



# Atomic Oxygen Erosion Yield Dependence Upon Texture Development in Polymers

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An Erratum was added to this report April 2016.

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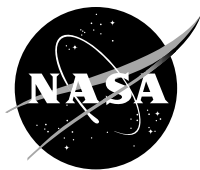
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# Erratum

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Bruce A. Banks, Ryan J. Loftus, and Sharon K. Miller

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Figures 5 and 8 have been corrected.

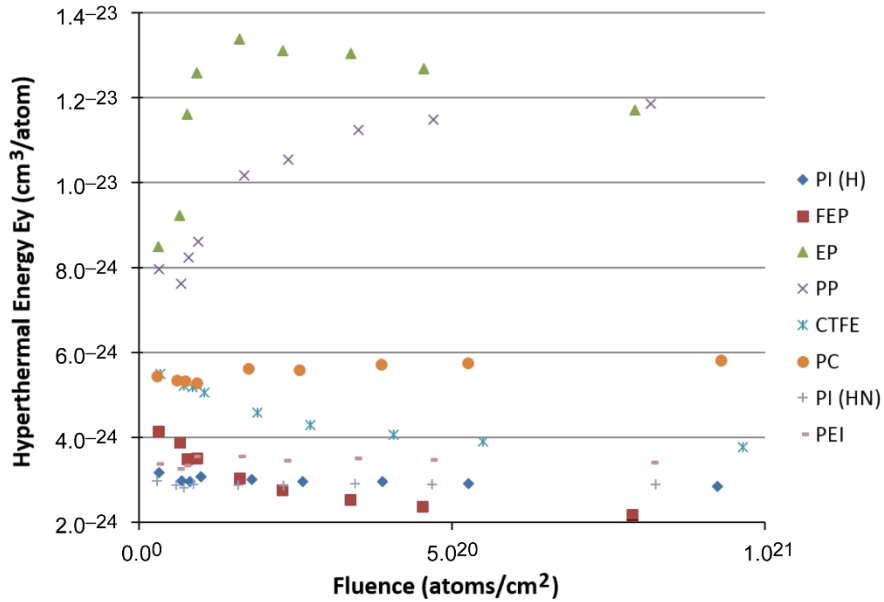


Figure 5.—Erosion yield as a function of fluence for the eight materials tested in the hyperthermal exposure environment.

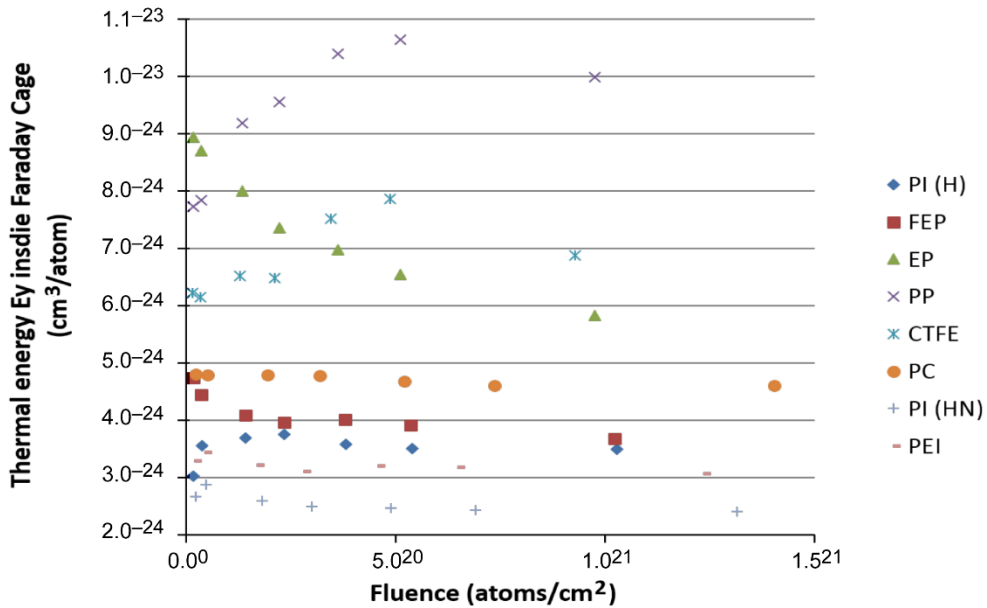


Figure 8.—Erosion yield as a function of fluence for the eight materials tested in the thermal energy environment of a Faraday cage.



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## Abstract

The atomic oxygen erosion yield (volume of a polymer that is lost due to oxidation per incident atom) of polymers is typically assumed to be reasonably constant with increasing fluence. However polymers containing ash or inorganic pigments, tend to have erosion yields that decrease with fluence due to an increasing presence of protective particles on the polymer surface. This paper investigates two additional possible causes for erosion yields of polymers that are dependent upon atomic oxygen. These are the development of surface texture which can cause the erosion yield to change with fluence due to changes in the aspect ratio of the surface texture that develops and polymer specific atomic oxygen interaction parameters. The surface texture development under directed hyperthermal attack produces higher aspect ratio surface texture than isotropic thermal energy atomic oxygen attack. The fluence dependence of erosion yields is documented for low Kapton H (DuPont, Wilmington, DE) effective fluences for a variety of polymers under directed hyperthermal and isotropic thermal energy attack.

## Introduction

The reaction of directed low Earth orbital (LEO) atomic oxygen with hydrocarbon and halocarbon polymers causes oxidative erosion which results in the development of left-standing cone structures (Refs. 1 to 4). One of the most important characteristics of a polymer relating to its LEO durability is its atomic oxygen erosion yield which is the volume lost per incident atomic oxygen atom. The measurement of this value is most reliably performed by space experiments. Measurement of the atomic oxygen fluence in atoms/cm<sup>2</sup> is typically performed by measurement of the mass loss of polyimide Kapton H witness samples. A significant amount of LEO erosion data for Kapton H has been accumulated over several decades indicates its atomic oxygen erosion yield is  $3.0 \times 10^{-24}$  cm<sup>3</sup>/atom (Ref. 5). Thus the measurement of Kapton H erosion has been adopted as the means of measuring the atomic oxygen fluence in LEO or the effective fluence in ground laboratory testing (Refs. 6 and 7). The Kapton H effective fluence is the fluence in LEO that would cause the same thickness of erosion of polyimide Kapton H as observed in ground laboratory testing.

It is well known that atomic oxygen that does not react with polymers upon first impingement has a chance of scattering and reacting with other polymer surfaces if the surface is rough. Surface roughness has been found to increase as the square root of the fluence (Ref. 8). Thus, the atomic oxygen erosion yield may increase if a greater fraction of ejected oxygen atoms impinge upon adjoining polymer cones.

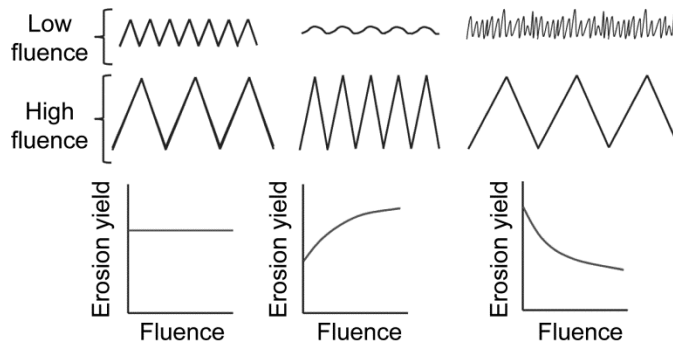


Figure 1.—Dependence of aspect ratio (cone height to cone separation) change with atomic oxygen fluence upon atomic oxygen erosion yield.

A growth in erosion yield should occur if the ratio of the height of the cones to the separation of the cones increases with fluence. If, as the cone height grows, the aspect ratio stays constant, then the erosion yield would not be expected to change with fluence. Similarly, if the aspect ratio decreases with fluence the erosion yield would be expected to decrease with fluence even though the roughness may increase. Such a decrease in erosion yield with fluence has been observed for FEP Teflon (DuPont, Wilmington, DE) (Ref. 9). This would imply that as the cones grow in height the spacing increases at a relatively higher rate causing a diminishing fraction of unreacted scattered atoms to impinge upon polymer surfaces. Figure 1 compares the effects of changing aspect ratios with fluence on erosion yield for various scenarios.

Knowledge of the erosion yield dependence on atomic oxygen fluence is important for Kapton H because Kapton H is the accepted measuring stick for fluence and if its erosion yield varies with fluence then accurate fluence measurement becomes more complex. Past and planned atomic oxygen fluence monitors utilize Kapton H fluence witness samples for calibration which rely on knowledge of erosion yield dependence on fluence (Refs. 10 and 11). To address these issues, an experimental investigation of the possible changes of the atomic oxygen erosion yield with fluence for Kapton H and a few other polymers was conducted for both hyperthermal and thermal energy atomic oxygen and is reported herein.

The intent of this paper is to compare the experimental change in the erosion yields of Kapton H and several other polymers with increasing fluence for relatively low fluences ( $<2 \times 10^{21}$  atoms/cm<sup>2</sup>) when the surface texture is developing.

## Apparatus and Procedure

A low frequency hyperthermal RF plasma asher was used to measure ground laboratory atomic oxygen erosion yields using dehydrated mass loss of Kapton H witness samples. The hyperthermal asher was an LF-5 Axic plasma asher, operated on air, at a frequency of 35 kHz and found to produce an atomic oxygen impact energy of  $\sim 0.26$  eV (Ref. 12). The low frequency LF-5 Axic plasma asher, with active electrodes above the grounded sample plane, produced an air plasma in which directed hyperthermal atomic oxygen beam impinges with normal incidence upon test samples. The result of this direct impingement causes left-standing cones to be etched on the sample surfaces that are typical of LEO hyperthermal atomic oxygen erosion. The energy of impacting atoms or ions is thought to be below 10's of electron volts because it was below the sputter threshold of silicon dioxide as measured in high fluence testing of thin film SiO<sub>2</sub> coated Kapton. The LF-5 Axic plasma asher was operated using two different sample exposure configurations. The first test consisted of ten 1.27 cm diameter samples placed on an aluminum plate and exposed to directed hyperthermal atomic oxygen attack as shown in Figure 2.





Figure 2.—Samples on a flat plate for directed hyperthermal atomic oxygen exposure in a 35 kHz hyperthermal asher.

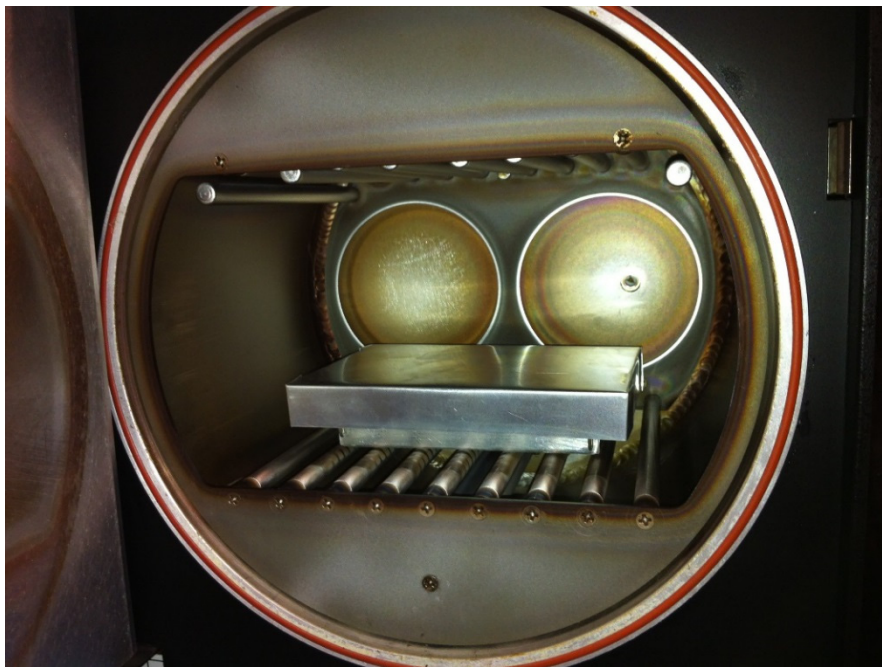


Figure 3.—Samples in a Faraday cage with a loose fitting lid for thermal atomic oxygen exposure in a 35 kHz hyperthermal asher.

The second configuration was with the same set of ten materials but placed within a Faraday cage which would cause thermal accommodation ( $\sim 0.04$  eV energy) of the atoms entering the cage as well as shield out most of the charged atoms, charged molecules, and ultraviolet radiation is shown in Figures 3 and 4.

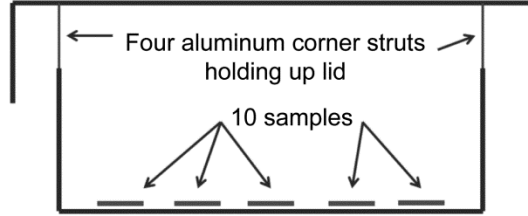


Figure 4.—Cross-section of the Faraday cage.

TABLE 1.—LIST OF MATERIALS TESTED FOR EROSION YIELD DEPENDENCE ON ATOMIC OXYGEN FLUENCE

Sample materials	Symbol
Kapton H	PI(H)
FEP Teflon	FEP
Epoxy	EP
Polypropylene	PP
Chlorotrifluoroethylene	CTFE
Polycarbonate	PC
Kapton HN	PI(HN)
Polyetherimide	PEI

During most of the testing the hyperthermal exposure tray was placed on top of the Faraday cage for convenience of allowing both atomic oxygen exposures to occur simultaneously. Each type of exposure occurred in increments where new Kapton H samples were used to measure each incremental fluence and placed in with other polymers (that were not changed) to measure their erosion yield (using mass loss) for each increment. The samples were weighed after 48 hour periods of vacuum dehydration using a Sartorius microgram balance. A list of materials tested in both the hyperthermal exposures and Faraday cage exposures is shown in Table 1.

Samples were simply laid on a flat aluminum surface rather than in sample holders for the hyperthermal exposures or in the Faraday cage. This minimized mass change inconsistencies if they were placed in sample holders due to varying areas of exposure to the directed atomic oxygen during the many weight measurements needed for each sample. Small increments of Kapton H effective fluence were used (typically  $1.46 \times 10^{20}$  atoms/cm<sup>2</sup>) to try to observe the effects of the development of surface texture upon the erosion yields.

## Results and Discussion

### Direct Hyperthermal Atomic Oxygen Exposure Tests

The 22.58 hour atomic oxygen exposure of the sample plate with 10 Kapton H samples was used to make a flux map for the exposure plate which resulted in an average Kapton H effective flux of  $4.86 \times 10^{15}$  atoms/(cm<sup>2</sup> sec) and a fluence of  $3.95 \times 10^{20}$  atoms/cm<sup>2</sup>. This flux map was used for the first two fluence increments. The resulting ratio of flux at each intended sample location relative to the sample in position one is given in Table 2.

For the remainder of the fluence increments, the witness plate was placed on top of the Faraday cage. A new flux map was made for this configuration and is shown in Table 3.

Two Kapton H fluence witness samples were changed out with new Kapton H samples for each increment of exposure while the other samples received accumulated atomic oxygen exposure increments. Tables 2 and 3 were then used to compute the accumulated Kapton H effective fluence of all the materials listed in Table 1.

Figure 5 shows the results of measuring the erosion yield as a function of fluence for the nine materials tested in the hyperthermal exposure environment.

TABLE 2.—RATIO OF FLUX AT EACH SAMPLE LOCATION  
RELATIVE TO THE FLUX IN POSITION 1 FOR  
THE FIRST TWO FLUENCE INCREMENTS

Sample position	Flux relative to position 1
1	1.00
2	0.96
3	0.96
4	0.99
5	1.06
6	0.95
7	0.89
8	0.89
9	0.92
10	1.01

TABLE 3.—RATIO OF FLUX AT EACH SAMPLE LOCATION  
RELATIVE TO THE FLUX IN POSITION 1 FOR  
THE LAST SEVEN FLUENCE INCREMENTS

Sample position	Flux relative to position 1
1	1.00
2	0.89
3	0.85
4	0.90
5	1.04
6	1.05
7	1.02
8	0.87
9	0.84
10	1.11

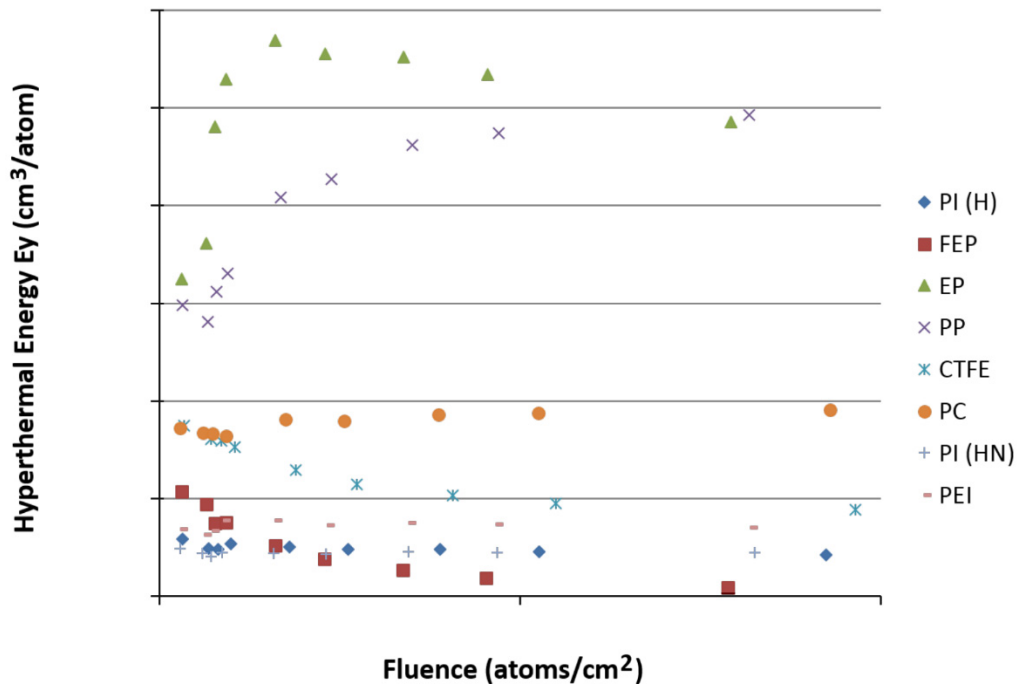


Figure 5.—Erosion yield as a function of fluence for the eight materials tested in the hyperthermal exposure environment.

As can be seen in Figure 5, the erosion yield for epoxy and polypropylene increase with fluence; polycarbonate, Kapton H (PI (H) DuPont, Wilmington, DE) and PI-HN, and polyetherimide (PEI) remain relatively constant with fluence; and chlorotrifluoroethylene (CTFE) and fluorinated ethylene propylene (FEP) have erosion yields that decrease with fluence. The fact the erosion yield for Kapton H and HN are relatively constant with fluence is helpful because it is relied upon for measuring atomic oxygen fluence. The decrease in erosion yield of FEP observed in the hyperthermal asher is in difference to the observed increase in erosion yield from low Earth orbital data (Ref. 6). This difference in results is thought to be due to a stronger effect on erosion yield that is dependent on equivalent sun hours (ionizing radiation and temperature) than atomic oxygen fluence. It should be noted that the erosion yield decreases with fluence for high hyperthermal fluences where ash content begins to play a shielding role as described (Ref. 13).

To compare the surface morphology of materials that had demonstrated erosion yields which increase or decrease with fluence, scanning electron microscopy was performed on FEP and epoxy samples which were exposed to two different fluences. Figures 6 and 7 show scanning electron microscope images of FEP and epoxy surfaces respectively that have been exposed to two different hyperthermal atomic oxygen fluences.

As can be seen from Figures 6 and 7, although the images appear different at different fluences, it is difficult to clearly attribute the differences in erosion yields for the fluences indicated to changes in aspect ratio of the surface features. It is thought that the material differences in the development of surface texture, trapping of scattered oxygen atoms, and angular dependent reaction characteristics may play a significant role in erosion yield dependence changes with fluence.

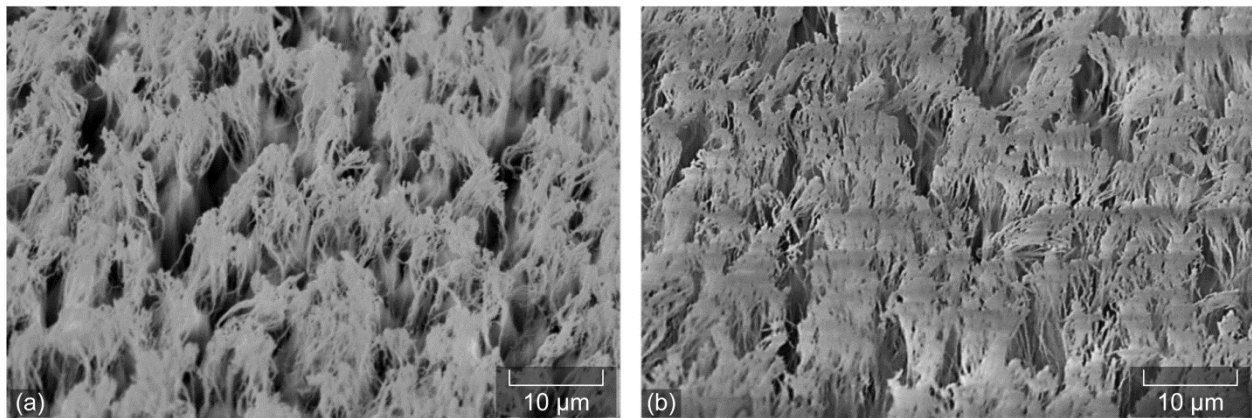


Figure 6.—FEP Teflon that has been exposed to two different hyperthermal atomic oxygen Kapton H effective fluences: (a)  $5.87 \times 10^{20}$  atoms/cm<sup>2</sup>, (b)  $1.94 \times 10^{21}$  atoms/cm<sup>2</sup>.

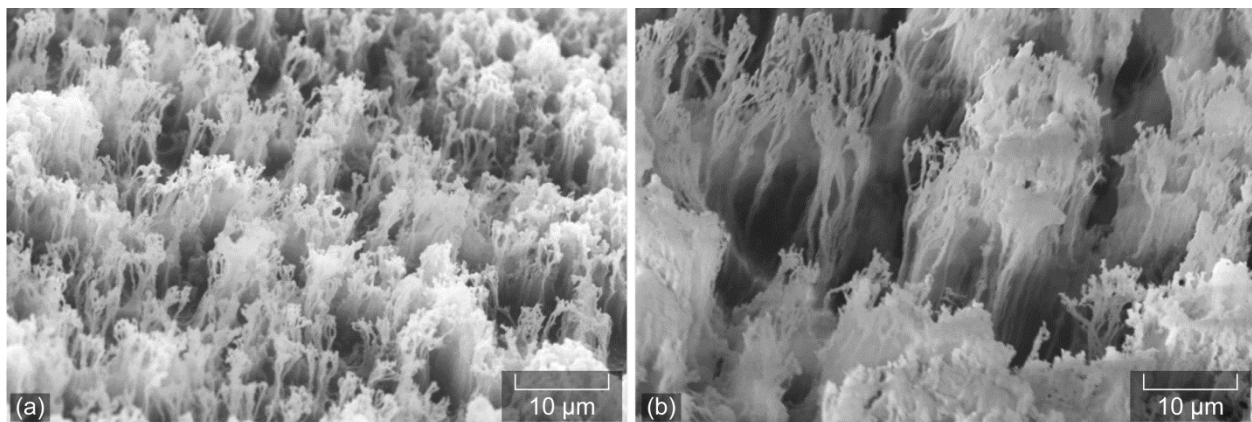


Figure 7.—Epoxy that has been exposed to two different hyperthermal atomic oxygen Kapton H effective fluences: (a)  $5.19 \times 10^{20}$  atoms/cm<sup>2</sup>, (b)  $1.22 \times 10^{21}$  atoms/cm<sup>2</sup>.

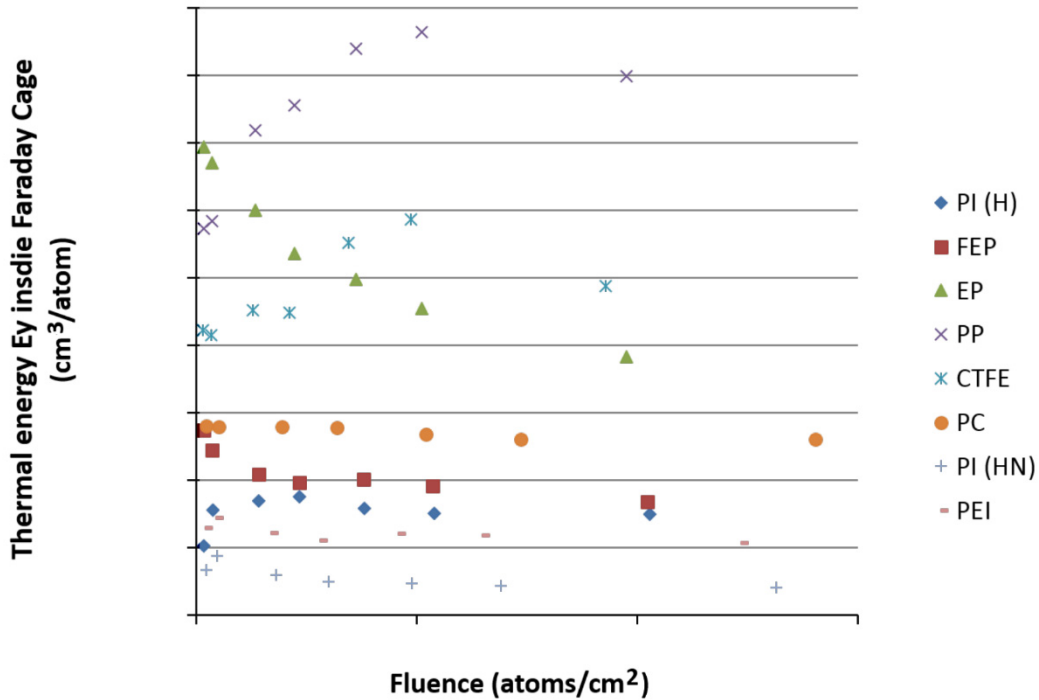


Figure 8.—Erosion yield as a function of fluence for the eight materials tested in the thermal energy environment of a Faraday cage.

### Thermal Energy Faraday Cage Exposure Tests

A 17.23 hour atomic oxygen exposure of the sample plate with 10 Kapton H samples was used to make a flux map for 10 Faraday cage sample locations which resulted in an average Kapton H effective flux of  $6.1 \times 10^{15}$  atoms/(cm<sup>2</sup> sec) and a fluence of  $3.73 \times 10^{20}$  atoms/cm<sup>2</sup>. The resulting ratio of flux at each intended sample location relative to the flux in a specific position was measured. Two sample positions were used for Kapton H fluence witness which were changed out with new Kapton H samples for each increment of exposure while the other samples were left in place to receive cumulative atomic oxygen exposure for each increment.

Figure 8 shows the results of measuring the erosion yield as a function of fluence for the eight materials tested in the thermal energy environment within a Faraday cage.

In contrast to the hyperthermal results in Figure 4, the erosion yield for epoxy decreases with fluence and for chlorotrifluoroethylene increases slightly with fluence. It is fortunate that Kapton H and HN (which are the most frequent materials used for fluence measurement) have erosion yields that are reasonably constant with fluence because their fluence measurements should not have to be adjusted as a function of fluence.

### Summary

The atomic oxygen erosion yield of some polymers was experimentally found to change with atomic oxygen fluence. Eight polymers were tested experimentally under hyperthermal and isotropic thermal energy atomic oxygen attack and were found to produce results that varied from increasing, decreasing, and staying rather constant with fluence. It is believed that the material differences in the development of surface texture, trapping of scattered oxygen atoms, and angular dependent reaction characteristics may play a significant role in erosion yield dependence changes with fluence. Fortunately the materials most used for atomic oxygen fluence measurement, polyimide Kapton H and HN, were found to have an erosion yield that was rather independent of fluence both when exposed to hyperthermal and isotropic thermal energy atomic oxygen.

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