



A Space Experiment to Measure the Atomic Oxygen Erosion of Polymers and Demonstrate a Technique to Identify Sources of Silicone Contamination

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A SPACE EXPERIMENT TO MEASURE THE ATOMIC OXYGEN EROSION OF POLYMERS AND DEMONSTRATE A TECHNIQUE TO IDENTIFY SOURCES OF SILICONE CONTAMINATION

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ABSTRACT

A low Earth orbital space experiment entitled, "Polymers Erosion And Contamination Experiment," (PEACE) has been designed as a Get-Away Special (GAS Can) experiment to be accommodated as a Shuttle in-bay environmental exposure experiment. The first objective is to measure the atomic oxygen erosion yields of ~40 different polymeric materials by mass loss and erosion measurements using atomic force microscopy. The second objective is to evaluate the capability of identifying sources of silicone contamination through the use of a pin-hole contamination camera which utilizes environmental atomic oxygen to produce a contaminant source image on an optical substrate.

INTRODUCTION

Atomic Oxygen Erosion Yield Measurements

Since the advent of low Earth orbital (LEO) spacecraft, such as the U.S. Space Shuttle, the Russian Mir Space Station and International Space Station, the oxidative and erosive effects of low Earth orbital atomic oxygen have had significant consequences upon the design and durability of spacecraft materials. Atomic oxygen in low Earth orbit is formed by photodissociation of diatomic oxygen by short wavelength ultraviolet radiation from the sun in an environment where the mean free path is sufficiently large that the probability of recombination or the formation of ozone is improbable. As a result, spacecraft orbiting the Earth run into the atomic oxygen with ram energies of ~4.5 eV. Both the impact energy as well as the high reactivity of atomic oxygen results in oxidative erosion of most hydrocarbon polymer materials. In-space data to measure the sensitivity of materials to atomic oxygen erosion are commonly quantified in terms of the atomic oxygen erosion yield, which is the volume of material which is removed per incident oxygen atom. Measurements of atomic oxygen erosion in space have been performed on numerous spacecraft by many investigators (refs. 1 to 3).

The atomic oxygen erosion yield of most polymeric materials (with the exception of silicones and materials containing metal atoms which produce nonvolatile oxides), is typically in the range of 0.337×10^{-24} to 6.3×10^{-24} cm³/atom (ref. 3). This wide range of atomic oxygen erosion yields spanning more than an order of magnitude does not give great comfort to spacecraft designers who need to know, more accurately, the durability of typical spacecraft materials. Because in-space testing is expensive with available space being limited, many materials have not been tested in space. In addition, new materials continue to emerge which are in need of atomic oxygen erosion yield characterization for them to be accepted for use by spacecraft designers. Ground laboratory testing has been used with varying degrees of success to assist in providing prediction of in-space durability (ref. 2). In addition

to ground-based testing, analytical models have been developed which are useful in predicting in-space atomic oxygen erosion yields based on the content and structure of hydrocarbon polymers (ref. 4).

The determination of atomic oxygen erosion yields from in-space exposure data has been dominated by the measurement of mass loss or thickness loss of samples exposed to either a fixed or a sweeping direction arrival atomic oxygen. Although the most reliable erosion yield data have been dominated by high fluence space experiments such as the Long Duration Exposure Facility (LDEF), which received fixed direction arrival of atomic oxygen, (ref. 5), most LEO spacecraft have surfaces which are exposed to atomic oxygen in a manner which sweeps at various angles across the surface during each orbit. Such sweeping arrival may have influences on the atomic oxygen erosion yield of the material. Thus, it is desirable to measure the atomic oxygen erosion yield for materials exposed to atomic oxygen arriving from a variety of directions and to be able to do so in a manner that reasonable uncertainty exists over the range of expected erosion yields. It is the intent of this proposed experiment to be able to make such measurements for a wide variety of polymeric materials. These results can then serve as a materials durability database as well as contribute to erosion yield predictive modeling techniques. This database will enhance the confidence in predictive techniques with the goal that accurate results could be achieved without requiring in-space characterization of every new material.

Silicone Contamination and Atomic Oxygen Interactions

The exposure of bulk silicones to atomic oxygen results in the oxidation of methyl groups which causes shrinkage and hardening of the surface of the silicones, and surface conversion of silicone to silica (refs. 6 and 7). Spacecraft exposed to the LEO environment for long durations such as the LDEF and Mir Space Station have indicated significant deposits of silica on nonsilica containing surfaces (ref. 5). This is a result of volatile silicone species depositing on surfaces which are exposed to atomic oxygen resulting in the conversion of silicone to silica (ref. 5). Depending on the composition of such oxidized silicone contaminants, UV darkening of the contaminant can occur, thus changing the solar absorptance properties which has the potential for altering the thermal balance of the spacecraft or diminishing the output of photovoltaic arrays. Perhaps what is most disturbing in many spacecraft experiments is that the source of the silicone contamination is not well understood. It is the intent of this experiment to demonstrate a technique for imaging sources of the silicone contamination.

SPACE EXPERIMENT

Objective Summary

The Polymer Erosion And Contamination Experiment (PEACE) is a collaborative research effort between Hathaway Brown School and the NASA Glenn Research Center under the sponsorship of the American Chemical Society (ACS). Project planning began in 1998 when ACS afforded Hathaway Brown School the opportunity to conduct an experiment using a Get-Away Special (GAS Can) reservation to conduct an experiment in the space shuttle bay. The PEACE objectives were developed to address atomic oxygen interaction issues related to polymer erosion and silicone contamination for spacecraft operating in the low Earth orbital (LEO) environment. The objectives of the experiment are to: (1) measure the atomic oxygen erosion yield of ~40 different polymeric materials, and (2) to evaluate the feasibility of identifying sources of silicone contamination using pin-hole contamination cameras which utilize atomic oxygen to produce images of the sources of contamination.

Approach

The overall drawing of the PEACE GAS Can experiment is shown in figure 1. To accomplish the first objective of the experiment, two types of thin polymer samples are to be used to allow for mass loss measurements as well as recession measurements to be made. The mass loss measurements will be used to calculate the atomic oxygen erosion yield of the samples, E_s , from the following equation.

$$E_s = \frac{\Delta M_s}{A_s \rho_s F_k} \quad (1)$$

where

ΔM_s mass loss of polymer sample
 A_s area of polymer sample
 ρ_s density of sample
 F_k atomic oxygen fluence measured by Kapton H polyimide witness samples

The atomic oxygen fluence measured by Kapton H polyimide witness samples is:

$$F_k = \frac{\Delta M_k}{A_k \rho_k E_k} \quad (2)$$

where

ΔM_k mass loss of Kapton H witness sample
 A_k area of Kapton H witness sample
 ρ_k density of Kapton H sample = 1.42 g/cm³ for Kapton Type H
 E_k erosion yield of Kapton H polyimide = 3.0 × 10⁻²⁴ cm³/atom (ref. 3)

If the atomic oxygen fluence is predicted using the same sample size as the polymer test sample, then $A_s = A_k$ and the equation of the atomic oxygen erosion yield of the sample polymers is then given by:

$$E_s = \frac{\Delta M_s \rho_k E_k}{\Delta M_k \rho_s} \quad (3)$$

Because most polymers absorb water, accurate weight measurements require a consistent state of hydration to accurately determine the mass loss due to atomic oxygen exposure in space. To allow accurate measurements to be made, mass loss measurements will be made after vacuum dehydration of samples for at least 48 hr prior to the exposure to air for immediate weighing. This results in the samples having minimal variation in weight because of their very low state of hydration.

Because the atomic oxygen fluence to which the samples will be exposed is not presently known, an additional more sensitive method will be used to measure atomic oxygen erosion yields by means of measurements of the recession of the polymer surfaces with respect to unexposed portions of the specimens. This will be accomplished by using either salt spray on polymers to deposit sodium chloride to protect sites on the polymer samples or by dusting with small mica flakes on the samples prior to flight. The salt or mica flakes, which are atomic oxygen durable, will be removed after flight to allow the use of atomic force microscopy to measure the erosion step between the protected and the exposed area of the polymers. This technique will also be used on the Kapton H atomic oxygen fluence witness samples to allow accurate atomic oxygen fluence measurements in the event of a low fluence flight. The atomic oxygen erosion yield of samples can be calculated from the atomic force microscopy recession data by the following equation:

$$E_s = \frac{X_{s,0} - X_{s,1}}{F_k} \quad (4)$$

where

$X_{s,0}$ initial height of sample surface
 $X_{s,1}$ final height of sample surface
 F_k atomic oxygen fluence measured by Kapton H polyimide witness samples

For these measurements, the atomic oxygen fluence is given by equation (5):

$$F_k = \frac{X_{k,0} - X_{k,1}}{E_k} \quad (5)$$

where

- $X_{k,0}$ initial height of Kapton H fluence witness sample
- $X_{k,1}$ final height of Kapton H fluence witness sample
- E_k erosion yield of Kapton H sample = 3×10^{-24} cm³/atom (ref. 3)

Thus, the erosion yield of the polymer samples is given by

$$E_s = \frac{(X_{s,0} - X_{s,1})E_k}{X_{k,0} - X_{k,1}} \quad (6)$$

The second objective of the experiment involves the use of several sources of silicone contamination on one side of the GAS Can with pin-hole contamination cameras on the opposite side of the GAS Can (see fig. 1). The arrangement of silicone contamination samples allows atomic oxygen to impinge on the samples which are facing up but protects from atomic oxygen exposure the samples which are facing down. Both atomic oxygen exposed and non-exposed samples will be in view of contamination pin-hole cameras on the opposite side of the GAS Can. The silicones evolve from the samples, pass through an orifice in the pin-hole camera, and then deposit on iridium-coated fused silica substrates which are also exposed to arriving atomic oxygen. Atomic oxygen impinging upon the depositing silicone contamination would convert the silicone to silica which would gradually increase in thickness with exposure duration in space. Upon retrieval from space, the silica contaminated iridium-coated fused silica substrates will then be optically characterized by means of scanning variable angle spectroscopic ellipsometry by Dr. John Woollam at the University of Nebraska at Lincoln, Nebraska to produce images of the contaminant sources. The technique is sufficiently sensitive that monolayer deposits are readily measurable. By having some silicone samples being exposed to atomic oxygen and others protected, it may be possible to determine what role, if any, atomic oxygen plays in the source of silicone contamination. If the technique proves viable, such contamination pin-hole cameras could then be used to look into the shuttle bay or on large space structures such as International Space Station to identify sources of silicone contamination.

Apparatus

The GAS Can for this experiment consists of an open ended cylinder to allow exposure of the samples and materials to occupy the space which is ~58 cm in diameter. An overall assembly drawing of the experiment shown as an exploded view is given in figure 2. The polymer samples for mass loss measurements will be 2.54 cm in diameter with desired thickness on the order of 0.05 mm thick. The polymer samples for erosion depth measurements will be only 1.27 cm in diameter and of similar thickness. Figure 3 shows a top view of the polymer sample tray and an exploded cross sectional view indicating how the sample is sandwiched between the top and bottom plate of the sample tray. There are 60 locations of both the large and small sized samples. This allows 40 polymer samples to be measured with respect to erosion yield by both mass loss and surface recession with an additional 20 Kapton atomic oxygen fluence witness samples of each size to be distributed over the sample tray. The Kapton fluence witness samples will allow measurement of the spatial variation of atomic oxygen fluence which may occur as a result of Shuttle-caused shadowing of the incoming atomic oxygen if it is arriving at small angles with respect to the plane of the sample tray surface.

To obtain a qualitative understanding of the amount of atomic oxygen flux arriving from various directions two atomic oxygen pin-hole cameras will be used. Silver coated glass hemispheres will be used in conjunction with pin-holes in orifice plates to make pin-hole cameras which allow atomic oxygen to oxidize silver to produce an image of the arrival direction of atomic oxygen during the entire space exposure. Figure 4 shows a drawing of one of the two atomic oxygen pin-hole cameras to be used in the experiment. The inside diameter of the glass hemispheres is

4.55 cm and the diameter of the orifices will be 0.51 and 1.27 mm in diameter. The purpose of having two different orifice diameters for the two atomic oxygen pin-hole cameras is to be confident that at least one of the cameras will have sufficient atomic oxygen fluence arrival to see the arrival direction and if a high fluence exposure occurs, the smaller pin-hole will allow more accurate angular determination.

The silicone contamination portion of the experiment is purposely mounted below the plane of the polymer samples to minimize contamination on the surface of the polymer samples. As can be seen from figure 1, there will be two contamination source assemblies each containing six silicone samples. Three of the samples will be exposed at $\sim 45^\circ$ with respect to the axis of the GAS Can to allow atomic oxygen to impinge upon the sample and to allow a line of sight to the contamination camera at the opposite side of the GAS Can cylinder. An additional three samples are facing downward such that incident atomic oxygen would be shielded from the samples, thus each contamination camera will view six samples. Figures 5(a) and (b) show drawings of a typical assembled contamination camera and an exploded view of the camera. The contamination cameras allow a small portion of the contaminant flux to enter through an orifice plate and deposit on an iridium-coated fused silica substrate. This substrate is oriented at 45° with respect to the axis of the GAS Can. The camera tube is open to allow arriving atomic oxygen flux to convert deposited silicones to silica thus immobilizing the surface contaminants and allowing their gradual build up in thickness as normally occurs in space. The small orifice (0.5 mm diameter) allows for images of the sources of contamination to form on the iridium-coated fused silica substrate.

Exposure Considerations

The atomic oxygen fluence needed for this experiment depends upon the uncertainty deemed acceptable for the erosion yield of the polymers tested and the technique used to compute these erosion yields (mass loss or surface recession measured by atomic force microscopy). For the mass loss samples, if one assumes that a typical sample will have an erosion yield and density of approximately that of Kapton H polyimide, then based on equations (1) to (3) the fractional uncertainty measured in terms of the standard deviation in erosion yield of the sample δE_s , divided by the erosion yield of the sample, E_s , is given by equation (4) where δ represents the standard deviation in their respective quantities.

$$\frac{\delta E_s}{E_s} = \left[2 \left(\frac{\delta \Delta M_k}{E_k A_k \rho_k F_k} \right)^2 + 2 \left(\frac{\delta \rho_k}{\rho_k} \right)^2 + \left(\frac{\delta E_k}{E_k} \right)^2 \right]^{1/2} \quad (7)$$

Where

E_k	erosion yield of Kapton H polyimide = 3.0×10^{-24} cm ³ /atom (ref. 3)
δE_k	7.4×10^{-26} cm ³ /atom (ref. 3)
ρ_k	1.42 gm/cm ³
$\delta \rho_k$	0.0074 gm/cm ³
A_k	5.067 cm ²
F_k	atomic oxygen fluence measured by Kapton H polyimide witness coupons (atoms/cm ²)
$\delta \Delta M_k$	1.28×10^{-5} g

A similar equation can be developed for the fractional erosion yield uncertainty for the surface recession measurement samples.

The standard deviation in sample surface height is believed to increase with atomic oxygen exposure proportional to the square root of the atomic oxygen fluence in a manner following Poisson statistics. Thus, through experimental measurement, if one knows the standard deviation in sample height, $\delta X_{k,0}$, after a known atomic oxygen fluence, $F_{k,0}$, in space, one can predict the standard deviation $\delta X_{k,t}$, at an arbitrary fluence, F_k . Thus:

$$\delta X_{k,t} = \delta X_{k,0} \sqrt{\frac{F_k}{F_{k,0}}} \quad (8)$$

One can use equations (6) and (8) along with data from a previous flight experiment to compute the following fractional uncertainty in erosion yield measured in terms of standard deviation of the erosion yield divided by the erosion yield as shown below:

$$\frac{\delta E_s}{E_s} = \left[2 \left(\frac{\delta X_k}{E_k F_k} \right)^2 + 2 \left(\frac{\delta X_{k,0}}{E_k F_k} \right)^2 \frac{F_k}{F_{k,0}} + \left(\frac{\delta E_x}{E_k} \right)^2 \right]^{1/2} \quad (9)$$

where

$F_{k,0}$	4.8×10^{19} atoms/cm ²
$\delta X_{k,0}$	2.28×10^{-6} cm
δX_k	standard deviation in sample height for unexposed Kapton H polyimide = 8.4×10^{-8} cm
E_k	3.0×10^{-24} cm ³ /atom
δE_k	7.4×10^{-26} cm ³ /atom
F_k	atomic oxygen fluence measured by Kapton H polyimide witness coupons atoms/cm ²

The numbers used for the variables are based on a combination of literature values (ref. 3), experimental measurements and statistical studies performed by the authors.

Based on the mass loss and recession measurement technique, the fractional uncertainty as a function of atomic oxygen fluence can be plotted as shown in figures 6(a) and (b) for mass loss and recession measurements. Although one can feel most comfortable in interpreting mass loss data for samples, the standard deviation in erosion yield for a typical polymer material rises above 10 percent for atomic oxygen fluences less than 1×10^{19} atoms/cm², whereas use of atomic force microscopy recession measurements has only a 6 percent uncertainty at the same fluence. Based on these two plots it appears that useful atomic oxygen erosion yield data will result provided the mission has an atomic oxygen fluence in excess of 1×10^{19} atoms/cm². For both measurement techniques the minimal uncertainty for high fluences is ~3 percent for fluences in excess of 5×10^{19} cm²/atom.

Materials

The 40 polymers currently being considered to be measured for atomic oxygen erosion yield are listed in table 1. These materials represent a variety of polymers currently being used, or considered for use, in space as well as polymers which were specifically chosen to assist in providing in-space data to correlate with computational methods to predict erosion yields.

The silicone samples will consist of silicones typically used in space of varying degrees of volatility. The silicones currently being considered consist of dimethylethoxysilane (which is sprayed on the Shuttle tiles to increase their water repellency), DC-93-500 (typically used to attach cover glass to solar cells) and CV-1147 (which is used between the solar cells on the International Space Station).

CONCLUSION

The Polymers Erosion And Contamination Experiment (PEACE) has been designed to be accommodated in a Get-Away Special (GAS Can) experiment as an open can experiment in the Shuttle bay. Approximately 40 polymers will be evaluated to determine their atomic oxygen erosion yield by mass loss measurements as well as erosion depth through atomic force microscopy. The experiment will contain atomic oxygen pin-hole cameras to image the arrival flux in a qualitative manner impinging upon the samples. The experiment also contains 2 contamination cameras which are designed to image sources of contamination from silicone sources located below the polymeric sample plane. The development of the pictures of the sources of contamination will be achieved by first fixing the contaminants by exposure to atomic oxygen to convert silicone to silica. Scanning variable angle spectroscopic ellipsometry will then be used to measure the very small thicknesses of contaminants which should deposit in a manner that images their sources. The atomic oxygen fluence necessary to achieve ~3 percent uncertainty in results appears to be 5×10^{19} atoms/cm².

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TABLE I.—POLYMER SAMPLES FOR EROSION YIELD CHARACTERIZATION

	Material	Abbreviation	Trade Names
1	Acrylonitrile butadiene styrene	ABS	Cyclocac; Lustran
2	Cellulose acetate	CA	Cellidor; Tenite
3	Cellulose nitrate	CN	Clelluloid; Xylonite
4	Chlorotrifluoroethylene	CTFE	Kel-F
5	Crystalline polyvinylfluoride w/white pigment	PVF	White Tedlar
6	Diallyl diglycol and triallyl cyanurate	ADC	CR-39
7	Epoxide or epoxy	EP	Epoxy resin 5208
8	Ethylene/propylene/diene	EPTR,EPDM	Nordel; Keltan
9	Tetrafluorethylene-ethylene copolymer	ETFE	Tefzel
10	Ethylene vinyl acetate copolymer	EVAC/EVA	Evatane
11	Ethylene vinyl alcohol copolymer	EVAL/EVOH	Clarene; Eval
12	Fluorinated ethylene propylene	FEP/TFE-HFP	Teflon FEP
13	Graphite	HOPG	Pyrolytic Graphite
14	Halar ethylene-chlorotrifluoroethylene	ECTFE	Halar
15	Polyimide (BPDA)	PI	Upilex-S
16	Melamine formaldehyde	MF	Melmex; Melopas
17	Phenol formaldehyde	PF	Bakelite; Stermite
18	Poly-(p-phenylenephthalamide)	PPTA	Kevlar 49, Twaron
19	Polyamide 6 or nylon 6	PA 6	Akulon K; Ultramid
20	Polyamide 66 or nylon 66	PA 66	Maranyl; Zytel
21	Polyacrylonitrile	PAN	Acrilan; Barex; Orlon
22	Polybenzimidazole	PBI	Celazole
23	Polycarbonate	PC	Lexan; Makrolon
24	Polychlorotrifluoroethylene	PCTFE	Hostafion C2; Kel-F
25	Polyethylene	PE	Alathon; Lupolen
26	Polyetheretherkeytone	PEEK	Victrex PEEK
27	Polyethylene terephthalate	PET	Mylar, Arnite A; Techster E
28	Polyimide (PMDA)	PI	Vespele; Kapton H
29	Polymethyl methacrylate	PMMA	Plexiglas; Lucite
30	Polypropylene	PP	Profax; Propathene
31	Polyoxymethylene:acetal;polyformaldehyde	POM	Delrin; Celcon
32	Polystyrene	PS	Lustrex; Polystyrol
33	Polysulphone	PSU	Udel; Ultrason/S
34	Polytetrafluoroethylene	PTFE	Fluon; Teflon; Halon
35	Polyvinylidene chloride copolymers	PVDC	Saran
36	Polyvinylidene fluoride	PVDF	Kynar
37	Polyvinyl fluoride	PVF	Tedlar
38	Pyrrone	PR, PMDA-DAB	Pyrrone
39	Urea formaldehyde	UF	Beetle, Scarab
40	Polyphenylene isophthalate	PPPA	Nomex

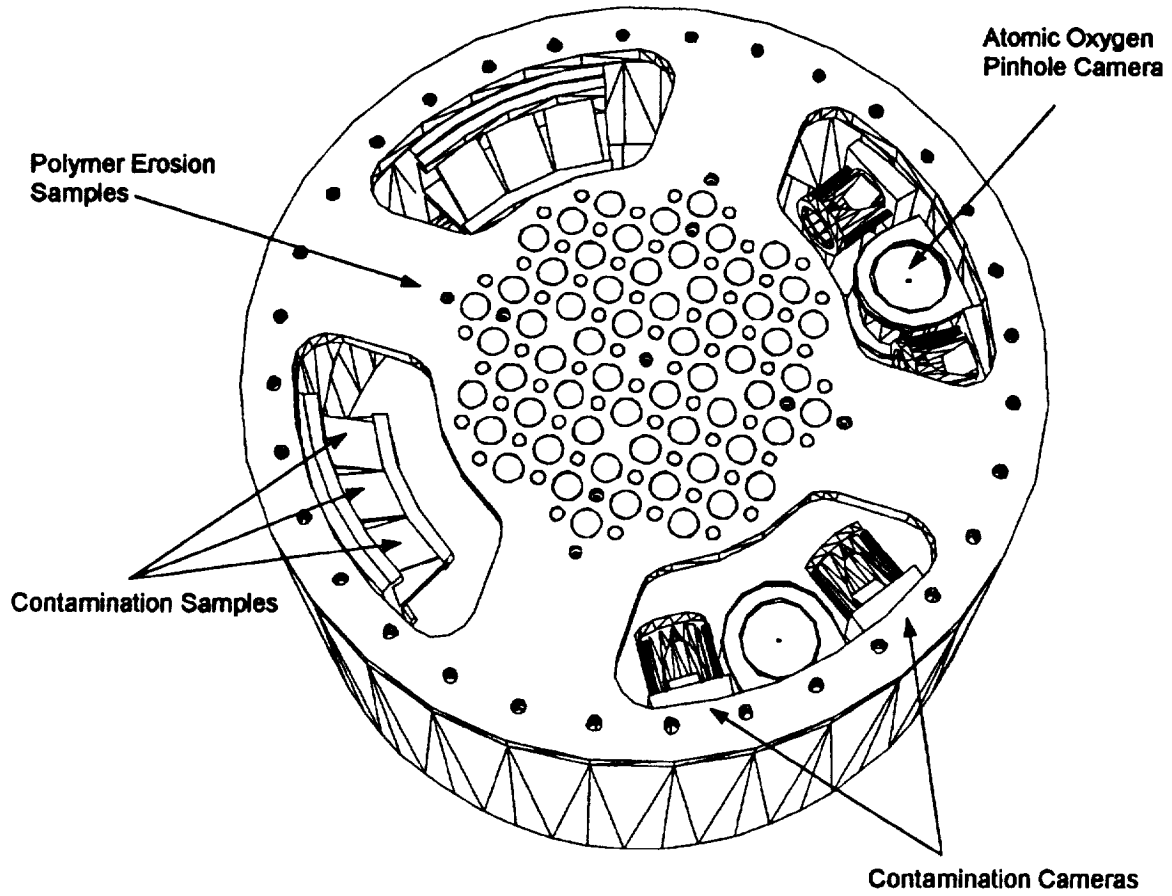


Figure 1.—Drawing of the Polymer Erosion and Contamination (PEACE) GAS can experiment.

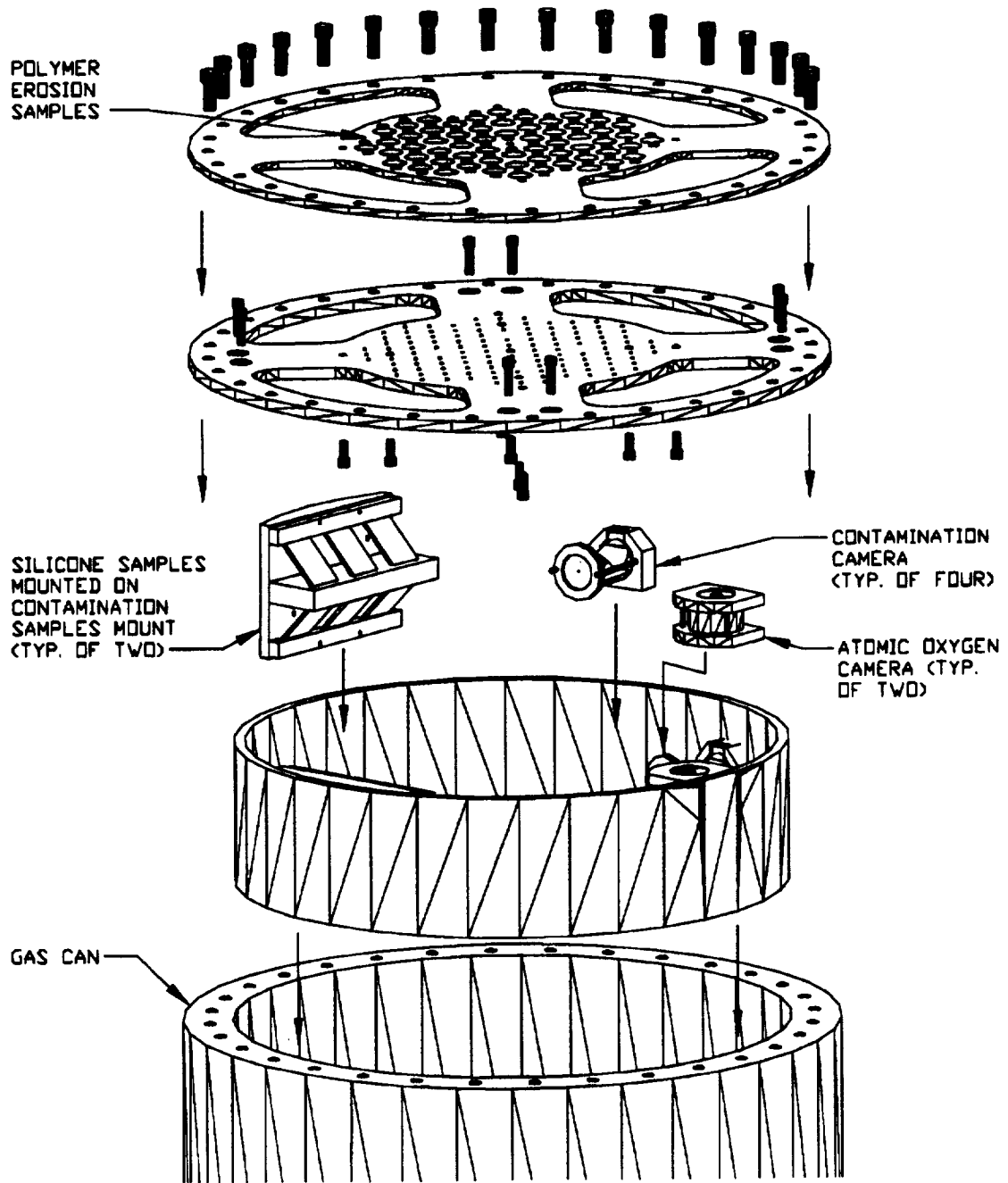
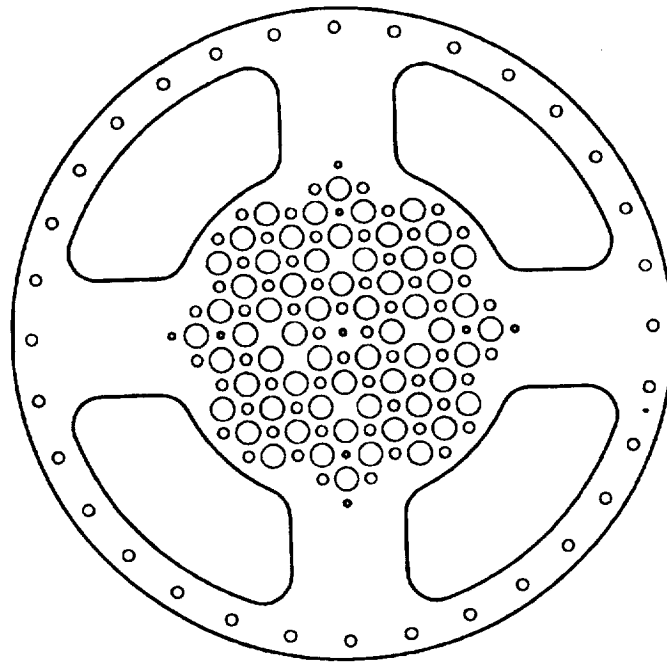
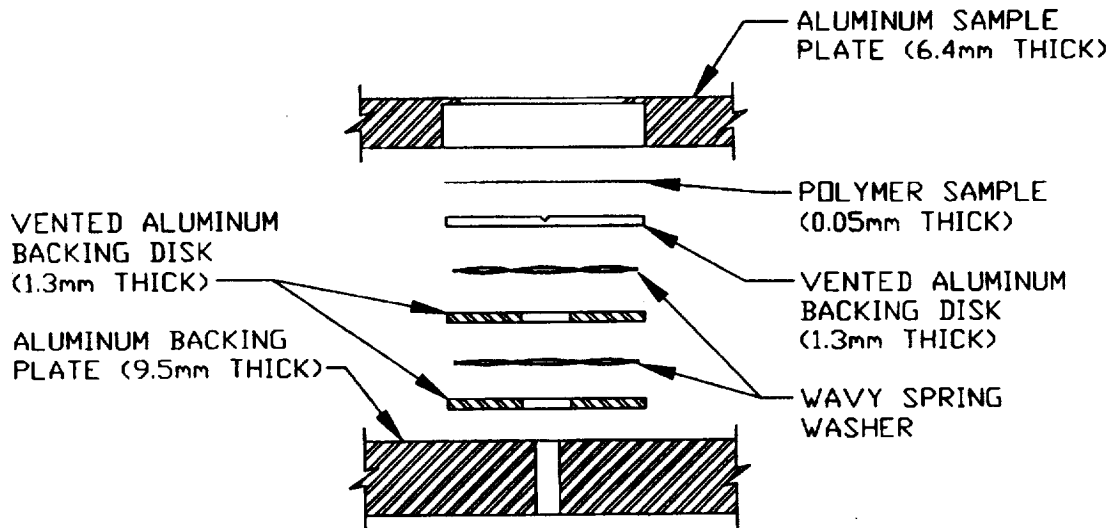


Figure 2.—Exploded view drawing of the experiment.



(a) Face view of sample tray.



(b) Exploded section view of typical sample.

Figure 3.—Polymer sample tray.

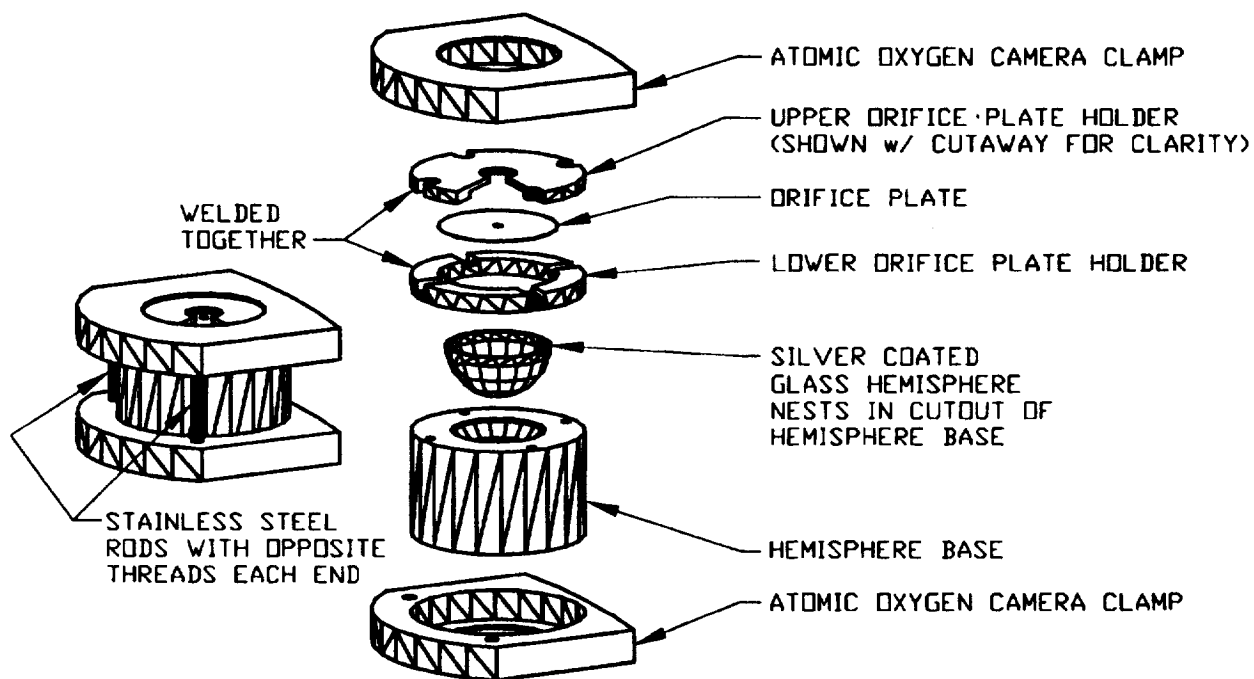
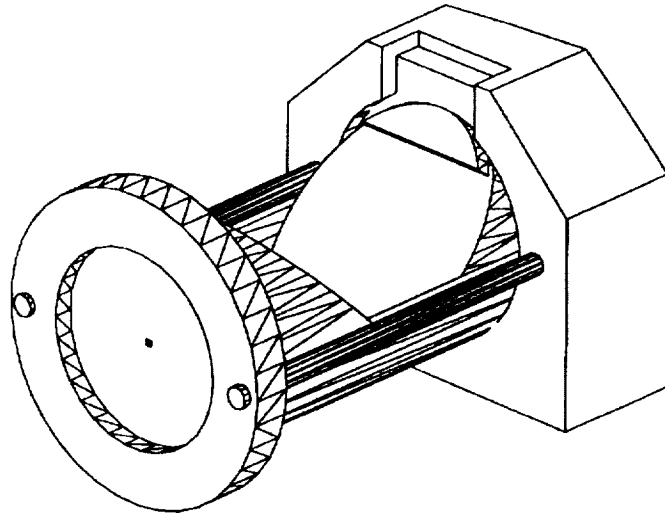
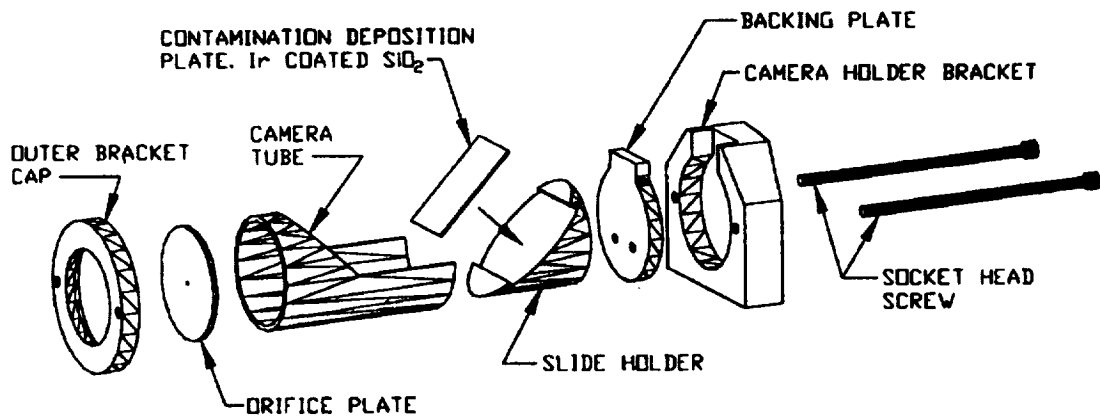


Figure 4.—Drawing of one of the atomic oxygen pinhole cameras to be used in the experiment.



(a) Perspective assembly view.



(b) Exploded view.

Figure 5.—Silicone contamination camera.

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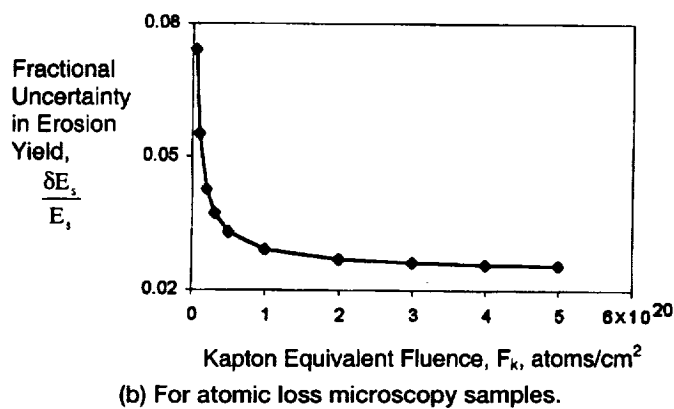
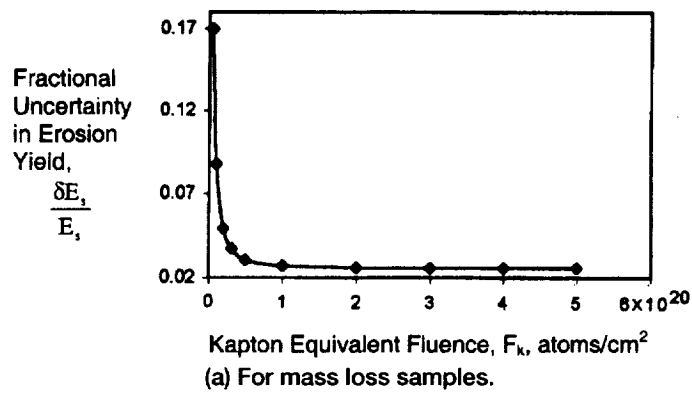


Figure 6.—Fractional uncertainty of erosion yield measured in terms of standard deviation and erosion yield divided by erosion yield as function of atomic oxygen fluence for polymer samples.