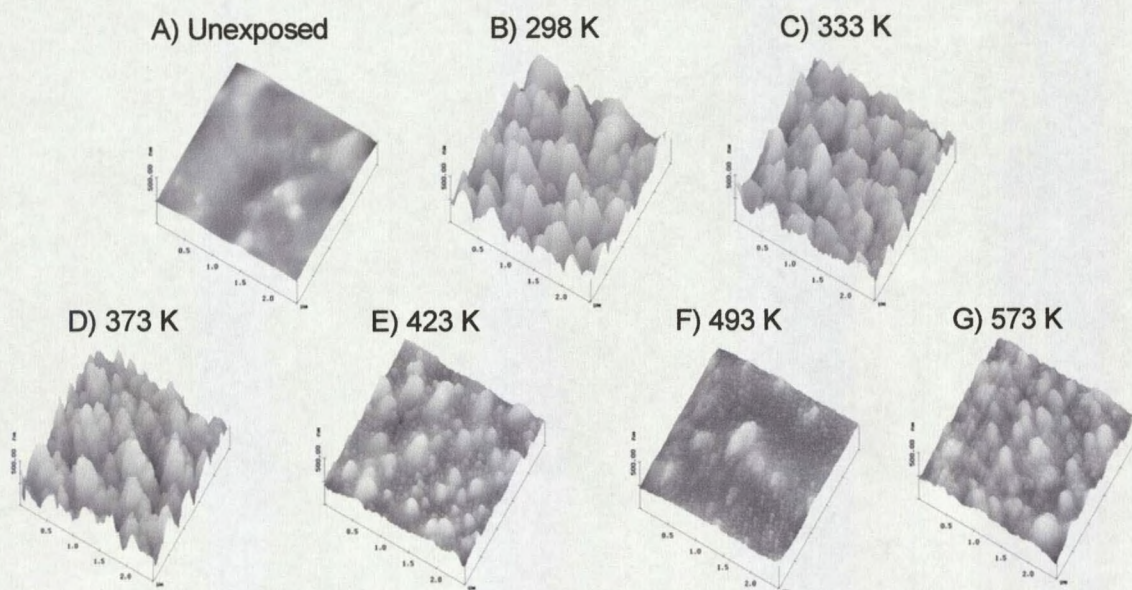


Figure 14. AFM images of unexposed (A) and exposed (B-G) Kapton H. All images are $2.5\ \mu\text{m} \times 2.5\ \mu\text{m}$ and have a Z scale of 500 nm.

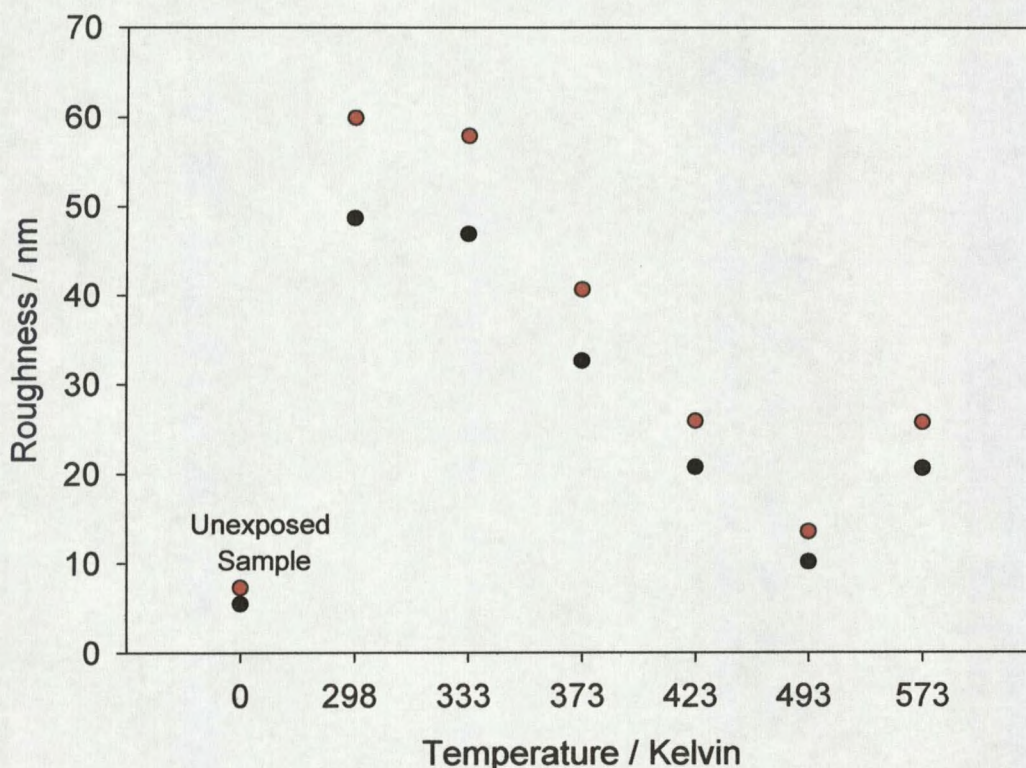


Figure 15. Surface roughness measurements of Kapton H. The black points represent R_q values and the red points represent the R_a values.

the reactivity of Kapton H with hyperthermal atomic oxygen. One mechanism is temperature dependent and has an activation energy, $E_a = 3.6 R \times 1000 = 29.5 \text{ kJ mol}^{-1}$ (0.31 eV), and the other mechanism is temperature independent (effective activation energy of zero).

The surface morphology of the Kapton H samples was examined by TM-AFM. All images were measured with the same area, $2.5 \mu\text{m} \times 2.5 \mu\text{m}$, and a Z scale of 500 nm; the R_q and R_a were measured by the Nanoscope IIIa software. Figure 14 shows an AFM image of a Kapton H control sample (unexposed), as well as images of Kapton H samples that were exposed at temperatures of 298 K, 333 K, 373 K, 423 K, 493 K, and 573 K. Before the exposure, the Kapton surface was smooth as illustrated in figure 14a with a roughness (R_q)

Figure 14	Temperature / Kelvin	R _a / nm	R _q / nm
A	Unexposed	5.483	7.283
B	298 K	48.686	59.918
C	333 K	46.91	57.888
D	373 K	32.619	40.605
E	423 K	20.729	25.878
F	493 K	10.146	13.526
G	573 K	20.605	25.713

Table 1. Surface roughness measurements of figure 14. The first column lists the corresponding image for each measurement.

of ~7 nm. The exposed polymers initially experienced an increase in surface roughness as the temperature increased. The erosion of Kapton was accompanied by the development of well defined “shag-carpet” features on the etched surface. It has been established that the carpet-like surface structures are formed when Kapton is exposed to an energetic atomic oxygen beam, leading to degradation of physical properties, i.e., optical, thermal, electrical, and mechanical properties.^{15,20} As the temperature increased further, the surface roughness decreased until the highest exposure temperature of 573 K, where the surface started to become rough again with the development of additional needle-like structures. The surface roughness measurements are listed in table 1 and illustrated in figure 15. The roughness measurements are consistent with the visual change in surface morphology.

CHAPTER 4

DISCUSSION OF KAPTON H STUDY

Kapton Erosion Study

Kapton H is used as a reference standard to determine total fluence and average flux of an experiment. The relationship between erosion and fluence is important to the study of polymer erosion in simulated LEO environments. If the reference standard does not have a linear erosion at higher fluences then it is important to know the functional dependence of Kapton erosion on fluence. Previously, it had been assumed that Kapton erosion was linear but it had not been quantified or reported. When the hyperthermal beam was studied over a three week period, Kapton eroded slightly non-linearly with fluence. These data (figure 11, red points) suggested that the fluence dependence might be slightly non-linear, but this non-linearity is so small that it can only be observed when the beam is operating very reproducibly. In general, practical variations in the exposure fluence are large enough to mask the slight non-linearity in the fluence dependence.

The plot of erosion depth versus exposure fluence had a y -intercept equal -0.31 instead of zero. This suggests that there is an oxidation induction period before the surface starts losing mass. Kinoshita *et al.*³⁷ studied the *in situ* mass loss of polyimide films undergoing exposure to a hyperthermal atomic oxygen beam. The mass of the polyimide film was measured from shifts in the resonance frequency of a quartz crystal microbalance (QCM).

They reported that the mass of the polyimide film first increased as a function of O-atom fluence and then started to decrease. XPS data showed an increase in surface oxygen content due to oxygen incorporation into the surface. They concluded that the initial mass increases when O-atoms attack a pristine polyimide surface until the surface becomes oxidized, at which time it begins to lose mass. These data are consistent with the observed y-intercept point. When Kapton H is initially exposed to O-atoms, the surface becomes oxidized until mass loss occurs from continued reactions of oxygen atoms being absorbed on the surface and products are released.

Temperature Dependence of Kapton H Erosion

The Kapton H erosion depth has a temperature dependence that grows exponentially as the temperature is increased, shown in figure 12. The rapid increase in erosion depth as a function of temperature appears to continue as the temperature is elevated. Unfortunately, due to limitations of the sample mount, this study had a maximum temperature of 573 K. The erosion ratio emphasizes the temperature dependence, as illustrated in figure 13a. The exposure temperature increased the erosion ratio between the experimental sample and the Kapton reference from a 1:1 ratio at 298 K to 3.3:1 at 573 K.

The erosion of Kapton had a strong temperature dependence. These observations contradict the temperature study by Tagawa *et al.*²² of a Kapton-like polymer. However, they studied the mass loss rate of the polymer over a relatively narrow temperature range.

They concluded that the polyimide film had a linear Arrhenius relationship and found the erosion activation energy to be $0.055 \text{ kJ mol}^{-1}$ ($5.7 \times 10^{-4} \text{ eV}$) for the temperature range of 253 K to 353 K. This measurement employed a QCM and data were obtained during the initial erosion period of the polymer. Although Tagawa's activation energy is significantly lower than found in this study, it was in good agreement when our curve fit was extrapolated to 253 K from 298 K. Since they only studied the erosion of a Kapton-like polymer over a relatively narrow range of temperatures, where we observed very little temperature dependence, it is understandable how they obtained a lower activation energy from a linear Arrhenius plot. At higher temperatures there is a deviation from this near temperature-independent behavior, suggesting that more than one mechanism is involved in the erosion of Kapton.

The curve fit of figure 13 ($y = A \exp[(-E_a/R)(1/T)] + C$) suggests that the loss of surface material appears to be the result of temperature-dependent and -independent mechanisms. The constant term (C) results from a mechanism ("mechanism 1") in which the effective activation energy approaches zero. The mechanism likely involves the direct reaction of O atoms with the surface on a time scale too short for thermal equilibrium to be achieved. The direct reactions of 5 eV O atoms with the surface should be able to overcome even a significant reaction barrier, so the reaction would proceed independently of surface temperature and yield an effective E_a of zero. For example, hydrogen abstraction has a barrier of $\sim 0.25 \text{ eV}$ which is small compared with the high energy of the incoming atomic oxygen. This research does not permit a conclusion about the nature of the temperature-

independent mechanism, but we can speculate that H-atom abstraction might be an initiation step in the degradation of polymers, based on previous work in this laboratory by Zhang *et al.*¹¹, who showed that direct H-atom abstraction is the dominant initial reaction when a hyperthermal O-atom strikes a polymer surface.

The first term in the function that fit the erosion ratio data is temperature dependent, and this term becomes important in the fit to the erosion data at higher temperatures. This dependence suggests a mechanism ("mechanism 2") that takes place in thermal equilibrium with the surface, implying that in order for this mechanism to occur, O atoms must transfer their energy to the surface and become trapped before the rate limiting erosion reaction occurs. Trapping becomes more likely on rough surfaces, which allow for multiple bounces at the surface that drive incident atoms toward thermal equilibrium. In the studies reported here, and in any situation where macroscopic amounts of material are removed by highly directional O atoms, the surface quickly reaches a steady-state roughness. Therefore, the conclusion of a mechanism that depends on thermal accommodation of incident O atoms is consistent with a steady-state erosion of rough surfaces. Thermal accommodation would be expected to be much less on smooth surfaces, possibly reducing the importance of the thermal mechanism and thus the temperature dependence of the erosion.

The surface roughness measurements from the AFM images support the conclusion that two basic mechanisms control the erosion of Kapton H. At lower temperatures, it appears that "mechanism 1" dominates, where the surface is bombarded with atomic oxygen and rough, shag-carpet-like features develop. This is shown in figure 14b-d. As tem-

perature increases, the surface becomes smoother as "mechanism 2" begins to take over, or at least dominate the reactions occurring on the surface. This is evident in figure 14e-f where the surface appears to become smooth for temperatures between 373 K and 493 K. The reason why this occurs is not clear. A combination of increased erosion rate and temperature increases the amount of material removed, possibly resulting in a smoother surface. As the erosion rate and temperature are further increased, the surface may become rougher due to more erosion occurring. However, the roughness at higher temperatures looks different from roughness at lower temperatures; this could suggest that "mechanism 1" leads to a different type of roughness compared to "mechanism 2".

Matveev *et al.*³⁸ studied the room temperature erosion of Kapton H after exposure to a 2-4 eV atomic oxygen beam. They observed that the "shag-carpet" looked like mushroom-shaped cylinders. They suggested that this material was more resistant to atomic oxygen attack, forming a protective cap on the needle-like structures. But at higher temperatures, where the thermal mechanism ("mechanism 2") takes over (or at least dominates), one would expect more undercutting of the protected areas, and as a result, the surface might appear smoother. As the temperature is raised, the large features suggest that there could again be some kind of protection developing on top of the cylinders.

Although the mechanism is still unknown, more information regarding what is occurring on the surface has become clear. It has been determined that Kapton erosion has a temperature dependence. At temperatures less than 373 K, loss of material does not depend on the surface temperature; however, when the temperature increases higher than 373 K,

material loss becomes strongly dependent on temperature. Two basic mechanisms are evident. The dominant mechanism at lower temperatures has an effective activation energy of zero and is likely the result of a direct (non-thermal) gas-surface interaction. The dominant mechanism at higher temperatures has an activation energy of 29.5 kJ mol^{-1} (0.31 eV) and corresponds to a rate-limiting reaction (or set of reactions) that occur in thermal equilibrium with the surface. Because of its high activation energy, this second mechanism plays only a minor role in the erosion of Kapton H at temperatures near room temperature.

CHAPTER 5

RESULTS OF FEP TEFLON TEMPERATURE DEPENDENCE STUDY

The steady-state temperature dependence of FEP Teflon erosion was studied. The polymer was exposed to 100,000 hyperthermal beam pulses at temperatures of 298 K, 333 K, 373 K, and 423 K. The average flux was 1.58×10^{15} O-atom cm^{-2} pulse $^{-1}$. The FEP Teflon samples were placed in position #8 and #9 of the sample mount, and the Kapton H reference sample was located in position #5, where it was maintained at a constant temperature (298 K). The FEP Teflon erosion depth is plotted as a function of increasing temperature in

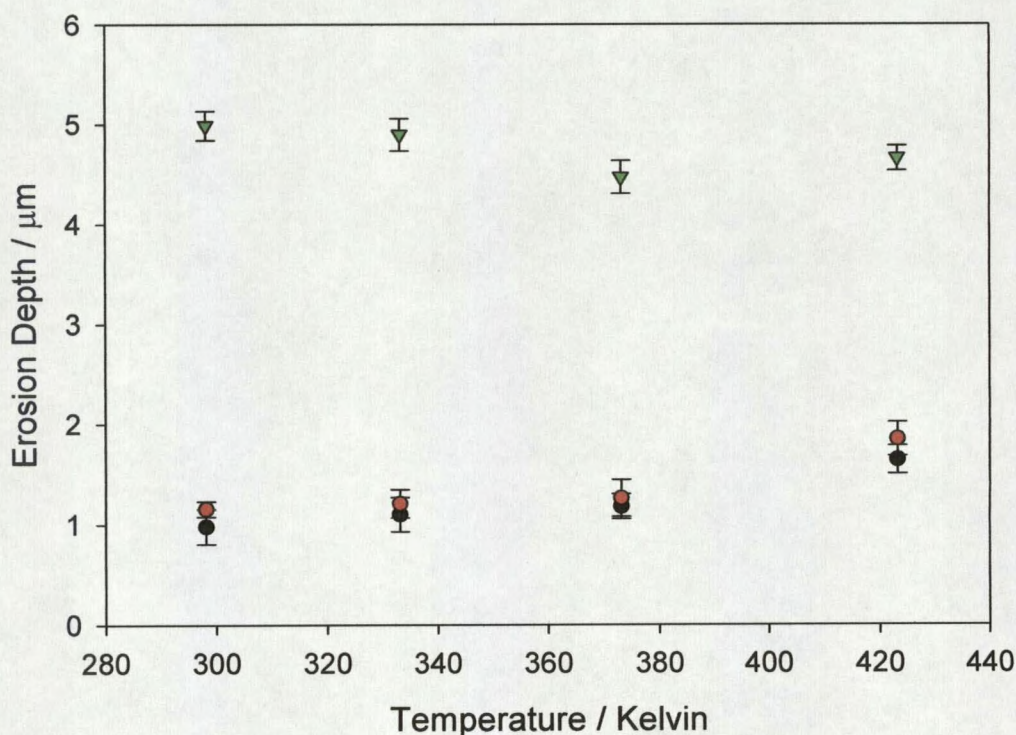


Figure 16. Erosion depth of FEP Teflon as a function of temperature. The black points represent position #8, the red points represent position #9, and the green triangles represent the Kapton H reference sample (maintained at 298 K).

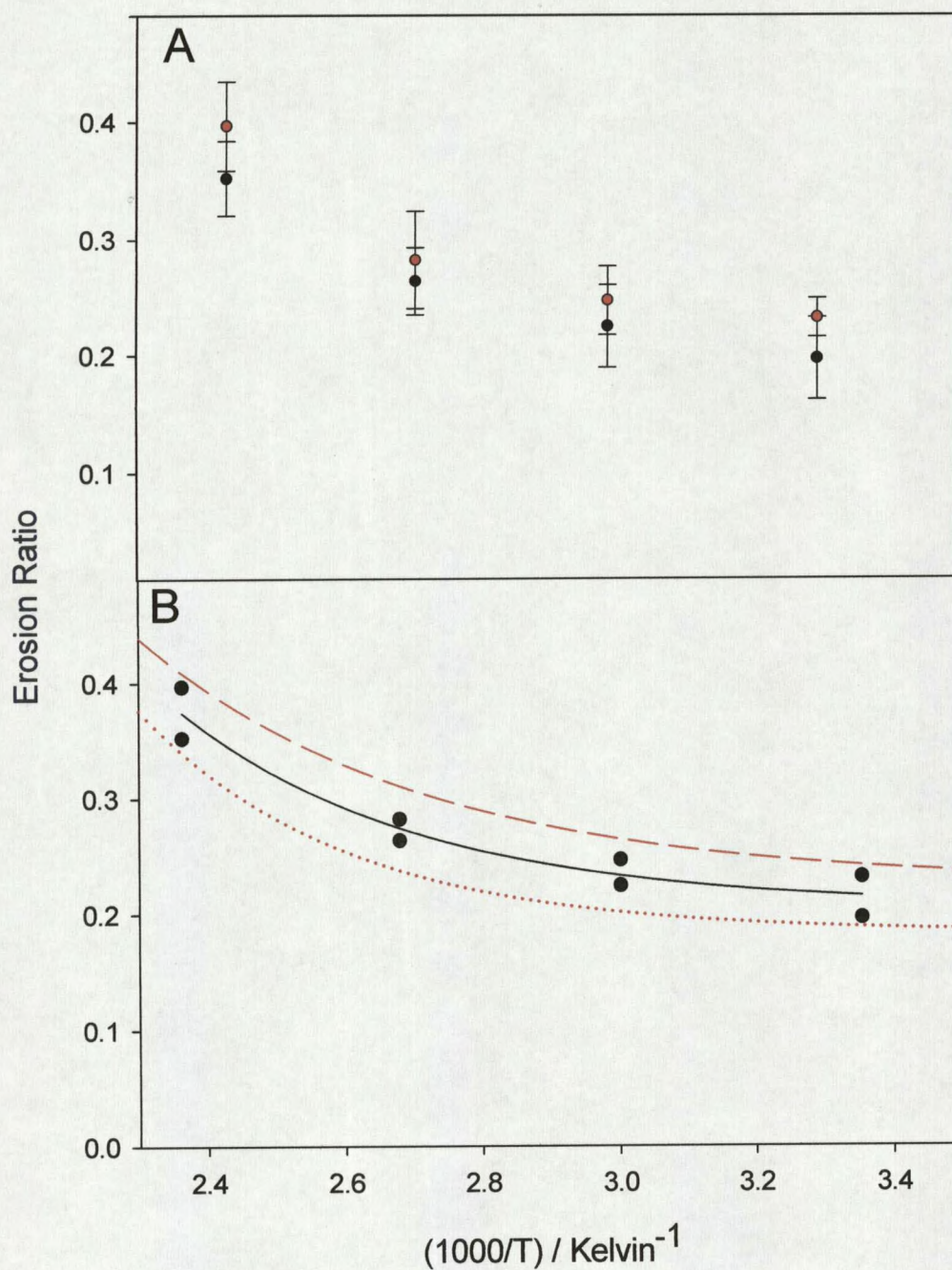


Figure 17. A) The erosion ratio of FEP Teflon to Kapton H as a function of inverse temperature. The black points represent position #8; the red points represent position #9. B) Curves fitted to the inverse temperature dependence of the FEP Teflon/Kapton H erosion ratio. The black line represents a curve fit to the experimental data. The area between the red long-dash line and the red short-dash line represents the range of uncertainty in the functional dependence of the erosion ratio of an inverse temperature.

figure 16. The erosion depth of each FEP Teflon sample was compared to the respective Kapton reference sample for each exposure. There appears to be a small temperature dependence of FEP Teflon erosion compared to the reference sample erosion. An erosion ratio was determined by dividing the erosion depth of FEP Teflon by that of the Kapton reference sample for each exposure. When this ratio is plotted as a function of inverse temperature (figure 17a), an exponential functional dependence is observed. The change was well fit by Sigma Plot (version 8) with the equation, $y = 1.34 \times 10^2 \exp(-2.8x) + 0.21$, as shown in figure 17b. The upper and lower limits were established by the standard deviation of the ratio and are illustrated in figure 17b.

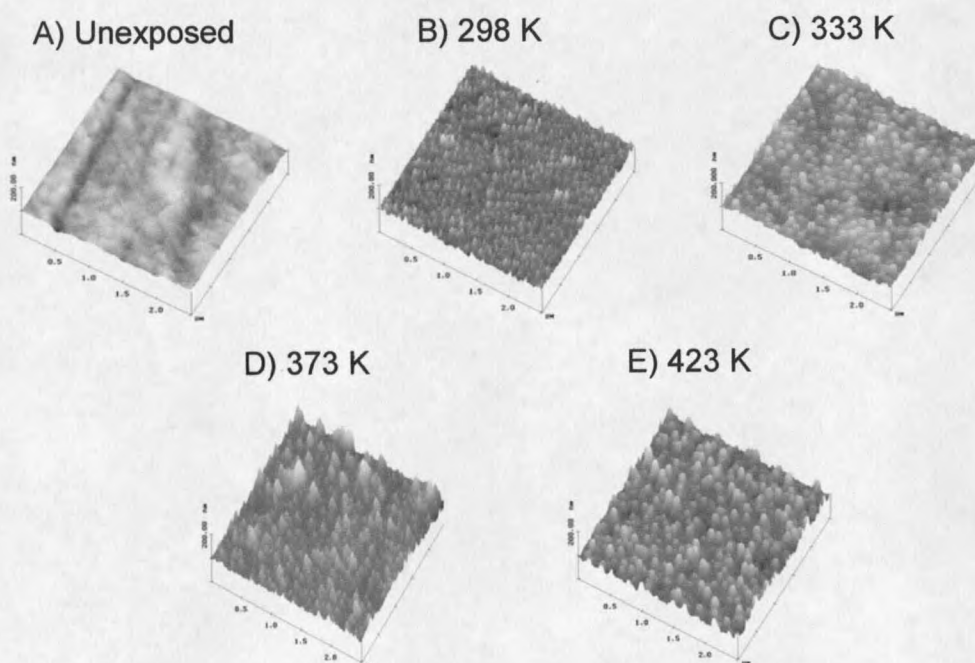


Figure 18. AFM images of unexposed (A) and exposed (B-E) FEP Teflon. All images are $2.5 \text{ mm} \times 2.5 \text{ mm}$ and have a Z scale of 200 nm.

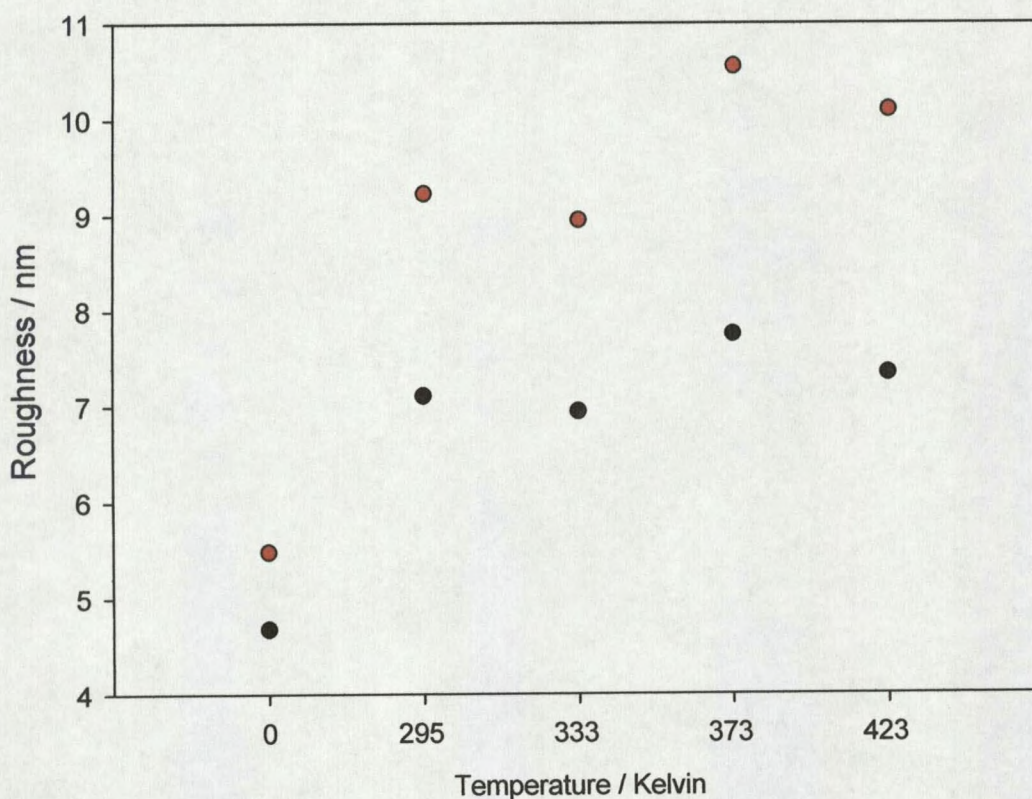


Figure 19. Surface roughness measurements of FEP Teflon. The black points represent R_q values and the red points represent the R_a values.

The FEP Teflon erosion rate can be described with a two-term Arrhenius-type function, similar to what was used to describe the temperature dependence of Kapton H. The constant term (0.21) is a manifestation of a reaction that is temperature independent, and the Arrhenius-like term can be used to describe an effective activation energy for a temperature-dependent reaction mechanism: $E_a = 2.8R \times 1000 = 23.5 \text{ kJ mol}^{-1}$ (0.24 eV).

AFM images of the surface morphology changes were obtained. All five images were measured with the same area, $2.5 \mu\text{m} \times 2.5 \mu\text{m}$, and a Z scale of 200 nm; the R_q and R_a were measured by the Nanoscope IIIa software. Figure 18 shows the AFM images of unexposed and exposed FEP Teflon at temperatures of 298 K, 333 K, 373 K, and 423 K. The R_q

Figure 18	Temperature / Kelvin	R_a / nm	R_q / nm
A	Unexposed	4.679	5.483
B	295 K	7.107	9.230
C	333 K	6.944	8.951
D	373 K	7.750	10.554
E	423 K	7.339	10.103

Table 2. Surface roughness measurements of Figure 18. The first column lists the corresponding image for each measurement.

surface roughness of unexposed FEP Teflon was ~5.4 nm. The erosion of FEP Teflon increased the development of the “shag-carpet” topography on the surface. As the sample temperature increased from 298 K to 333 K there was a small increase in surface roughness, but the roughness remained nearly constant as the sample exposure temperature was increased further. The surface roughness values are listed in table 2 and illustrated in figure 19.

CHAPTER 6

DISCUSSION OF FEP TEFLON TEMPERATURE DEPENDENCE STUDY

Although FEP Teflon eroded less than the Kapton reference, presumably due to the relatively small reactivity of a fluorinated hydrocarbon, its erosion depth grew exponentially as a function of temperature. The increase in erosion depth as the temperature was increased from 298 K to 423 K indicates that there is a dependence of the erosion rate of FEP Teflon on temperature. Due to the thermal stability of the polymer, 423 K was the highest temperature studied. The erosion ratio increased from 0.2 at 298K to ~0.4 at 423 K. The erosion ratio was curve fitted with the equation $y = A\exp(-Bx) + C$, where $A = 1.34 \times 10^2$, $B = 2.8$, and $C = 0.21$. The range of uncertainty was established by the standard deviation illustrated in figure 17b. The B term has an uncertainty of ± 0.40 and represents E_a/R . The activation energy for the temperature-dependent mechanism was 23.5 kJ mol⁻¹ (0.24 eV) for the erosion of FEP Teflon. The two-term fit to the temperature-dependent data (figure 17b) suggests the presence of two mechanisms, one temperature-dependent and the other temperature-independent with an activation energy approaching zero.

The temperature-independent term dominates at temperatures lower than approximately 373 K. Gindulytė *et al.*⁹ conducted *ab initio* calculations of fluorocarbons in which they reported that direct C-C bond breakage by O(³P) has an activation energy of ~2.9-3.2 eV. If this is the case, then atomic oxygen bombards the surface with enough energy to

overcome any reaction barrier imposed by the presence of fluorine, resulting in material carried away from the surface. The O-atom collisions, with no loss of translational energy, react quickly on the surface via a direct mechanism. This results in surface roughening. Other mechanisms might also break the C-F or C-C bonds.

The temperature-dependent erosion becomes apparent at temperatures higher than 423 K. This dependence suggests that the atomic oxygen reacts in thermal equilibrium with the polymer surface. When the oxygen atom hits the surface, it bounces between the "hillocks" formed and loses energy and becomes thermalized. As the surface temperature increases, the probability of reactions occurring between the thermal oxygen atoms and the surface increases.

The FEP Teflon erosion ratio did not increase as significantly as that of Kapton H when the surface temperature was raised. FEP Teflon has a lower activation energy than does Kapton H, indicating that the reaction rate is slightly less dependent on temperature. The temperature-independent mechanism involved in FEP Teflon erosion occurs with lower probability than the temperature-independent mechanism in Kapton H. Whatever these reaction mechanisms are, they still have barriers, even though they appear to be temperature-independent. That is because the temperature-independent reactions do not occur in thermal equilibrium with the surface, *i.e.*, they are direct reactions that depend on the center-of-mass collision energy of the incident atom within a localized region of the surface. So, the direct reactions still require a barrier to be overcome. It seems that FEP Teflon is less reactive than Kapton H because direct reactions with Kapton H require less energy

than direct reactions with FEP Teflon. Perhaps no direct reaction would occur with FEP Teflon if VUV light did not play a role in generating reactive sites. In any case, the baseline erosion rate of FEP Teflon is only about 20% that of Kapton H. As the temperature is increased, we see the involvement of a second reaction mechanism that has a high barrier, both in the case of FEP Teflon and Kapton H. This second mechanism appears to occur in thermal equilibrium with the surface (otherwise, the erosion depth would not depend on the surface temperature). If we look at the increase in erosion yield, it doubles over the range of temperatures studied for Teflon and only slightly more than doubles over the same temperature range for Kapton. The reason that the high temperature erosion yield for Teflon remains much lower than that of Kapton is because the baseline erosion yield (from the temperature-independent mechanism) for Teflon is much lower than that of Kapton H. It is possible that the temperature-dependent mechanisms for both materials are similar (they do have similar activation energies) but that the temperature-independent mechanisms (*i.e.*, direct reaction mechanism) are very different. The ultimate dependence of roughness on temperature represents some kind of complicated interplay between the temperature-dependent and temperature-independent mechanisms.

The AFM surface roughness measurements support the two-term mechanism. Although the change in roughness is small, there is an increase as a function of temperature. Oxygen atom attack on the surface at temperatures below 373 K results in the formation of the "shag-carpet" topography (figure 18). When the surface temperature is raised, the thermal equilibrium reactions begin to enhance the amount of material lost; therefore, the sur-

face roughness begins to increase. Although the same roughness pattern of Kapton does not occur with FEP Teflon, the high resistance of Teflon to O-atom attack and the temperature limitations of the polymer support differences in the observed roughness trend. Though the mechanism is still unknown, the activation energy has been determined to be 0.24 eV, and it is clear that two mechanisms are involved. The presence of VUV light in the hyperthermal beam may enhance oxygen atom reactions with FEP Teflon for the temperature-independent mechanism. It should be noted that a synergistic mechanism, probably involving the temperature-independent mechanism, has not been ruled out.

CHAPTER 7

CONCLUSION

The temperature dependence of Kapton H and FEP Teflon erosion resulting from hyperthermal atomic oxygen attack in a simulated LEO environment has been investigated with the aid of surface profilometry and AFM. The erosion of Kapton H had an apparently greater temperature dependence than did FEP Teflon. Teflon eroded only 20% to 40% of the reference standard, while Kapton eroded 100% to 330%. The curve of the erosion rate verses Temperature^{-1} was fitted to a $y = A\exp(-Bx) + C$. This suggests that two mechanisms are involved in polymer erosion, temperature-dependent and -independent. At lower temperatures, the rate-limiting step in erosion is a direct (temperature-independent) reaction. This reaction may still have a significant barrier that can be overcome by the high energy of the atom-surface collision. If oxygen atoms come into thermal equilibrium with the surface at lower temperatures, they will have a low probability to react because the barriers are too high. As the temperature is increased, the thermal O-atom reactivity increases, as described by an Arrhenius-type temperature dependence. The form of the temperature dependence at high temperatures is due to a mixture of the two mechanisms; however, at lower temperatures, the temperature-independent mechanism is favored over the other.

The AFM images of Kapton H and FEP Teflon support this picture. Kapton H roughness increases at temperatures less than 373 K and then begins to decrease at higher

temperatures until 573 K, where it begins to rise again. The decrease in surface roughness followed by an increase is the result of a complicated interplay between the two basic mechanisms, and this behavior can not really be explained with the current knowledge. These reactions remove the "hillock" material as the temperature increases. The FEP Teflon images do not share the same trend as Kapton H.

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