CHARACTERIZATION OF A 5-EV NEUTRAL ATOMIC OXYGEN BEAM FACILITY

J. A. Vaughn, R. C. Linton, M. R. Carruth, Jr., and A. F. Whitaker
NASA/Marshall Space Flight Center
Huntsville, AL 35812

and

J. W. Cuthbertson, W. D. Langer, and R. W. Motley
Princeton Plasma Physics Laboratory
Princeton, NJ 08453

ABSTRACT

An experimental effort to characterize an existing 5-eV neutral atomic oxygen beam facility being developed at Princeton Plasma Physics Laboratory (PPPL) will be described. This characterization effort includes atomic oxygen flux and flux distribution measurements using a catalytic probe, energy determination using a commercially designed quadrapole mass spectrometer (QMS), and the exposure of oxygen-sensitive materials in this beam facility. Also, comparisons were drawn between the reaction efficiencies of the materials exposed in this facility, the reaction efficiencies of materials exposed in plasma ashers, and the reaction efficiencies previously estimated from space flight experiments. The results of this study show that the beam facility at PPPL is capable of producing a directional beam of neutral atomic oxygen atoms with the needed flux and energy to simulate low Earth orbit (LEO) conditions for real time accelerated testing. The flux distribution in this facility is uniform to ±6% of the peak flux over a beam diameter of 6 cm.

INTRODUCTION

The recent return of the Long Duration Exposure Facility (LDEF) provided startling evidence of how volatile the LEO environment is on materials. One of the most material destructive constituents in the LEO environment is atomic oxygen which oxidizes many metals and erodes the surface of many polymers. Considering the expense and logistical demands of doing flight experiments, such as LDEF, for investigating a materials susceptibility to atomic oxygen, the need for a ground-based facility capable of simulating the LEO atomic oxygen environment exists. The requirements of a facility capable of simulating LEO atomic oxygen conditions are: (1) the beam facility must be able to produce a high energy, directional beam of atomic oxygen atoms (i.e., the atom would have an energy equal to the mass of the atomic oxygen atom times the orbital velocity of the spacecraft of 5 eV for atomic oxygen atoms); and (2) the beam facility should produce a flux of atomic oxygen compatible with LEO mission-integrated flux in a reasonable test period (i.e., at least a flux in the range of 10^{14} to 10^{16} atoms/cm^2*s).

The 5-eV neutral atomic oxygen beam facility discussed in this paper was developed at PPPL under contract with NASA/Marshall Space Flight Center (MSFC). This neutral beam system is capable of producing atomic oxygen atoms with a flux of 10^{15} to 10^{16} atoms/cm^2*s and an energy of 4 to 20 eV which meets the previously stated requirements. Also, the facility has features which make it possible to study the combined effects of UV and other LEO environmental concerns on materials.

The results of this study include a direct measurement of the atomic oxygen flux in this beam facility using a catalytic probe similar to the one described by Carruth et al. [1] and a measurement of the flux uniformity by evaluation of material erosion at discrete intervals across the beam diameter. Reaction efficiencies from materials exposed in this facility and the reaction efficiencies of materials exposed in ground-based plasma ashers are compared to the published reaction efficiencies of materials exposed during STS-8 and STS-41G missions. This comparison includes graphic evidence of the enhanced simulation of the surface morphology effects of LEO atomic oxygen with a directional beam then with plasma ashers. Scanning electron microscope (SEM) photographs will be shown to demonstrate how this beam facility closely reproduces the surface morphology of materials that have been exposed to atomic oxygen during flight experiments. The SEM photos of the flight and PPPL neutral atom beam facility exposed materials will be compared to materials exposed in plasma ashers at MSFC.

ATOMIC OXYGEN BEAM FACILITY

The PPPL atomic oxygen beam facility produces a low energy neutral atomic oxygen beam by placing a metal plate in contact with a magnetically confined (3 to 4 kG) oxygen plasma. The oxygen plasma is produced by a radio frequency (RF)-driven lower hybrid source [2] which operates on molecular oxygen gas. A magnetron supplies 1 kW of power at an RF frequency of 2.45 GHz to the center pin to break down the gas. The RF frequency of this facility produces a plasma with

complete dissociation of the molecular oxygen gas [3]. Because of the intense heat built up on the center pin, the plasma source is currently pulsed at a 5% to 15% duty cycle over a few milliseconds. Cycling the plasma source in this manner enables the beam source to operate in oxygen for over 100 hours per pin. The plasma is confined by a magnetic field (3 to 4 kG) which produces an intense plasma column about 1 cm in diameter. Because of the tightly confined plasma column, the neutral atom distribution beyond the neutralizer plate is well collimated. The metal neutralizer plate is biased negative of plasma potential, and plasma ions are accelerated toward the surface of the plate by an energy determined by the potential difference between plasma potential and the plate bias. The ions impact and are reflected from the neutralizer plate picking up an electron in the process. The reflection is designed to be an inelastic reflection causing the atoms to lose a fraction of their energy. The fraction of energy retained by each atom depends primarily on the ratio of the particle mass to the neutralizer plate atomic mass. Because the energy of each particle depends on plasma potential and not all ions exit at the same potential, not all ions will be reflected at the same energy, producing a small spread in energy.

The lower hybrid plasma source is capable of supplying 4 amps of atomic oxygen ions to the neutralizer plate per pulse producing an estimated flux of >5x10^{16} atoms/cm^2*s atomic oxygen atoms 10 cm from the neutralizer plate. The average integrated flux over the entire duty cycle is 1x10^{15} to 7x10^{15} atoms/cm^2*s at this same axial position. The limiting factor in the flux level for this beam facility is the duty cycle of the plasma source.

ATOMIC OXYGEN BEAM FACILITY DIAGNOSTICS

Energy Diagnostics

A commercially designed energy-analyzing QMS was used to detect the energy of the neutral atoms in this facility. The QMS is a standard QMS with a cylindrical mirror energy analyzer downstream of the quadrapole rods. With this instrument, a neutral beam entering the ionizing chamber can be scanned measuring both the energy spectrum of a particle at a single mass or the mass spectrum at a single energy. Special pumping stages were designed to incorporate the QMS in the neutral beam facility to minimize scattering of the incoming high energy atomic oxygen atoms and reduce the production of energetic atomic oxygen atoms (Franck-Condon atoms) created by the dissociation of molecular oxygen in the ionizing region of the QMS.

The energy measurements to date have been limited to monatomic gases such as argon and krypton. These measurements were compared to calculations using the TRIM model [3]. The beam energy and energy spread with these measurements agreed well with the TRIM calculations. The atomic oxygen

energies presented in this paper have been estimated from the plasma potential, the neutralizer bias, and using conservation of momentum results from argon and krypton gases. Attempts to measure molecular gases like nitrogen and oxygen have been unsuccessful to date because of complicating effects caused by the dissociation of the thermal background molecules in the QMS ionizing section. A further reduction in background pressure is being considered to eliminate the background molecules.

Atomic Oxygen Flux Diagnostics

A special catalytic probe for monitoring the atomic oxygen flux was designed for use in this neutral beam facility. The catalytic probe was modeled after the one used by Carruth et al. [1] in a conventional plasma asher. The catalytic probe uses silver oxide as the catalyst to produce an increase in temperature caused by the recombination of atomic oxygen atoms on the surface of the catalyst. Since the inelastic collisions of the high energy neutral atoms with the catalytic probe also cause the probe to heat up, a method to account for this heating was devised. The catalytic probe designed for this facility consisted of two thin (approximately 1-mm thick) circular glass substrates with a type k thermocouple attached to the back. One probe was coated with 1,000 angstroms of silver and oxidized in an oxygen plasma atmosphere, while the other probe was left uncoated. The silver oxide probe was used as the active probe to measure the flux of atomic oxygen atoms in the facility, and the plain glass probe was used as a dummy probe to monitor the increase in the catalytic probe temperature due to the atomic oxygen atoms impacting on the catalytic probe surface. A separate thermocouple was placed near the chamber wall to measure the ambient temperature.

A simple first-order model was developed to make flux calculations from the recorded temperature data. The model assumes that both the catalytic probe and the dummy probe radiate their heat to the surrounding environment, and that conduction and convection heat losses were negligible. Because the thermocouple wire is the only heat conductive path possible and the wire is very thin, the assumption is correct to a first order approximation. The model also takes into account the energy of atomic oxygen recombination on the catalyst (5.2 eV per every two atoms [4]) and the fact that only a fraction of incoming atoms recombine on the surface of the probe (i.e. recombination coefficient).

A literature search shows that the value of the recombination coefficient used in previous work ranged from 0.25 to 1 [1]. Carruth et al. [1] assumed that the recombination coefficient for atomic oxygen on silver oxide was near unity for the work they did in an oxygen plasma asher knowing that some inaccuracy may exist. Because the inelastic collisions of the incoming atomic oxygen atoms with the silver oxide surface scatters a fraction of the atoms away, the

recombination coefficient in the work in this paper was assumed to be 0.5. This assumption is inherently uncertain to within a factor of two because of the uncertainty in the scattering efficiency of the atomic oxygen atoms during the collision. However, data will be presented comparing the flux computed from catalytic probe temperature data and flux data from ion current measurements. Also, data showing the error in the recorded flux measurements caused by varying the recombination coefficient from 0.25 to 0.75 will be shown.

Below is the final result of the model. Equation (1) was used to make the atomic oxygen flux calculations for this particular study.

$$\text{flux} = \frac{1.4 \times 10^7}{n} \left[e_p (T_p^4 - T_w^4) - \frac{A_{gp} e_{gp}}{A_p} (T_{gp}^4 - T_w^4) \right] \tag{1}$$

In equation (1) the constant (1.4×10^7) takes into account the 5.2-eV energy per every two atoms of recombination and converting the flux into the correct units of atoms/cm^2*s. The values of the emissivity and the area of both the active probe and the glass probe are listed as e_p, A_p and e_{gp}, A_{gp}, respectively. The silver oxide probe emissivity was 0.85, and the glass probe emissivity ranged from 0.7 to 0.8. The area of both probes was the same. Finally, n represents the recombination coefficient.

MATERIAL EXPOSURE PROCEDURE

Samples exposed to the atomic oxygen neutral beam were placed in an aluminum sample holder and insulated from the metal to keep them from being heated as the aluminum increased in temperature during operation of the plasma source. A type K thermocouple was placed in contact with the rear surface of the specimen for continuous monitoring. The samples were placed in the PPPL neutral beam facility at a location 8 to 10 cm downstream of the neutralizer plate.

The atomic oxygen beam was turned on when a pressure of 10^{-6} torr was reached inside the vacuum vessel. The samples were exposed to an atomic oxygen flux of 1×10^{15} to 7×10^{15} atoms/cm^2*s for 1 to 12 hours, depending on the desired fluence. The temperature was recorded as well as the ion current to the neutralizer plate at intervals during the exposure process. The ion current was measured by biasing the neutralizer sufficiently negative to repel all electrons from reaching the plate. This process lasted only a few seconds to minimize the effect on the samples.

The flux of atomic oxygen atoms arriving at the surface of the sample was calculated using a $1/R^2$ dependence (the $1/R^2$ dependence was measured and the data is shown in Fig. 3 of this paper). Equation (2) was used to calculate the atomic oxygen flux using the measured ion current data.

$$\text{flux} = \frac{J_n \, DC}{q \pi z^2} K \quad . \tag{2}$$

In this equation, J_n is the ion current hitting the neutralizer plate, DC is the duty cycle, q is the electronic charge, z is the axial distance from the neutralizer, and K is a constant. The factor K takes into account scattering of the neutral atoms due to collisions with molecules, the cosine distribution of the atoms leaving the plate, and the efficiency of the neutralizer plate to produce neutral atoms. This factor was calculated to be 0.5 based on available data.

NEUTRAL BEAM DIAGNOSTIC RESULTS

Beam Energy Data

Preliminary energy results using krypton reflecting off a tantalum neutralizer plate are shown in Fig. 1. The voltage bias (V_n) on the tantalum neutralizer plate for this set of data was -10 V. Figure 1 is a plot of QMS intensity as a function of beam energy. This QMS neutral beam profile shows that the krypton neutral beam has a peak energy of 7 eV and beam energy spread defined by the full-width-half-maximum (FWHM) of ±3 eV. The large signal in the few eV range, which is typical of all profiles taken in the PPPL neutral beam facility, is caused by the background pressure. The beam energy profile shown in Fig. 1 is typical of all QMS profiles taken with argon and krypton. Future work in this area will concentrate on producing reliable energy measurements of atomic oxygen by reducing the background pressure.
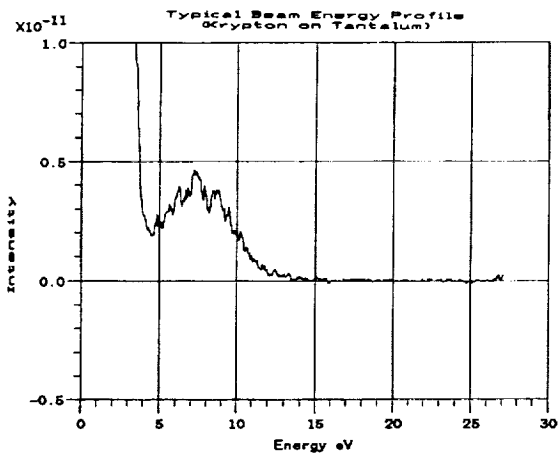

Figure 1 – Typical QMS Neutral Beam Energy Profile (Krypton on Tantalum Neutralizer Plate)

Atomic Oxygen Flux Results

Measurement of the flux and the flux distribution
of the neutral atomic oxygen beam has shown much
consistency. The flux level has been detected
using the catalytic probe described earlier and
verified from mass loss data of high density
polyethylene (HDPE) exposed for a known period of
time. Figure 2 shows the flux calculated using
the temperature measured by the catalytic probes
at various times over an 80-minute test period.
The squared symbols indicate the atomic oxygen
flux computed using equation (1), and the dashed
line shows the atomic oxygen flux computed by
measuring the ion current impinging on the
neutralizer plate. The flux computed from cata-
lytic probe temperature measurements agree well
with the flux computed from the ion current meas-
urements. The rise in the flux level at 42
minutes was caused by an increase in the duty
cycle of the plasma source. The increase in the
duty cycle was done deliberately to demonstrate
the ability of the catalytic probe to adjust its
temperature with a change in the level of atomic
oxygen flux in the vacuum chamber. It should be
noted that because the catalytic probe does not
cool down quickly, all tests were done by start-
ing at the lowest atomic oxygen flux level and
working toward the highest in order to get a
response in a reasonable amount of time. The
error bars associated with computed flux from the
catalytic probe temperature data signify the
amount of uncertainty induced by varying the
recombination coefficient (n in equation (2))
from 25% to 75% and by taking into account the
uncertainty involved in the emissivity of both
the glass and catalytic probes. Samples of HDPE
were exposed to the same conditions as those data
in Fig. 1. HDPE was used to make flux calcula-
tions based on the mass loss data because HDPE is
not hydroscopic. The results of mass loss data
similarly indicate the flux varied from 1.5×10^{15}
and 4×10^{15} atoms/cm^2*s at the 5% duty cycle.
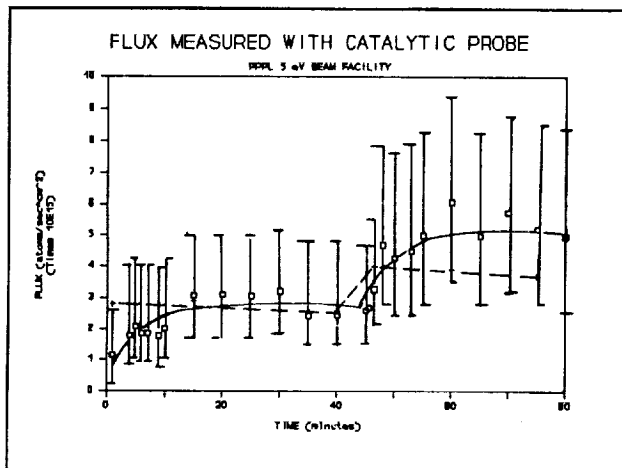

Figure 2 - Flux of Atomic Oxygen Atoms
Measured With a Catalytic Probe

Figure 3 shows the atomic oxygen flux computed
from catalytic probe temperature measurements
made at three axial positions in the vacuum cham-
ber. These measurements were made to demonstrate
the $1/R^2$ dependence of the atomic oxygen flux in
this chamber. The line in Fig. 3 represents the
$1/R^2$ dependence line which was computed knowing
the flux of atomic oxygen atoms just leaving the
surface of the neutralizer plate and correcting
the flux at incremental axial positions. The
data points in Fig. 3 were computed from the mean
catalytic probe temperature measured over a 15-
minute period. The error bars indicate the
spread in the flux computations caused by the
variation in the measured temperature data. This
figure indicates that the atomic oxygen flux
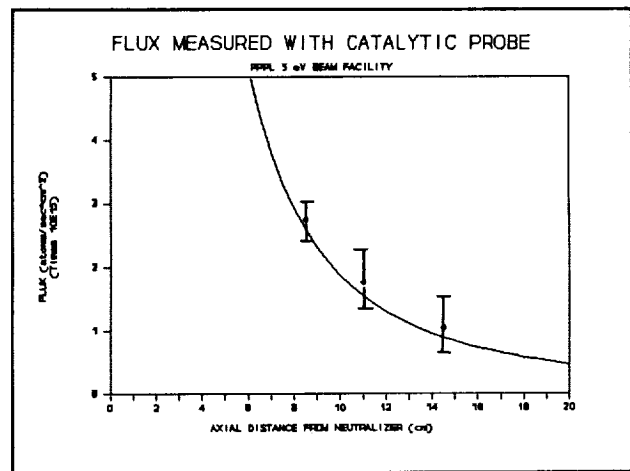closely follows the prescribed $1/R^2$ distribution.

Figure 3 - Dependence of Atomic Oxygen Flux
on Axial Distance

The flux uniformity in the sample exposure region
was also measured from the mass loss data of 1.5
mil HDPE spaced equally across the specimen
exposure plane. The flux calculations were made
using the published reaction efficiency for
polyethylene of 3.7×10^{-24} cm^3/atom [5]. The
results of the flux distribution in the PPPL beam
facility are shown in Fig. 4. The angle of the
neutralizer plate was set to provide the highest
flux down the center (or 0 cm radial distance) of
the specimen exposure plane. The angle of the
neutralizer was set at 35 degrees from the center
of the sample region to the normal of the neu-
tralizer plate. The square data points are the
flux data computed from the measured mass loss of
each polyethylene sample, and the inverse tri-
angular symbols are for the flux data computed
from the ion current data and corrected for the
$1/R^2$ dependence from the center of the neutral-
izer plate. In this case, the flux computed from
the mass loss data is four times higher than that
indicated by the ion current measurements. The
discrepancy in the data is not known at this
time, but there is evidence that indicates the
reaction efficiency may depend on the energy of
incident atoms [6]. The higher energy atoms in
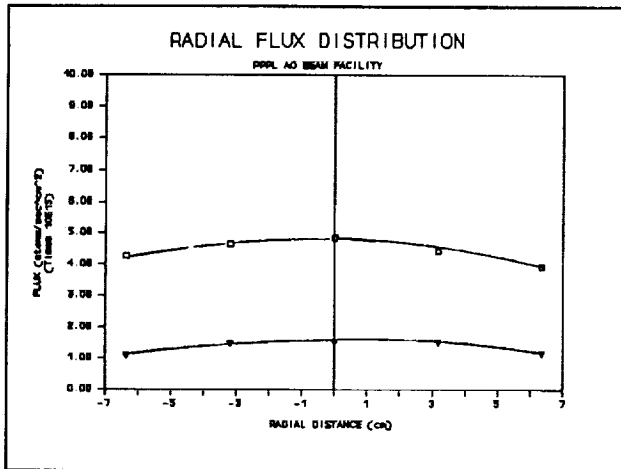the PPPL facility, with a higher reaction

Figure 4 – Atomic Oxygen Flux Distribution
in PPPL Beam Facility

efficiency, would lower the calculated flux.
However, from a relative perspective, it is
important to notice that the peak in both curves
occurs at the center of the vacuum chamber, and
the flux decreases 11% over a 13-cm beam diameter
with the plate positioned at this angle. A ques-
tion arose during these tests as to the extent
the uniformity would be affected by a rotation in
the plate. This question was addressed in a
second 1.5-mil HDPE sample exposure. The same
procedure was followed, except the plate was
rotated 20 degrees from the first position (i.e.,
the normal of the plate was 55 degrees from the
horizontal axis down the center of the sample
chamber). Figure 5 shows the atomic oxygen flux
distribution in the sample chamber measured from
this test. It should be noted that the energy
was intentionally reduced closer to 5 eV during
this test to get the data in better agreement.
The peak atomic oxygen flux in this case has
shifted from the center of the vacuum vessel
toward the right side of the vacuum chamber, but
the uniformity has not changed. The atomic
oxygen flux still decreases 6% 3.2 cm from the
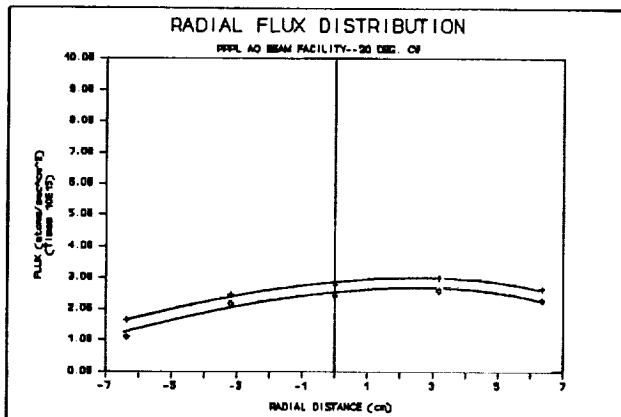location of the peak atomic oxygen flux.

Figure 5 – Atomic Oxygen Flux Distribution
in PPPL Beam Facility

Atomic Oxygen Beam Directionality

In order to examine the directionality versus the
randomness of the atomic oxygen beam at the test
sample location, experiments were conducted to
address this question. Polycarbonate (LEXAN)
samples were spaced evenly across the specimen
exposure plane and exposed to a known fluence of
atomic oxygen. Polycarbonate was chosen for this
test because its crystalline structure preferen-
tially etches under the attack of atomic oxygen
[9]. The samples were covered with a transparent
(90%) nickel mesh screen to protect a portion of
the sample from atomic oxygen atoms. If the beam
is directional, then the samples should be etched
in the unprotected area producing a checkerboard
pattern that has near vertical, uniform edges.
If the beam is random, the checkerboard pattern
will still be observed, but the face of the
raised edges will not be vertical and uniform.

Figure 6 is an SEM photograph of the polycar-
bonate surface. The surface features shown in
this photograph are typical of all the samples
exposed in the beam. In Fig. 6, the light-
colored area is the area attacked by the atomic
oxygen, and the dark-colored area was protected
by the screen. The SEM photograph shows that a
distinct checkerboard pattern was etched in the
polycarbonate sample by the atomic oxygen beam.
One interesting characteristic of this photo
which confirms the beam directionality is the
sharp, square corners observed. A measure of the
atomic oxygen erosion depth, and how straight and
uniform the atomic oxygen eroded the polycar-
bonate, is shown in Fig. 7. The data shown in
Fig. 7 were taken using a Dektak II surface
profilometer, by scanning across one individual
square. Fig. 7a is a scan across one of the
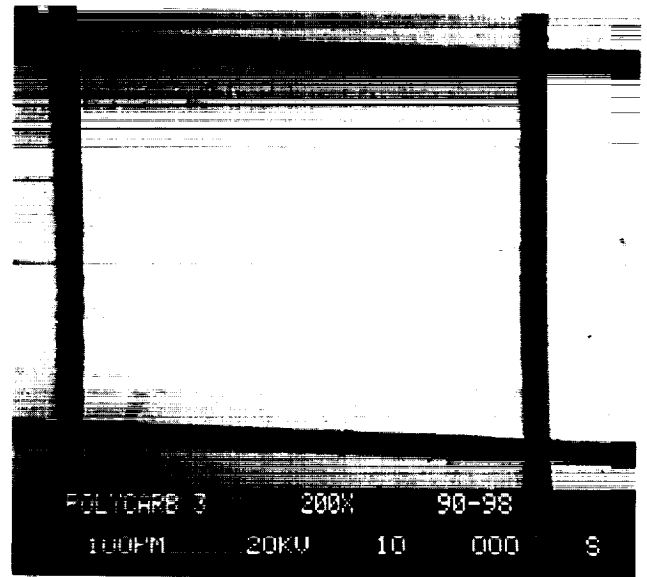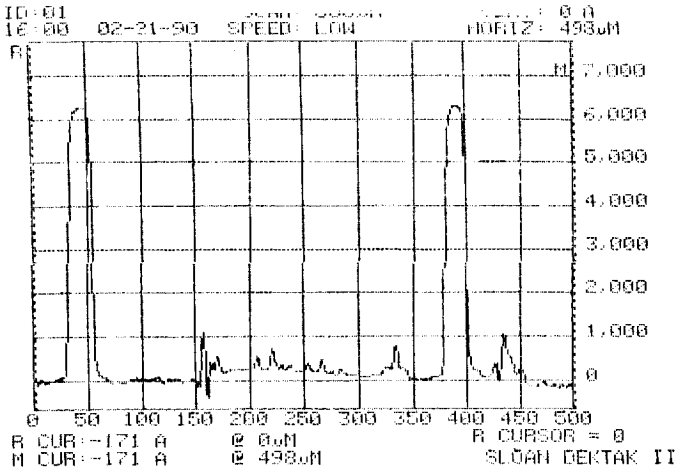squares etched in the polycarbonate samples, and

Figure 6 – SEM of Polycarbonate Covered With
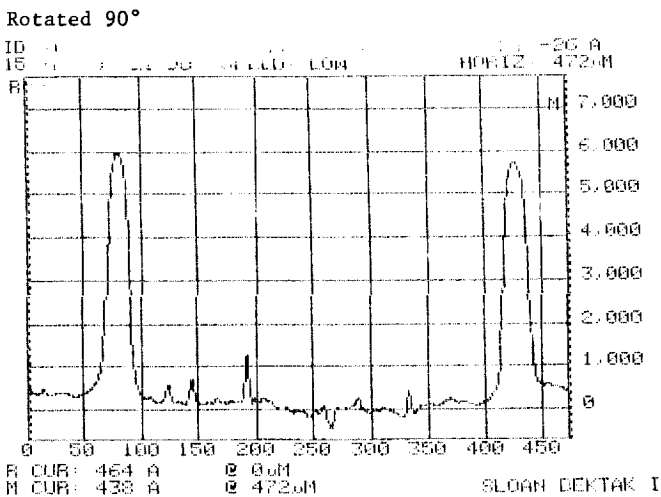Nickel Screen Exposed in PPPL
Neutral Beam Facility

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

ID 01
16 00 02-21-90 SPEED LOW   HORIZ 493uM

7,000
6,000
5,000
4,000
3,000
2,000
1,000
0

0 50 100 150 200 250 300 350 400 450 500

R CUR -171 A   @ 0uM   R CURSOR = 0
M CUR -171 A   @ 498uM   SLOAN DEKTAK II

7(a)

Rotated 90°

ID
15   HORIZ 472uM

7,000
6,000
5,000
4,000
3,000
2,000
1,000
0

0 50 100 150 200 250 300 350 400 450

R CUR: 464 A   @ 0uM
M CUR: 433 A   @ 472uM   SLOAN DEKTAK I

7(b)

Figure 7 - Polycarbonate Depth Profile

Fig. 7b is the same etched square rotated 90 degrees. These data indicate that the atomic oxygen etched the polycarbonate to a depth of 6,000 angstroms, and these traces show the precise, sharp edge between the unexposed and exposed polycarbonate. Comparing the distance between the two raised edges of each profile shows the etched area to be close to a perfect square. These data provide evidence that the neutral beam in this facility is likely directional.

COMPARISON OF VARIOUS ATOMIC OXYGEN EXPOSED MATERIALS

The effectiveness of this neutral beam facility in simulating the LEO atomic oxygen environment can be judged by comparing the results of material tests done in this facility to space flight material tests. The data compiled to make this comparison are shown in Table I. The data presented in this table were taken from various flight experiments [5,7,8] flown on STS-8 and STS-41G, the results of material tests done in

the PPPL neutral beam facility, and material tests done in plasma ashers at MSFC. The data from which these results were compiled are for the same sample temperature. The results of this comparison show the reaction efficiency of the PPPL neutral beam facility to accurately reproduce the data from flight results. It should be noted that a large error was induced in the reaction efficiency of the kapton sample because of the hydroscopic nature of the sample and poor control over mass measurements to determine mass loss.

Table I - Reaction Efficiency of Atomic
Oxygen (AO) Exposed Materials
(Reaction Efficiencies in units of cm^3/atoms)

	Kapton-H 2 mil	Black Kapton	Lexan 5 mil	HDPE 1.5 mil
Space (STS-8 & STS-41G)	$3x10^{-24}$ *	$8x10^{-24}$ +	$6x10^{-24}$ **	$4x10^{-24}$ *
PPPL 5eV AO Beam	$11x10^{-24}$	$8x10^{-24}$	$5x10^{-24}$	$4x10^{-24}$
MSFC Plasma Asher	$4x10^{-26}$	$6x10^{-26}$	$5x10^{-26}$	$2x10^{-25}$

*[5] Visentine
+[8] Whitaker et al.
**[7] Gregory et al.

The reaction efficiency calculated from mass loss data of plasma asher exposed materials at MSFC differs from both space flight determined reaction efficiencies and PPPL determined reaction efficiencies. Two possible explanations have been identified as to the large discrepancy. The first suggests that the reaction efficiency of polymer materials is dependent upon the energy of the incoming atom. Secondly, the flux used to compute the above reaction efficiencies was $1x10^{18}$ atoms/cm^2*s which was measured by Carruth [2] using a catalytic probe. However, attempts to reproduce the flux measurements using HDPE indicate a flux two orders of magnitude different.

A final comparison of the surface morphology of materials exposed to the different atomic oxygen simulation techniques can be made from SEM photographs. Figure 8 is an SEM photograph of the surface of silver covered with 5 mils of FEP Teflon (Ag/FEP) exposed to a fluence of $1x10^{20}$ atoms/cm^2 in the PPPL neutral beam facility, and Fig. 9 is an SEM of similar material taken from the covering on The Transverse, Flat-Plate Heat Pipe Experiment (S1005) aboard LDEF. Comparing both Figs. 8 and 9 it is difficult to tell them apart at first glance. The slight difference observed in aspect ratio (ratio of height of peak to diameter of the base of peak) may be due to the difference in energy or dose of atomic oxygen (LDEF fluence was $9x10^{21}$ atoms/cm^2). In contrast,
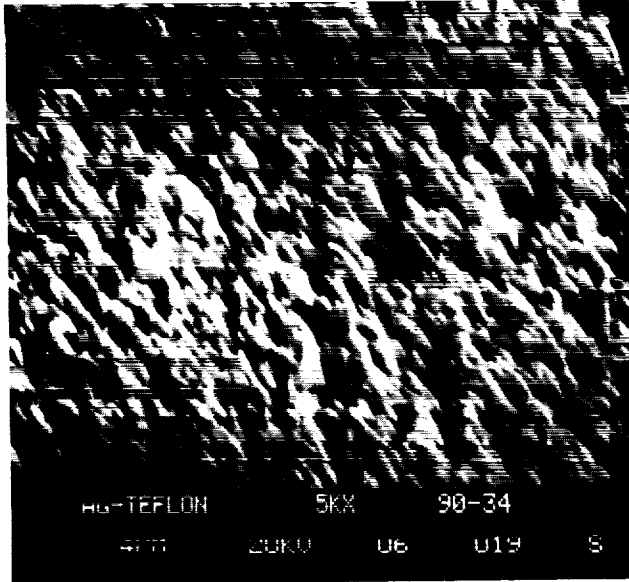
Figure 8 - SEM of Ag/FEP Exposed in PPPL
Neutral Beam Facility (5k X)
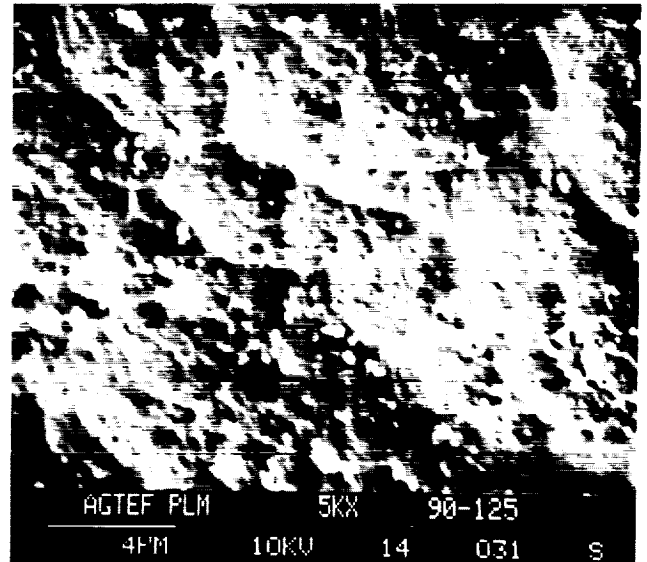
Figure 10 - SEM of Ag/FEP Exposed in
Plasma Asher at MSFC

The reaction efficiency of materials exposed in this facility compares quite well to those exposed during flight experiments aboard STS-8 and STS-41G.

The high energy atomic oxygen beam source developed at PPPL is capable of producing atomic oxygen flux levels of 1×10^{15} atoms/cm^2*s and greater. These flux levels have been verified using both the catalytic probe and material mass loss data. The flux uniformity at the specimen exposure plane decreases 11% over a 13-cm diameter beam. While the flux distribution is not effected by rotating the neutralizer plate, the peak flux can be displaced from the centerline axis of the specimen exposure plane by this rotation.

The beam energy measurements, made with the QMS using argon and krypton reflecting off either tantalum or molybdenum neutralizer plates, show the facility does produce neutral beams with an energy range of 4 to 20 eV. Attempts to measure the energy of an atomic oxygen neutral beam have not been successful to date because of the creation of energetic particles produced during the dissociation of molecular oxygen in the ionizing region of the QMS. Further reductions in background pressure are needed to measure atomic oxygen atoms.
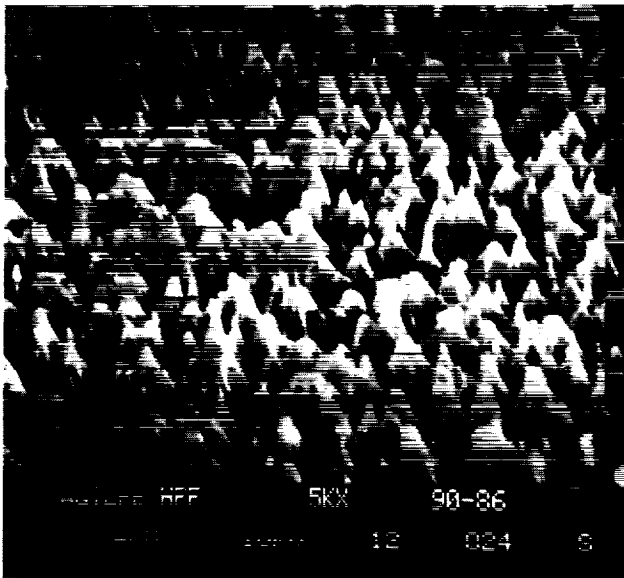

Figure 9 - SEM of Ag/FEP Exposed in Space
Aboard LDEF (5k X)

Fig. 10 shows an SEM of the surface of Ag/FEP exposed in the MSFC plasma asher to a fluence of 1×10^{20} atoms/cm^2. The surface of the sample exposed in the plasma asher does not show the spiked nature seen in the other two SEM photographs. Overall, from the evidence provided, the PPPL neutral beam facility provides a better simulation to the LEO environment than the plasma asher.

CONCLUSIONS

The high energy atomic oxygen beam facility developed at PPPL is capable of simulating an LEO environment better than an ordinary plasma asher.

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