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Comparison of the Atomic Oxygen Erosion Depth and Cone Height of Various Materials at Hyperthermal Energy

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Abstract

Atomic oxygen readily reacts with most spacecraft polymer materials exposed to the low Earth orbital (LEO) environment. If the atomic oxygen arrival comes from a fixed angle of impact, the resulting erosion will foster the development of a change in surface morphology as material thickness decreases. Hydrocarbon and halopolymer materials, as well as graphite, are easily oxidized and textured by directed atomic oxygen in LEO at energies of ~4.5 eV. What has been curious is that the ratio of cone height to erosion depth is quite different for different materials. The formation of cones under fixed direction atomic oxygen attack may contribute to a reduction in material tensile strength in excess of that which would occur if the cone height to erosion depth ratio was very low because of greater opportunities for crack initiation. In an effort to understand how material composition affects the ratio of cone height to erosion depth, an experimental investigation was conducted on 18 different materials exposed to a hyperthermal energy directed atomic oxygen source (~70 eV). The materials were first salt-sprayed to provide microscopic local areas that would be protected from atomic oxygen. This allowed erosion depth measurements to be made by scanning microscopy inspection. The polymers were then exposed to atomic oxygen produced by an end Hall ion source that was operated on pure oxygen. Samples were exposed to an atomic oxygen effective fluence of 1.0×10^{20} atoms/cm^2 based on Kapton H (DuPont) polyimide erosion. The average erosion depth and average cone height were determined using field emission scanning electron microscopy (FESEM). The experimental ratio of average cone height to erosion depth is compared to polymer composition and other properties.

1. Introduction

Most spacecraft polymer materials exposed to the low Earth orbital (LEO) environment will erode due to atomic oxygen exposure. If the atomic oxygen arrival comes from a fixed angle of impact, a change in surface texture will develop as the material oxidizes and becomes thinner.

Surface texture can change the optical reflectance of a material from specular to diffuse and increase the solar absorptance of opaque materials. Surface texture can also be the cause of crack initiation or tearing of thin film polymers that have become embrittled as a result of ionizing radiation. As a thin film polymer develops texture, the valleys of the texture can erode through the polymer before the polymer is completely eroded thus making the polymer only partially protective of materials beneath it. Thus, there is merit in understanding the degree of surface texturing that will occur for materials that erode by atomic oxygen attack. The amount of surface texturing increases as the atomic oxygen fluence increases. Thus, it is of interest to examine the cone height to erosion depth ratio for various materials to better understand this phenomenon. Table 1 shows that the ratio of cone height to erosion depth is quite different for different materials in LEO (ref. 1).

TABLE 1.—RATIO OF CONE LENGTH TO EROSION DEPTH AS A FUNCTION OF FLUENCE FOR VARIOUS MATERIALS EXPOSED TO DIRECTED LEO ATOMIC OXYGEN

Material	Ratio of average cone length to erosion depth	Space mission	Atomic oxygen fluence, atoms/cm^2
Pyrolytic graphite	0.60	EOIM III	2.3×10^{20}
Kapton H	0.28	EOIM III	2.3×10^{20}
FEP Teflon	0.07	LDEF	8.43×10^{21}

These changes in surface morphology, depending on atomic oxygen arrival, could have an impact on tensile properties possibly leading to increased surface crack initiation. Various material compositional properties are analyzed to determine their potential effect on the cone height versus erosion depth of the polymer samples exposed at hyperthermal energy.

2. Experimental Methods

Table 2 lists the materials, abbreviations, trade names, densities, LEO erosion yields, and atomic fractional composition that were tested (refs. 1 and 2). Table 2 also lists the hyperthermal (~70 eV) erosion yield relative to Kapton H for fifteen materials (ref. 2). The densities listed are from either literature values or actual density gradient column measurements. The LEO erosion yields listed in table 2 are those determined from in-space exposures on the Long Duration Exposure Facility (for polyimide Kapton H), or recently retrieved results from a four year exposure on the International Space Station (for all the remaining polymers) (refs. 3 and 4).

TABLE 2.—MATERIALS TESTED AND THEIR ABBREVIATIONS, TRADE NAMES, DENSITIES, AND EROSION YIELDS

						Atomic percent						
Material	Abbrev.	Trade name	Density, gm/cm^3	LEO erosion yield, $\times 10^{-24}$ cm^3/atom	~70 eV Erosion yield, relative to Kapton H (ref. 2)	H	C	N	O	F	S	Cl
Polyimide H (PMDA)	PI-H	Kapton H	1.427	3.0	1.00	26	56	5	13	0	0	0
Polyimide-HN (PMDA)	PI-HN	Kapton HN	1.435	2.81	1.03	26	56	5	13	0	0	0
Black polyimide	Black PI	Kapton CB	1.42	-----	0.90	--	--	--	--	0	0	0
Pyrolytic Graphite normal to beam	PG	-------------	2.22	0.415	0.58	0	100	0	0	0	0	0
Highly-Oriented Pyrolytic Graphite	HOPG	-------------	2.26	-----	-----	0	100	0	0	0	0	0
Fluorinated ethylene propylene	FEP	Teflon FEP	2.144	0.200	1.89	0	33	0	0	67	0	0
Ethylene Chlorotrifluoro-ethylene	ECTFE	Halar	1.676	1.79	1.59	33	33	0	0	25	0	8
Polycarbonate	PC	Lexan	1.123	4.29	1.07	42	48	0	9	0	0	0
Chlorotrifluoro-ethylene	CTFE	Aclar	2.133	0.831	2.86	0	33	0	0	50	0	17
Polyethylene	PE	Lupolen	0.918	3.97	1.06	67	33	0	0	0	0	0
Polyethylene Terephthalate	PET	Mylar A200	1.393	3.01	1.30	38	48	0	14	0	0	0
Polyoxymethylene	POM	Delrin	1.398	9.14	10.87	50	25	0	25	0	0	0
Polytetrafluoro-ethylene	PTFE	Teflon PTFE	2.150	0.142	1.86	0	33	0	0	67	0	0
Polyvinyl fluoride	PVF	Tedlar	1.379	3.19	1.10	50	33	0	0	17	0	0
Polyvinylidene Fluoride	PVDF	Kynar	1.762	1.29	1.42	33	33	0	0	33	0	0
Polytetrafluoro-ethylene ethylene Copolymer	ETFE	Tefzel ZM	1.740	0.961	1.08	33	33	0	0	33	0	0
Polymethyl methacrylate	PMMA	Lucite; Plexiglass	1.163	>5.60	----	53	33	0	13	0	0	0
Polystyrene	PS	Styron; Lustrex; Polystyrol	1.503	3.74	----	50	50	0	0	0	0	0

The eighteen samples were salt-sprayed with a fine mist of saturated sodium chloride which dried and provided sites protected from atomic oxygen erosion. This allowed the erosion depth to be distinguished from the cone development. The samples were held in place in the facility with a circular wire copper holder with attached mounting clips which held the samples at approximately equal distance radially around the axis of the beam. This assured that they all received approximately the same Kapton effective fluence (fig. 1). Various polymers and graphite were chosen because of potential spacecraft interest and usage. These materials have been studied in space (refs. 3 and 4), which allows for potential conclusions to be drawn.

The atomic oxygen effective fluence was measured using Kapton H polyimide based on a density of 1.42 gm/cm^3 and a LEO erosion yield of 3.0×10^{-24} cm^3/atom for fluence witness

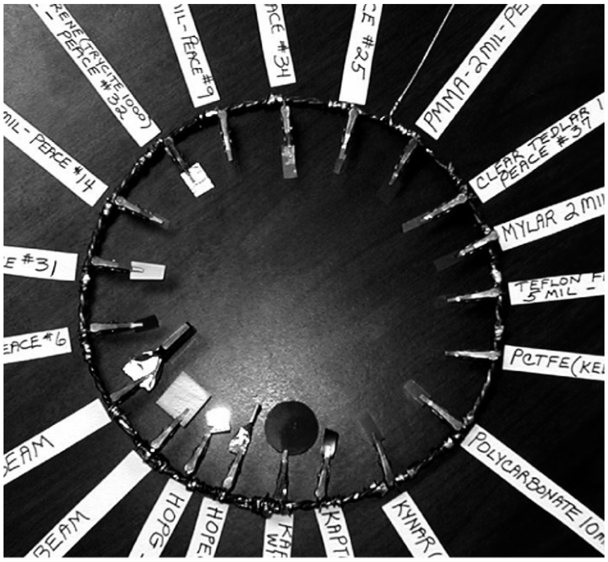

Figure 1.—Samples mounted on wire ring sample holder.

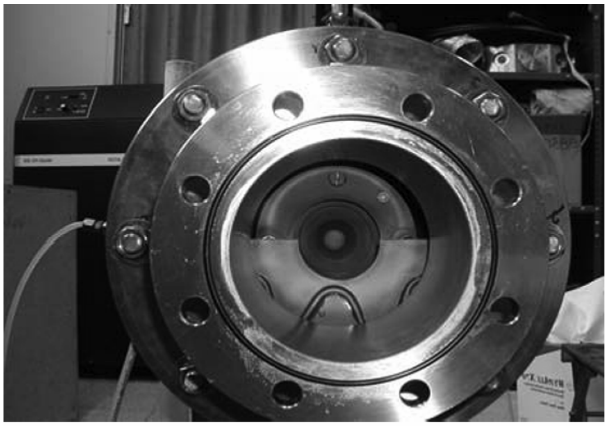

Figure 2.—End Hall ion source.

samples (ref. 5). The Kapton H polyimide effective fluence was determined by measuring its weight loss immediately after removal from vacuum desiccators after at least 48 hr of vacuum exposure at approximately 100 µm (1.33×10^5 Pa).

Hyperthermal energy oxygen ions were used to simulate LEO atomic oxygen. The exposure was performed at normal incidence using a Veeco Mark II end Hall ion source, shown in figure 2, operated on pure oxygen with a background pressure of 1×10^{-4} torr (1.33×10^{-2} Pa). The ion source was operated with an anode voltage of 90 eV, an anode current of 3.5 Amps, and a neutralizer current of 300 mAmps. These end Hall operating parameters produce an average ion energy of 70 eV (hyperthermal energy) which consisted almost exclusively of O_2^+ ions (ref. 6). The samples were exposed for an accumulated time of 4 hr. Erosion of the samples was visually evident due to the matte texture noted on each material.

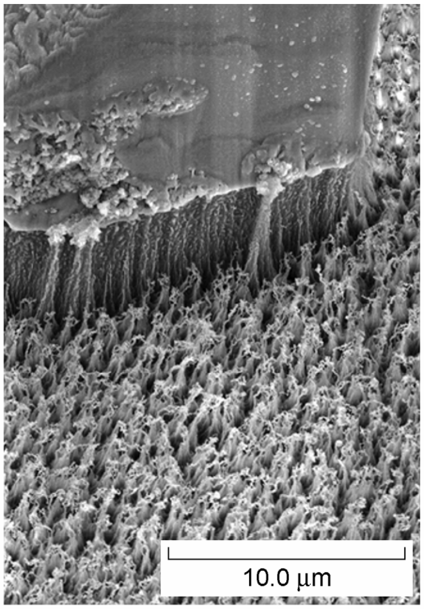

Figure 3.—FESEM image of protected PMMA exposed to a hyperthermal directed beam atomic oxygen source. The area protected from atomic oxygen is on top.

The samples were examined using a Hitachi S–4700 Field Emission Scanning Electron Microscope (FESEM). The samples were viewed at 6.0 kV using a mixed detector signal. A 45-degree tilt was used to facilitate viewing the erosion depth and cone height of the various samples. The erosion depth and cone heights were measured using Quartz PCI 4.0 software calibrated to the Hitachi FESEM photo capture process. Each material was optimized for measuring the cone height and erosion depth by taking different magnifications at three different salt-protected sites. Each photo then was measured using the Quartz program at ten different locations for the cones and ten different locations for the buttes. A typical photo, shown below in figure 3, allows comparison of the erosion depth and cone height. The results were then tabulated and are presented in this report.

3. Results and Discussion

The cone height, erosion depth and the ratio results are tabulated in table 3. The standard deviation is included in these calculations. The samples were exposed to a Kapton H atomic oxygen fluence of 1.0×10^{20} atoms/cm^2.

It is generally assumed that the lower the cone height to erosion depth ratio, the more effective the polymer is at hindering tall cone structure development while still allowing bulk erosion to occur. When comparing the ratios among the various polymer types, it is observed that the more halogenated polymers have a lower ratio of cone height to erosion depth compared to less halogenated or non-halogenated polymers. Polymers with oxygen functionalities

TABLE 3.—RATIO OF CONE HEIGHT TO EROSION DEPTH FOR THE VARIOUS MATERIALS

Materials	Average cone height (μm)	Standard deviation of cone height (μm)	Average erosion depth (μm)	Standard deviation of erosion depth (μm)	Ratio of cone height to erosion depth	Standard deviation of ratio
Fluorinated ethylene Propylene, Teflon FEP	0.478	0.094	2.853	0.238	0.168	0.213
Polyoxymethylene, Delrin	3.742	0.779	20.597	1.918	0.182	0.228
Chlorotrifluoroethylene, Aclar	0.758	0.099	3.732	0.417	0.203	0.172
Polytetrafluoroethylene, Teflon PTFE	0.723	0.156	3.547	0.465	0.204	0.252
Polyvinylidene fluoride, Kynar	0.530	0.085	2.164	0.221	0.245	0.190
Ethylene chlorotrifluoroethylene, Halar	0.711	0.096	2.841	0.265	0.250	0.164
Polycarbonate, Lexan	0.523	0.065	2.023	0.157	0.259	0.146
Polyethylene terephthalate, Mylar	0.568	0.104	2.113	0.232	0.269	0.213
Polytetrafluoroethylene ethylene copolymer, Tefzel	0.523	0.077	1.928	0.360	0.271	0.237
Polymethyl methacrylate, Lucite	0.927	0.141	3.191	0.266	0.290	0.173
Polyvinyl fluoride, Tedlar	0.656	0.149	2.238	0.327	0.293	0.270
Polystyrene, Styron	0.524	0.092	1.728	0.214	0.303	0.215
Polyimide H (PMDA), Kapton H	0.595	0.101	1.935	0.180	0.307	0.194
Black polyimide, Kapton CB	0.565	0.121	1.824	0.192	0.310	0.239
Polyimide-HN (PMDA), Kapton HN	0.545	0.089	1.724	0.239	0.316	0.214
Polyethylene, Lupolen	0.430	0.074	1.352	0.171	0.318	0.214
Pyrolytic graphite, normal to beam, PG	0.250	0.049	0.453	0.066	0.553	0.243
Highly-oriented pyrolytic graphite, normal to beam, HOPG	0.131	0.021	0.183	0.022	0.716	0.203

generally show a ratio of cone height to erosion depth that is less than non-halogenated and non-oxygen containing hydrocarbons and carbon but greater than the halogenated polymers. Delrin, a polymer with oxygen functionality has the highest ratio for polymers with oxygen functionality, actually in the same range as the halogenated polymers. In comparing the various polymer materials, it appears to be a trend that the ratio of cone height to erosion depth correlates with the polymer functionality, being lowest for halogenated polymers, intermediate for oxygen functionalities and highest for simple hydrocarbons and carbon. Density and LEO (~4.5 eV) atomic oxygen erosion yield values, shown in table 2, were also examined to determine if they correlate with the ratio of cone height to erosion length. However, no consistent trends are evident in correlating the cone height to erosion depth ratio with either of these material properties.

Banks et al. (ref. 2) also utilized fifteen of the materials used in this report in a study of erosion yields at hyperthermal energies (~70 eV). The ratios of cone height to erosion depth shown in table 3 were compared to hyperthermal energy atomic oxygen erosion yield values (ref. 2), shown in table 2, and are plotted in figure 4 indicating a gradual reduction cone height to erosion depth with increasing end Hall erosion yield. Atomic oxygen erosion is a spatially independent erosion process similar to snow accumulation or the fluence of cosmic rays arriving at a surface. As such, it should obey Poisson statistics where the standard deviation in erosion (or cone height) should increase as the square root of the erosion (fluence times erosion yield) (ref. 7). Thus there should be a linear relationship between cone height/erosion depth and 1/(end Hall erosion yield)$^{0.5}$ for a constant fluence.

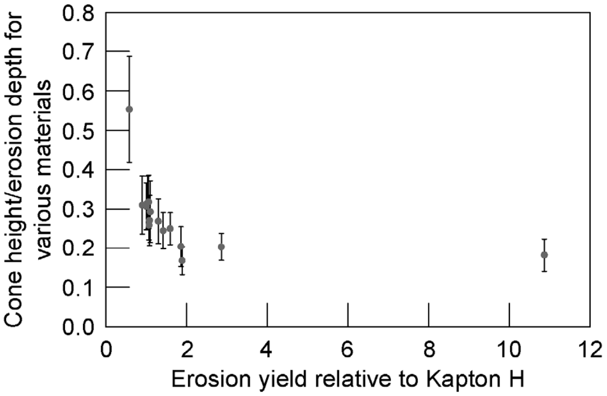

Figure 4.—Graph of the cone height/erosion depth versus the hyperthermal energy erosion yield relative to Kapton H for 15 different materials.

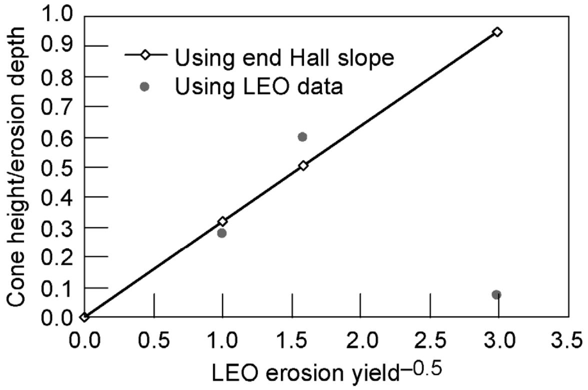

Figure 6.—Cone height/erosion depth versus 1/(erosion yield)$^{–0.5}$ for LEO data for Kapton H, pyrolytic graphite and FEP Teflon.

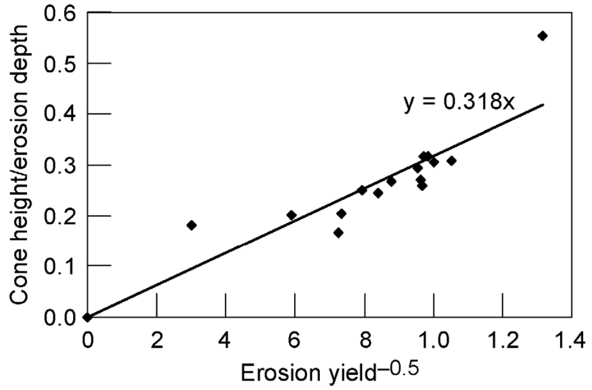

Figure 5.—Cone height/erosion depth versus 1/(erosion yield)$^{–0.5}$ for end Hall ion source data.

Using this end Hall data relative to Kapton H, a graph was constructed with cone height/erosion depth versus 1/(end Hall erosion yield)$^{0.5}$. This graph with a trend line is shown in figure 5. It indicates a linear relationship for most polymers with some exception for Delrin and pyrolytic graphite. Based on figure 5, the cone height can be predicted as:

$$\text{Cone height} = (0.318)\ (\text{erosion depth})\ (\text{erosion yield})^{-0.5} \quad (1)$$

or

$$\text{Cone height} = (0.318)\ (\text{fluence})\ (\text{erosion yield})^{0.5} \quad (2)$$

where all quantities are in cm units. When using the above formulas, it is important to use the erosion yield of the material in its relevant environment (such as hyperthermal, LEO or thermal energy atomic oxygen).

The slope of the trend line in figure 5 was transferred over to known data (ref. 1) from LEO for three materials to produce the graph shown in figure 6. This plot also shows good correlation in predicting the cone height over erosion depth with the exception of FEP where very small cones formed in spite of reasonable fluence. The exception for FEP may be caused by an erosion yield for fluoropolymers that is solar flux and temperature dependent unlike most other polymers.

4. Summary

Predicting in-space atomic oxygen erosion phenomena based on ground laboratory testing is valuable in understanding expected LEO changes in optical, mechanical, and atomic oxygen protection properties of materials. The development of surface texture expressed as the ratio of cone height to erosion depth is not a simple constant for all materials. The ratio of cone height to erosion depth was evaluated for eighteen different polymers exposed to a hyperthermal oxygen ion beam using an end Hall oxygen ion source operated at an average energy of 70 eV. Several compositional factors were examined for trends. In correlating the trend with polymer composition, it was observed that the halogenated polymers showed the lowest ratios of cone height to erosion depth, simple hydrocarbons and carbon showed the highest ratio, and polymers with oxygen functionalities generally showed ratios higher than halogenated hydrocarbons and lower than simple hydrocarbons and carbon. Directed atomic oxygen texturing appears to obey Poisson statistics where the texture grows as the square root of the erosion. The hyperthermal erosion yield of fifteen materials was found to yield a linear relationship between cone height/erosion depth and 1/(end Hall erosion yield)$^{0.5}$ for most materials. This linear relationship was found to be consistent with ratios of cone height to erosion depth data for Kapton H and pyrolytic graphite with the exception of FEP Teflon. Thus, for most all polymers, with the exception of fluoropolymers such as FEP Teflon, prediction of texture development with atomic oxygen fluence appears possible if one knows the erosion yield of the material.

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