

# THE EFFECT OF ASH AND INORGANIC PIGMENT FILL ON THE ATOMIC OXYGEN EROSION OF POLYMERS AND PAINTS

(ISMSE-12)

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## ABSTRACT

Low atomic oxygen fluence (below  $1 \times 10^{20}$  atoms/cm<sup>2</sup>) exposure of polymers and paints that have a small ash content and/or inorganic pigment fill does not cause a significant difference in erosion yield compared to unfilled (neat) polymers or paints. However, if the ash and/or inorganic pigment content is increased, the surface population of the inorganic content will begin to occupy a significant fraction of the surface area as the atomic oxygen exposure increases because the ash is not volatile and remains as a loosely attached surface layer. This results in a reduction of the flux of atomic oxygen reacting with the polymer and a reduction in the rate of erosion of the polymer remaining. This paper presents the results of ground laboratory and low Earth orbital (LEO) investigations to evaluate the fluence dependence of atomic oxygen erosion yields of polymers and paints having inorganic fill content.

## 1. INTRODUCTION

Post-retrieval inspection of the Long Duration Exposure Facility indicated that titanium dioxide white pigmented polyurethane paint (Chemglaze A276), which was exposed to atomic oxygen in low Earth orbit (LEO) for 5.75 years, was not eroded away in spite of the very high atomic oxygen fluence (a fluence of  $9.02 \times 10^{21}$  atoms/cm<sup>2</sup>). Although the polyurethane binder in the surface of the paint had been eroded, the pigment particles remained in place and helped protect the underlying polyurethane from atomic oxygen erosion [1]. Ground laboratory testing has also indicated that even polymers without any purposely-added inorganic pigment contain a measurable inorganic content that remains as ash when the polymer is fully oxidized [2]. Samples of clear and white polyvinylfluoride (Tedlar) were flown for 3.95 years (August 16, 2001 to July 30, 2005) on the International Space Station (ISS) as part of the Materials International Space Station 2 (MISSE 2) PEACE polymers experiment and exposed to a low Earth orbital (LEO) ram fluence of  $8.43 \times 10^{21}$  atoms/cm<sup>2</sup> [2]. The results of the experiment on these two samples indicated that the 0.295 mass fractional fill of titanium dioxide resulted in a factor of 31.5 reduction

in the erosion yield (from  $3.19 \times 10^{-24}$  atoms/cm<sup>2</sup> for clear Tedlar to  $1.01 \times 10^{-25}$  for white Tedlar) [2, 3]. Samples of clear and white Tedlar were also flown on the MISSE 7 polymers experiment (November 2009 to July 2011). The intent of this investigation is to explore the effect on erosion yield (volume loss per incident atomic oxygen atom) of inorganic ash-filled polymers and paints as a function of fluence and model the results, comparing ground laboratory plasma results with in space LEO results from MISSE 2 and 7.

## 2. APPARATUS AND PROCEDURE

### 2.1. Space Experiments

In addition to the white Tedlar samples that were exposed on ISS as part of MISSE 2 polymers experiments, samples of white Tedlar samples were also exposed on MISSE 7 to three levels of ram atomic oxygen by having samples uncovered (exposed samples to a ram fluence of  $4.18 \times 10^{21}$  atoms/cm<sup>2</sup>), covered with 0.5 mil (0.0127 mm) of Kapton H (samples exposed to a fluence of  $3.76 \times 10^{21}$  atoms/cm<sup>2</sup>), and 1 mil (0.0254 cm) of Kapton H (samples exposed to fluence of  $2.91 \times 10^{21}$  atoms/cm<sup>2</sup>), as shown in Fig. 1.

The polymers that were covered with Kapton H were intended to provide erosion yield data for two lower fluences than the full mission ram fluence because they blocked the fluence that would normally arrive at the samples until the Kapton H was fully eroded. The mass loss of a Kapton H sample also on MISSE 7 was used to determine the MISSE 7 ram fluence. All samples were vacuum dehydrated in accordance with ASTM E 2089-00 prior to mass measurements [4].

### 2.2. Ground Laboratory Experiments

Samples of epoxy (Loctite Professional Heavy Duty Epoxy, 60 Minute Set) and polyurethane paint (Minwax Indoor/Outdoor Helmsman Spar Urethane, Clear Gloss) were tested both in an unfilled state and filled with measured quantities of various pigment particles, including fumed silica, aluminum oxide, calcium carbonate, zinc oxide, and powdered (-325 mesh)

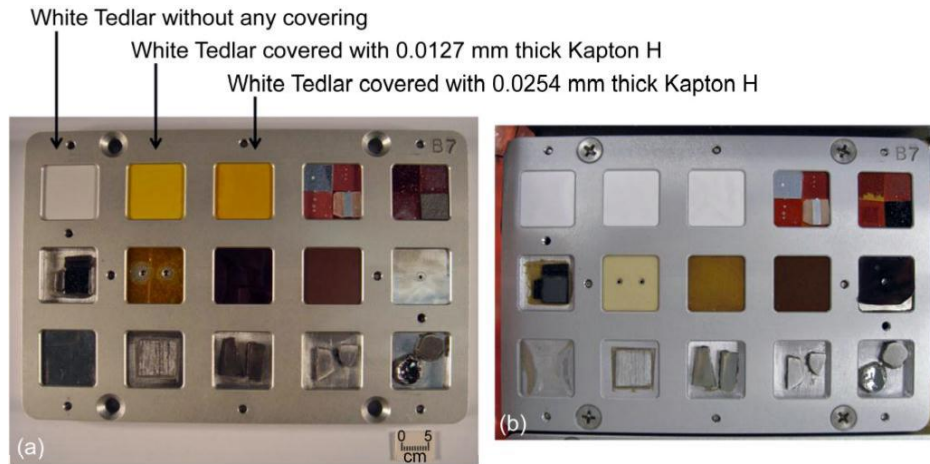


Figure 1. MISSE 7 experiment tray B7 showing uncovered and Kapton H covered samples: (a) Pre-flight, (b) Post flight (notice all Kapton covers are eroded away).

muscovite mica. Samples of white Tedlar were also used to compare MISSE space results with ground laboratory results. To prevent mass loss from atomic oxygen being confused with mass loss due to loss of volatile species, the samples of epoxy and polyurethane were vacuum dehydrated in an oven at 48 to 50 °C for a sufficient duration to indicate negligible mass loss on a microgram balance prior to any atomic oxygen exposure.

Two types of ground laboratory atomic oxygen exposure experiments were performed: Radio frequency (13.56 MHz, Plasma Prep II, operated on air) plasma asher tests which produced an isotropic arrival of thermal energy atomic oxygen; and low frequency (35 kHz, LF-5, Axic, operated on air) plasma tests which produced directed hyperthermal atomic oxygen air plasmas. Evidence of the more directed energetic atomic oxygen exposure in the low frequency asher could be seen by microscopic cone formation on samples of pyrolytic graphite that are typical of hyperthermal atomic oxygen erosion. The structures were similar to typical LEO results of atomic oxygen attack on pyrolytic graphite. In isotropic thermal energy ashers only wide microscopic craters develop.

The mass loss from dehydrated Kapton H samples was used to obtain the Kapton H effective atomic oxygen fluence for the ground laboratory samples as described in ASTM E 2089-00 [3] using a Sartorius microgram balance. Samples consisted of as-received white Tedlar and epoxy and polyurethane filled with weighed amounts of metal oxides. High fluence white Tedlar samples were exposed by placing 2.54 cm diameter by 0.0508 mm thick samples in aluminum holders next to Kapton H fluence witness samples in identical holders (see Fig. 2(a)). The epoxy and polyurethane samples were spread onto 2 by 2 cm fused silica slides using a doctor's knife technique to produce a coating of approximately 0.05 to 0.5 mm thick and 1.27 cm in diameter (see Fig. 2(b)).

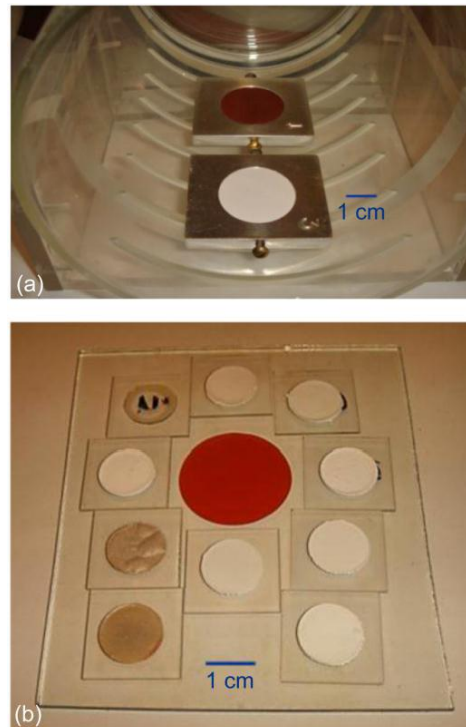


Figure 2. Erosion yield test samples. (a) White Tedlar and Kapton H samples in aluminum holders. (b) Ash-filled epoxy and polyurethane samples on fused silica slides along with a larger Kapton H fluence witness sample.

### 3. RESULTS AND DISCUSSION

The mass and volume fraction of ash (or inorganic fill) of the as-received polymers as well as the fractional ash contents after adding additional inorganic fill are shown in Tab. 1.

Table 1. Polymers and volume fraction ash

Polymer	Native ash material	Native volume fraction ash	Density of native ash material, gm/cm <sup>3</sup>	Added ash material	Density of added ash material, gm/cm <sup>3</sup>	Filled total volume fraction ash
White Tedlar	TiO <sub>2</sub>	0.1134	4.23	none	NA	0.113
Epoxy	SiO <sub>2</sub>	0.0137	2.2	none	NA	0.0137
Epoxy	SiO <sub>2</sub>	0.0137	2.2	SiO <sub>2</sub>	2.20	0.248
Epoxy	SiO <sub>2</sub>	0.0137	2.2	Al <sub>2</sub> O <sub>3</sub>	3.97	0.151
Epoxy	SiO <sub>2</sub>	0.0137	2.2	CaCO <sub>3</sub>	2.71	0.364
Epoxy	SiO <sub>2</sub>	0.0137	2.2	ZnO	5.6	0.125
Epoxy	SiO <sub>2</sub>	0.0137	2.2	mica	2.83	0.109
Polyurethane	SiO <sub>2</sub>	0.0233	2.2	none	NA	0.0233
Polyurethane	SiO <sub>2</sub>	0.0233	2.2	SiO <sub>2</sub>	2.20	0.417
Polyurethane	SiO <sub>2</sub>	0.0233	2.2	Al <sub>2</sub> O <sub>3</sub>	3.97	0.371
Polyurethane	SiO <sub>2</sub>	0.0233	2.2	CaCO <sub>3</sub>	2.71	0.507
Polyurethane	SiO <sub>2</sub>	0.0233	2.2	ZnO	5.6	0.259
Polyurethane	SiO <sub>2</sub>	0.0233	2.2	mica	2.83	0.198

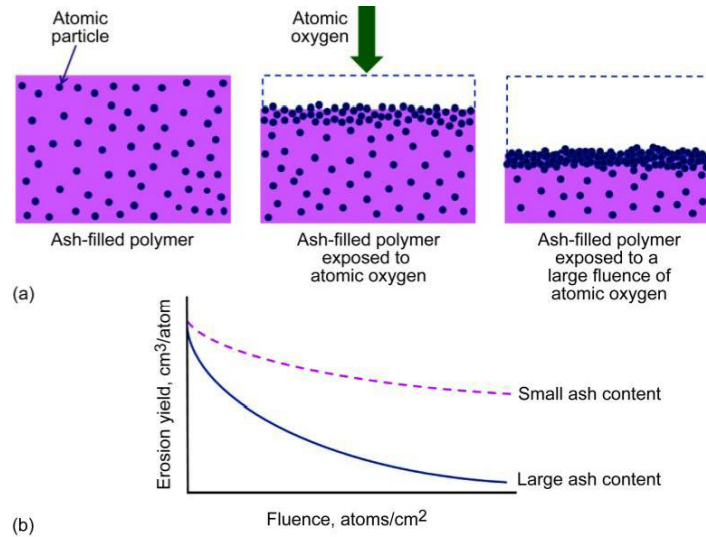


Figure 3. Effect of atomic oxygen fluence on the atomic oxygen erosion yield of ash containing polymers: (a) Evolution of ash surface concentration with fluence, (b) Resulting erosion yield dependence on fluence.

For purposes of estimation, the small fractional volume fill of ash in the native epoxy and polyurethane was assumed to be SiO<sub>2</sub>. The atomic oxygen erosion yield,  $E$ , is the volume of polymer removed by atomic oxygen per incident atom and is given by

$$E = \frac{\Delta M}{\rho A F} \quad (1)$$

Where:

$F = \text{Fluence, atoms/cm}^2$

$A = \text{Exposed area of sample, cm}^2$

$\rho = \text{density, grams/cm}^3$

$\Delta M = \text{Mass loss, grams}$

The thickness,  $x$ , of polymer eroded is simply given

$$x = EF \quad (2)$$

However, for materials that consist of significant fractional contents of inorganic ash fill or inorganic fibers (such as fiberglass), the mass loss is just from the polymer loss and the relevant density is just that of the polymer (not including the added inorganic ash or fiber fill). With such materials, the thickness removed becomes difficult to measure because the remaining ash forms a fragile porous coating over the remaining filled polymer material. For the polymers used in this investigation, although the erosion yield of the polymers with very little ash content reduces least with fluence, the erosion yield of the ash-filled polymers reduce at a greater rate with fluence because of atomic oxygen shielding afforded by the gradual accumulation of residual ash on the surface of the material as shown in Fig. 3.

The fluence is measured as actual fluence in LEO based on the erosion (mass loss) of Kapton H witness samples where the erosion yield,  $E$ , for Kapton H is  $3.00 \times 10^{-24} \pm 0.07 \times 10^{-24} \text{ cm}^3/\text{atom}$  [4, 5]. The fluence measured in ground laboratory facilities is the Kapton H effective fluence, which is defined as the fluence in LEO that would be needed to cause the same amount of erosion as observed in the specific ground laboratory facility. For the effective flux to be the same in both the environments of LEO and traditional thermal energy plasma ashers, the actual flux in thermal energy ashers has to be very high in comparison to LEO because the energy of the atomic oxygen is only a fraction of an electron volt in comparison to  $\sim 4.5 \text{ eV}$  in LEO thus reducing the reaction probability of the atoms. By comparison of the erosion yields of white Tedlar measured for various fluences in LEO from MISSE 2 and 7 data with erosion yields in ground laboratory plasma asher facilities, one can gain an understanding of how much thermal energy fluence is needed to simulate a specific fluence in LEO for white Tedlar.

The Kapton H effective atomic oxygen erosion yields of white Tedlar for isotropic asher exposures, for directed asher exposures, and from LEO data from MISSE 2 and 7 are compared in Fig. 4.

As can be seen in Fig. 4, the erosion yields in all environments decrease with fluence. Although an exponential curve fit to this data and other samples listed in Tab. 1 has a high correlation coefficient,  $R^2$ , for data in the fluence range from 0 to  $1 \times 10^{20} \text{ atoms/cm}^2$ , for fluences above  $1 \times 10^{20} \text{ atoms/cm}^2$ , the data indicated a better fit to a power law dependence. In Fig. 3, the fluence measurement is made in the respective environment (LEO, isotropic thermal energy plasma asher, or hyperthermal directed asher).

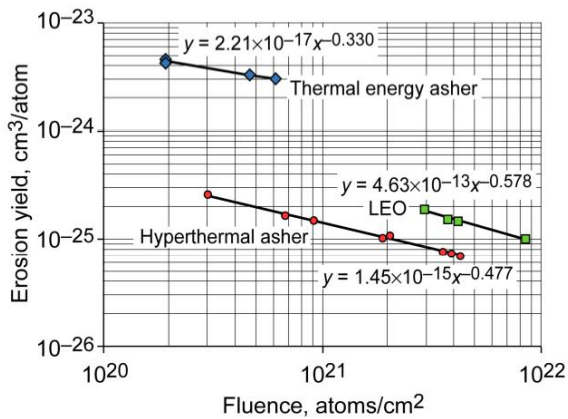


Figure 4. Erosion yield dependence upon fluence for white Tedlar in LEO (MISSE 2 and 7), isotropic thermal energy plasma asher, and hyperthermal directed asher environments.

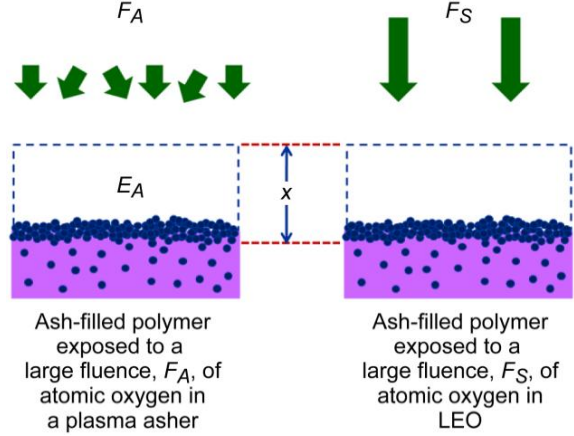


Figure 5. Comparison of asher and LEO exposed ash-filled polymer eroded the same amount of polymer thickness loss,  $x$ .

To be able to determine what fluence in LEO is represented by a specific ground laboratory plasma asher fluence at fluences above  $1 \times 10^{20} \text{ atoms/cm}^2$ , a comparison can be made where the thickness of polymer that has been eroded in both LEO and an asher are identical. Fig. 5 compares a section view illustration of an ash-filled polymer that was eroded to the same thickness,  $x$ , in both asher and LEO environments.

Based on this comparison and the power series modeling, we can state:

$$x = E_A F_A = E_S F_S \quad (3)$$

Where

- $E_A$  = Erosion yield of polymer in the asher,  $\text{cm}^3/\text{atom}$
- $E_S$  = Erosion yield of polymer in LEO,  $\text{cm}^3/\text{atom}$
- $F_A$  = Kapton effective fluence measured in the asher,  $\text{atoms/cm}^2$
- $F_S$  = Fluence measured in LEO,  $\text{atoms/cm}^2$

Using the best-fit power law dependence for the LEO and isotropic thermal energy plasma asher data, a reasonable fit to the data is

$$E_A = K_A F_A^{-C_A} \text{ and } E_S = K_S F_S^{-C_S} \quad (4)$$

Thus one can state that

$$x = F_A K_A F_A^{-C_A} = F_S K_S F_S^{-C_S} \quad (5)$$

Where the constants  $K_A$ ,  $K_S$ ,  $C_A$ ,  $C_S$  are:

$$\begin{aligned} K_A &= 2.21 \times 10^{-17} \text{ cm}^3/\text{atom} \\ K_S &= 4.63 \times 10^{-13} \text{ cm}^3/\text{atom} \\ C_A &= 3.30 \times 10^{-1} \\ C_S &= 5.78 \times 10^{-1} \end{aligned}$$

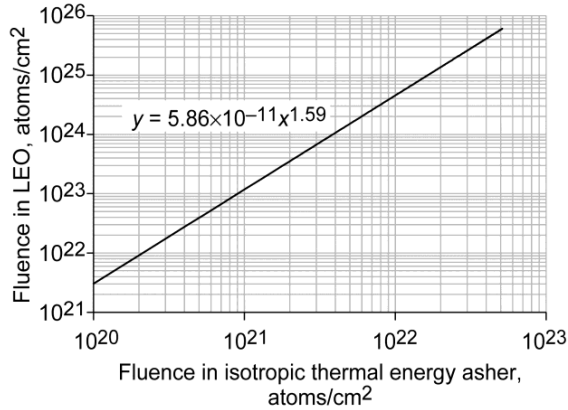


Figure 6. Fluence in LEO simulated by Kapton H effective fluence in an isotropic thermal energy plasma asher for white Tedlar.

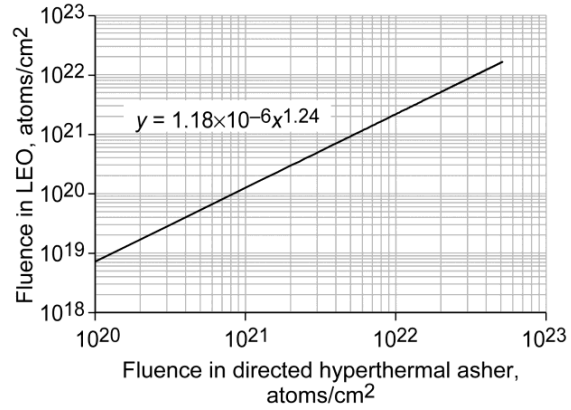


Figure 7. Fluence in LEO simulated by Kapton H effective fluence in a directed hyperthermal energy plasma asher for white Tedlar.

Solving for  $F_S$  in terms of  $F_A$  results in

$$F_S = \left( \frac{K_A}{K_S} \right)^{\left[ \frac{1}{1-C_S} \right]} F_A^{\left[ \frac{1-C_A}{1-C_S} \right]} \quad (6)$$

Which results in  $F_S = 86 \times 10^{-11} F_A^{1.59}$  (7)

Fig. 6 shows a plot of the fluence in LEO that is represented by the Kapton H effective fluence in an isotropic thermal energy plasma asher for white Tedlar.

As can be seen from Fig. 6 the ratio of LEO fluence to isotropic thermal energy asher fluence goes up with increasing asher fluence. This is probably due to the fact that the LEO fluence becomes increasingly less effective with higher arrival doses due to the hyperthermal oxygen atoms becoming increasingly more thermally accommodated thus reducing the probability of reaction. If one examines the comparison between the erosion yield of white Tedlar in LEO with the erosion of white Tedlar in a hyperthermal directed asher, the same equations apply, but one has to enter the appropriate set of constants for that asher:

$$\begin{aligned} K_A &= 1.45 \times 10^{-17} \text{ cm}^3/\text{atom} \\ K_S &= 4.63 \times 10^{-13} \text{ cm}^3/\text{atom} \\ C_A &= 4.77 \times 10^{-1} \\ C_S &= 5.78 \times 10^{-1} \end{aligned}$$

This results in the equation  $F_S = 1.18 \times 10^{-6} F_A^{1.24}$  (8)

which is plotted in Fig. 7.

As can be seen from Fig. 7, the ratio of LEO fluence to directed hyperthermal energy plasma asher fluence indicates that the hyperthermal energy asher is actually more aggressive in oxidizing white Tedlar than LEO

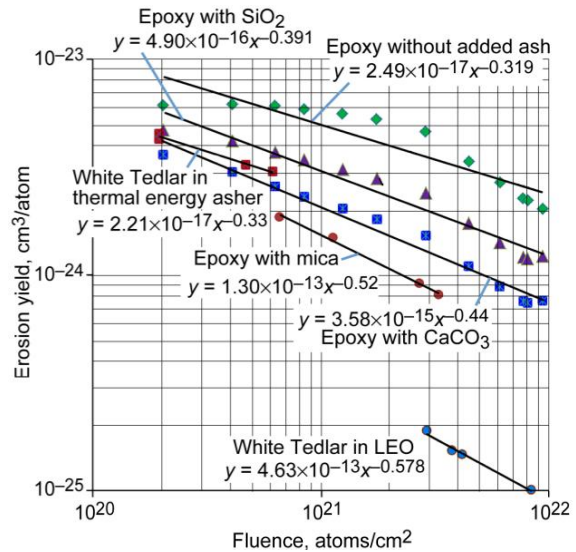


Figure 8. Atomic oxygen erosion yield fluence dependencies for epoxy with no added ash, and with added  $\text{SiO}_2$ ,  $\text{CaCO}_3$ , and mica, in an isotropic thermal energy plasma asher as well as white Tedlar in LEO and white Tedlar in an isotropic thermal energy plasma asher.

exposure is. This is probably due to the energy of the atomic oxygen in the hyperthermal energy plasma actually being greater than the energy of atomic oxygen in LEO.

If one compares the reduction in erosion yield as a function of fluence in an isotropic thermal energy plasma asher for a variety of ash fill materials in epoxy and white Tedlar in LEO, the dependence shows reasonable agreement with a power law dependence with the least fluence dependent material being epoxy with no added ash. A selection of erosion yield dependencies investigated is shown in Fig. 8 for comparison purposes.

Although the erosion yields of various ash-fill materials decrease in a similar manner with fluence as shown in Fig. 6, the exponent for the fluence dependencies is not totally consistent with a dependence on the volume fraction ash fill for the polymers. Tab. 2 lists the volume fraction ash of various fill materials investigated along with the erosion yield power law dependence constant,  $C_A$ , which provided the best fit to the data taken in the isotropic thermal energy plasma asher.

Table 2. Volume fraction ash of various fill materials in epoxy and polyurethane and the erosion yield power law dependence constant,  $C_A$ , for these materials exposed in an isotropic thermal energy plasma asher.

Polymer	Added ash	Volume fraction fill	Fluence exponent, $C_A$
Epoxy	none	0.0137	0.319
Epoxy	$Al_2O_3$	0.2481	0.562
Epoxy	$SiO_2$	0.1509	0.391
Epoxy	ZnO	0.1254	0.483
Epoxy	$CaCO_3$	0.3638	0.440
Epoxy	Mica	0.1130	0.520
PU	$SiO_2$	0.4166	0.653
White Tedlar	NA	0.1133	0.330

Fig. 9 shows a plot of the exponent for the power law dependence of erosion yield as a function of the volume fill fraction for the materials listed in Tab. 2.

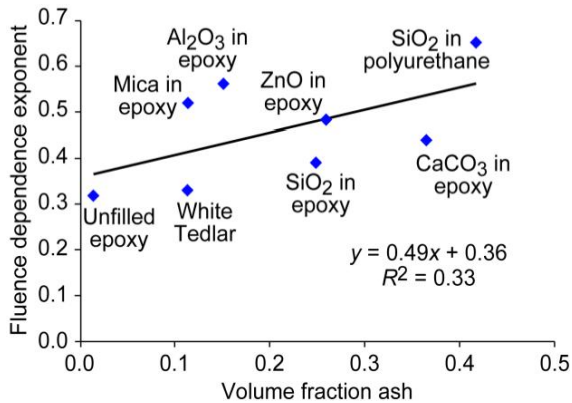


Figure 9. Power law exponent,  $C_A$ , dependence of erosion yield on the volume fill fraction ash for the materials listed in Tab. 2 in an isotropic thermal energy plasma asher.

As can be seen from Fig. 9 and Eq. (4), in general, the erosion yield generally decreases, as expected, with increasing volume ash fill and fluence. However, the correlation with volume fraction ash is considerably poorer than that associated with the erosion yield as a function of fluence. This could potentially be due to the shape and size of the ash particles not being identical in which case particle surface area may not be determined only by the volume fraction ash [3].

Table 3. Volume fraction ash of various fill materials in polyurethane, epoxy, and white Tedlar and the erosion yield power law dependence constant,  $C_A$ , exposed in a directed hyperthermal energy plasma asher.

Polymer	Added ash	Volume fraction fill	Fluence exponent, $C_A$
Polyurethane	none	0.0233	0.3190
Polyurethane	$Al_2O_3$	0.3709	0.5990
Polyurethane	$SiO_2$	0.4166	0.7880
Polyurethane	ZnO	0.2587	0.7825
Polyurethane	$CaCO_3$	0.5066	0.7678
Epoxy	Mica	0.1130	0.5202
White Tedlar	NA	0.1133	0.4767

The erosion yield dependence on fluence for ash-filled polymers exposed in the directed hyperthermal energy plasma asher indicated power law dependencies closer to the white Tedlar in LEO environments and with better correlation of the power law exponent,  $C_A$ , with volume fraction ash as shown in Figs. 10 and 11 as well as in Tab. 3.

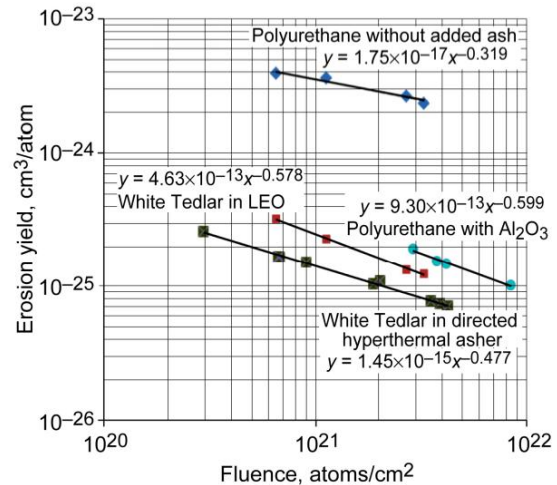


Figure 10. Atomic oxygen erosion yield fluence dependencies for polyurethane with no added ash, and filled with  $Al_2O_3$ , with comparisons to white Tedlar in the same directed hyperthermal energy plasma asher environment and in LEO.

However, the erosion yield of white Tedlar in both LEO and the directed hyperthermal energy plasma asher environments were closer to each other and had more comparable fluence exponents than did the exposure in the isotropic thermal energy plasma asher. For this reason using a directed hyperthermal asher for exposure of ash-filled composites to a fluence between  $10^{20}$  and  $10^{22}$  atoms/cm<sup>2</sup> and then applying Eq. (7) to determine the LEO fluence that is represented by the asher fluence may provide the most reasonable estimates of erosion yield in LEO.

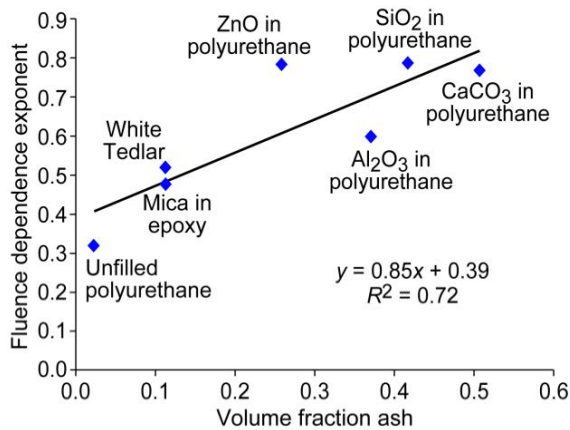


Figure 11. Power law exponent, CA, dependence on the volume fill fraction ash for the materials listed in Tab. 3 in a directed hyperthermal energy plasma asher.

The lack of high correlation of power law exponent dependence with volume fill fraction ash could potentially be caused by the size and shape of ash particles playing a more important role on erosion in a directed hyperthermal environment than in an isotropic thermal energy environment. Based on Tabs. 2 and 3 as well as on Figs. 9 and 11, it appears that only a small volume fraction ash (~11%) is sufficient to cause a reduction in erosion yield by more than order of magnitude when the fluence is increased by from 1020 to 1022 atoms/cm<sup>2</sup>. This implies significant benefits in comparison to durability assumptions based on a fixed erosion yield independent of fluence. Because the reduction in the erosion yield of white Tedlar with fluence is typical of other ash-filled polymers in both hyperthermal and thermal energy environments, the prediction of erosion yield in LEO can reasonably be based on taking data in either the isotropic thermal energy plasma asher and/or hyperthermal directed asher and using Eqs. (7) or (8) (respectively) to determine what fluence in LEO is represented by the asher exposure. However, the hyperthermal directed asher has the greatest potential to more accurately predict LEO erosion yields with fluence because the fluences and erosion yields in LEO are more closely represented by the hyperthermal directed asher than the thermal energy isotropic asher environment. Further comparison of LEO data with asher data is needed to more accurately determine the degree to which white Tedlar data is representative of a range of ash-filled polymers.

#### 4. SUMMARY

A comparison of erosion yields as a function of fluence for white Tedlar in the LEO environment retrieved from the ISS MISSE 2 and 7 polymers experiments was made with exposures in ground laboratory ashers. Two types of ground laboratory atomic oxygen asher were used: a radio frequency (at 13.56 MHz, Plasma Prep II, operated on air) plasma asher which produced an isotropic arrival

of thermal energy atomic oxygen; and a low frequency (35 kHz, LF-5, Axic, operated on air) plasma asher which produced directed hyperthermal atomic oxygen air plasmas. In addition, polyurethane paint and epoxy in an unfilled state and filled with measured quantities of various pigment particles, including fumed silica, aluminum oxide, calcium carbonate, zinc oxide, and powdered mica were tested in the ashers.

By comparison of the LEO erosion yield results and the asher results, for white Tedlar, the amount of LEO fluence that is represented by fluence in each asher environment could be determined. Based on the similar power law dependence for filled epoxy, polyurethane and white Tedlar in both asher environments as well as white Tedlar in the LEO environment, prediction of erosion yield in LEO can reasonably be based on taking erosion yield data in either the isotropic thermal energy plasma asher or and the hyperthermal directed asher. However, the hyperthermal directed asher has better potential to more accurately predict LEO erosion yields with fluence. A comparison between additional ash-filled polymers exposed in LEO and the same polymers in plasma ashers is needed to more accurately determine the ratio of LEO fluence to asher fluence.

The results of the investigation indicate significant durability benefits in that the erosion yields are not independent of fluence but drop with increasing fluence with power law dependence. The addition of only a small volume fraction ash (~11%) is sufficient to cause a reduction in erosion yield by more than order of magnitude when the fluence is increased by from 10<sup>20</sup> to 10<sup>22</sup> atoms/cm<sup>2</sup>.

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