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Translational Energy Dependence of the O + Polyimide Reaction

G. S. ARNOLD, D. R. PEPLINSKI, and F. M. CASCARANO
Chemistry and Physics Laboratory
Laboratory Operations
The Aerospace Corporation
El Segundo, CA 90245

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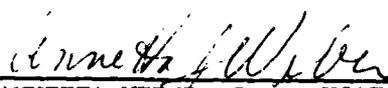**DISTRIBUTION UNLIMITED**

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ANNETTA WEBER, Capt, USAF
MOIE Project Officer
SD/YNSA

JOSEPH HESS, GM-15
Director, AFSTC West Coast Office
AFSTC/WCO OL-AB

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Atomic beam experiments are reported in which atomic oxygen, produced in a conventional microwave discharge beam source, struck Kapton samples at a translational energy of approximately 0.14 eV. After being exposed to an atomic oxygen fluence in excess of 2×10^{19} cm^{-2}, the Kapton samples showed no evidence of reaction, either to the naked eye or under microscopic examination. These experiments, and others performed in this laboratory and elsewhere, strongly support the notion that there is a true dynamic barrier

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20. ABSTRACT *(Continued)*

(in incident translation) of between 0.2 and 1 eV for the etching of polyimide films by atomic oxygen. Keywords → to ρ A)

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I. INTRODUCTION

The work reported herein was undertaken in an attempt to answer more clearly the question of whether the rate of reaction of atomic oxygen with polyimide films depends, for energies up to 5 eV, on the translational energy of the atom. This section presents the background and motivation for this work. The second section of this report describes the experimental apparatus and procedure. The final section presents the results, and a discussion of their implications.

It has become well known that ambient atomic oxygen reacts with many organic materials at rates sufficient to cause significant erosion of the surface of spacecraft in low Earth orbit (LEO) during the course of typical satellite missions.[1-6] The effect of ambient oxygen on the du Pont polyimide Kapton* has been particularly well documented. Although the reaction of atomic oxygen with polymers has been reported in the literature,[7] the rates at which polymers and paints are removed in LEO were surprisingly high.[1,8] The origin of this discrepancy between erosion rates observed in LEO and those which one might infer from the mass-loss data of Hansen et al.,[7] who exposed a variety of polymers to essentially room-temperature atomic oxygen, has not been conclusively identified.

It has been suggested that the rate at which an organic material is eroded in LEO is accelerated by the fact that atomic oxygen strikes a spacecraft surface (normal to the velocity vector) at a translational energy of approximately 5 eV.[1,8,9] It is certainly the case that gas-phase abstraction and insertion reactions of $O(^3P)$ with hydrocarbons exhibit dynamic barriers ranging from 0.05 to 0.20 eV.[10-14]

The theory that the O atom reaction with polymers is translationally activated is supported by some recent laboratory investigations. Ferguson has reported that O^+ impacting at kilovolt energies reacts even faster with Kapton

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*Kapton is a registered trade name of E. I. du Pont de Nemours & Co., Inc.

than does ambient oxygen in low Earth orbit.[9] However, the comparability of the reactions of oxygen atoms and ions may be open to some doubt. Tennyson et al. reported that oxygen atoms with a translational energy of approximately 0.14 eV, produced from a microwave discharge atomic beam source, apparently react with Kapton much more slowly than do the 5-eV atoms encountered on orbit;[15] however, the absolute reaction probabilities they report are somewhat uncertain, inasmuch as their experimental geometry renders measurement of the oxygen atom beam flux difficult. The flux which Tennyson et al. calculate for their source is, as they point out, an upper limit, and so the reaction probabilities they report are lower limits.[15]

The experimental and analytical support for the notion that O atom reactions with organic polymers are translationally activated is by no means unanimous. Work reported earlier from this laboratory showed that atomic oxygen striking Kapton surfaces at a translational energy of 1 eV reacted at a rate indistinguishable from orbital reaction rates.[9] Furthermore, the analysis of the thermal mass-loss data of Hansen et al.[7] is not straightforward. Depending upon the assumptions one makes about atomic oxygen production and transport in Hansen's experiments, one calculates in those experiments absolute O atom reaction efficiencies that vary by more than two orders of magnitude.[9,16]

The question of the translational energy dependence of the reaction of atomic oxygen with polymers is not a matter of merely academic interest. If the reaction rate were insensitive to translational energy (below 5 eV), there would be two major implications:

1. Spacecraft materials could be screened for stability (or lack thereof) to exposure to the residual atmosphere in low Earth orbit by the use of equipment that is considerably less expensive and less complex than atomic beam facilities currently used or proposed.

2. One would be obliged to consider that spacecraft surfaces covered with organic polymers not directly exposed to atmospheric impingement may be eroded by atomic oxygen that scatters (presumably at reduced velocity) from exposed surfaces.

II. EXPERIMENTAL PROCEDURE

A. ATOMIC BEAM APPARATUS

The beam apparatus used to perform these experiments has been described elsewhere.[17] It consists of a four-chamber, differentially pumped vacuum system (see Fig. 1). The oxygen beam source is mounted in the first chamber, which provides differential pumping for the source. The second and third chambers provide for additional pressure reduction between the source and the target. Chamber 2 also contains a beam flag and chopper to aid in analysis of the beam's composition and velocity.

The fourth chamber contains the solid target, mounted in a temperature-controlled holder on a three-axis-plus-rotation precision manipulator. The fourth chamber also houses a quadrupole mass spectrometer used to measure beam composition and velocity.

A conical nickel skimmer 0.9 mm in diameter forms the connection between the first and second chambers. The 2/3 and 3/4 connections are round holes approximately 2.5 mm in diameter.

During sample exposures, the pressures in chambers 1 through 4 were approximately 5×10^{-5}, 1×10^{-6}, 5×10^{-7}, and 2×10^{-7} Torr, respectively.

B. ATOMIC OXYGEN SOURCE

Oxygen atoms were produced by inducing a microwave discharge in a gas mixture containing O_2. The beam source used in this experiment (see Fig. 2) employs a cavity very similar to that described in detail by Murphy and Brophy.[18] The cavity is a 3-1/4 wave foreshortened coaxial type, with microwave power coupling and cavity tuning of the Evenson design.[19] Power was supplied to the cavity by a Raytheon PGM-10 2450-MHz magnetron power supply. The discharge tube was fused silica, 6 mm in diameter, with an orifice diameter of 1.0 mm.

The beam composition and velocity were measured with the quadrupole mass spectrometer. The formula of Miller and Patch[20]
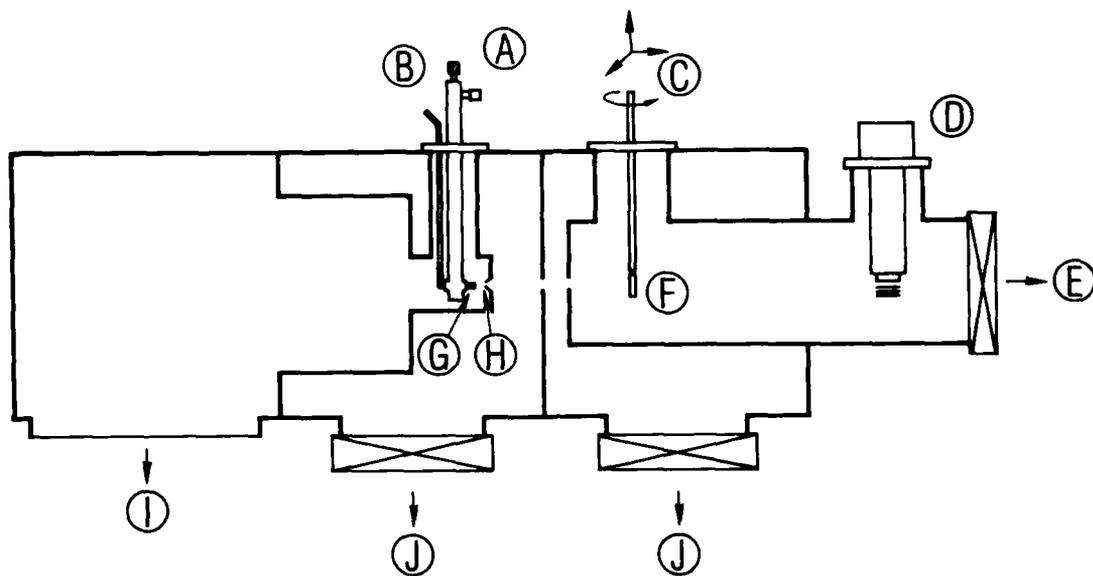
Fig. 1. Atomic Beam Facility Schematic. (a) Microwave cavity. (b) Source gas inlet. (c) Three-axis-plus-rotation precision manipulator. (d) Quadrupole mass spectrometer. (e) 350 ℓ sec^{-1} turbomolecular pump. (f) Temperature-controlled sample mount. (g) Source nozzle. (h) Skimmer. (i) 16" oil-diffusion pump. (j) 10" oil-diffusion pump with liquid-nitrogen baffle.
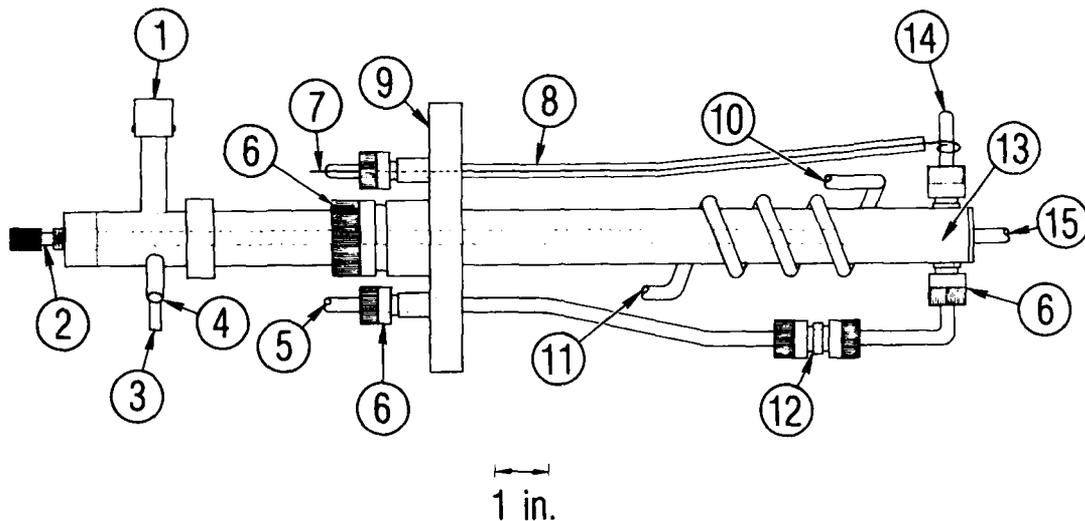
Fig. 2. Microwave Discharge Atomic Beam Source. (1) Microwave power
connection. (2) Cavity tuning adjustment. (3) Coupling
adjustment. (4) Cooling gas outlet. (5) Source gas inlet.
(6) Ultra-Torr compression fitting. (7) Ignition electrode.
(8) Glass insulator. (9) Mounting flange (O-ring sealed).
(10) Cooling water inlet. (11) Cooling water outlet. (12)
Ultra-Torr union. (13) Quartz discharge tube. (14) Nozzle.
(15) Cooling gas inlet.

$$y = \frac{[O]}{[O_2]} = P \left[\frac{\sigma(O_2)}{\sigma(O)}\right]\left[\frac{1}{n} \frac{I(O)}{I(O_2)} - 1\right] \tag{1}$$

was used to calculate the ratio of O to O_2 in the mass spectrometer ionizer; P is the probability of dissociative ionization of O_2; $\sigma(O_2)$ and $\sigma(O)$ are the cross sections for ionization of O_2 and O, respectively; $I(O)$ and $I(O_2)$ are the mass spectrometer signals for masses 16 and 32; and n is the ratio of those two signals with the source discharge off. The velocities of various species in the beam were measured by mechanically chopping short pulses (approximately 20 µsec wide) and measuring the time for the beam pulse to travel from the chopper to the mass spectrometer.

In earlier tests using this source, it was found that a mixture of approximately 10% O_2 and 1% H_2O, diluted in helium, provided the best compromise between the competing demands for high flux and low pressure in the apparatus.[21] The addition of a small amount of water to the discharge mixture produces a nearly fivefold enhancement in the fraction of molecular oxygen that is dissociated. (This effect has been documented by other investigators.[22]) Figure 3 shows mass spectra of the beam, with and without the discharge on.

The measurement of an absolute reaction rate or efficiency in an experiment of this type requires a knowledge of the absolute flux of atomic oxygen in the beam. Measuring or calculating that flux is, at best, problematical. Two experimental approaches, based on calibrating the mass spectrometer absolutely, and one approximate calculation were used to ascertain the beam flux. These approaches are described below.

The experimental approaches to measuring the beam flux were those used in earlier experiments employing this facility.[23] Each was intended to calibrate the sensitivity of the mass spectrometer to molecular oxygen. The first, static gas addition, was to add O_2 directly into the detector chamber, measuring the added oxygen pressure with an ultrahigh-vacuum ionization gauge. The second was to measure the beam signal with low pressures of pure
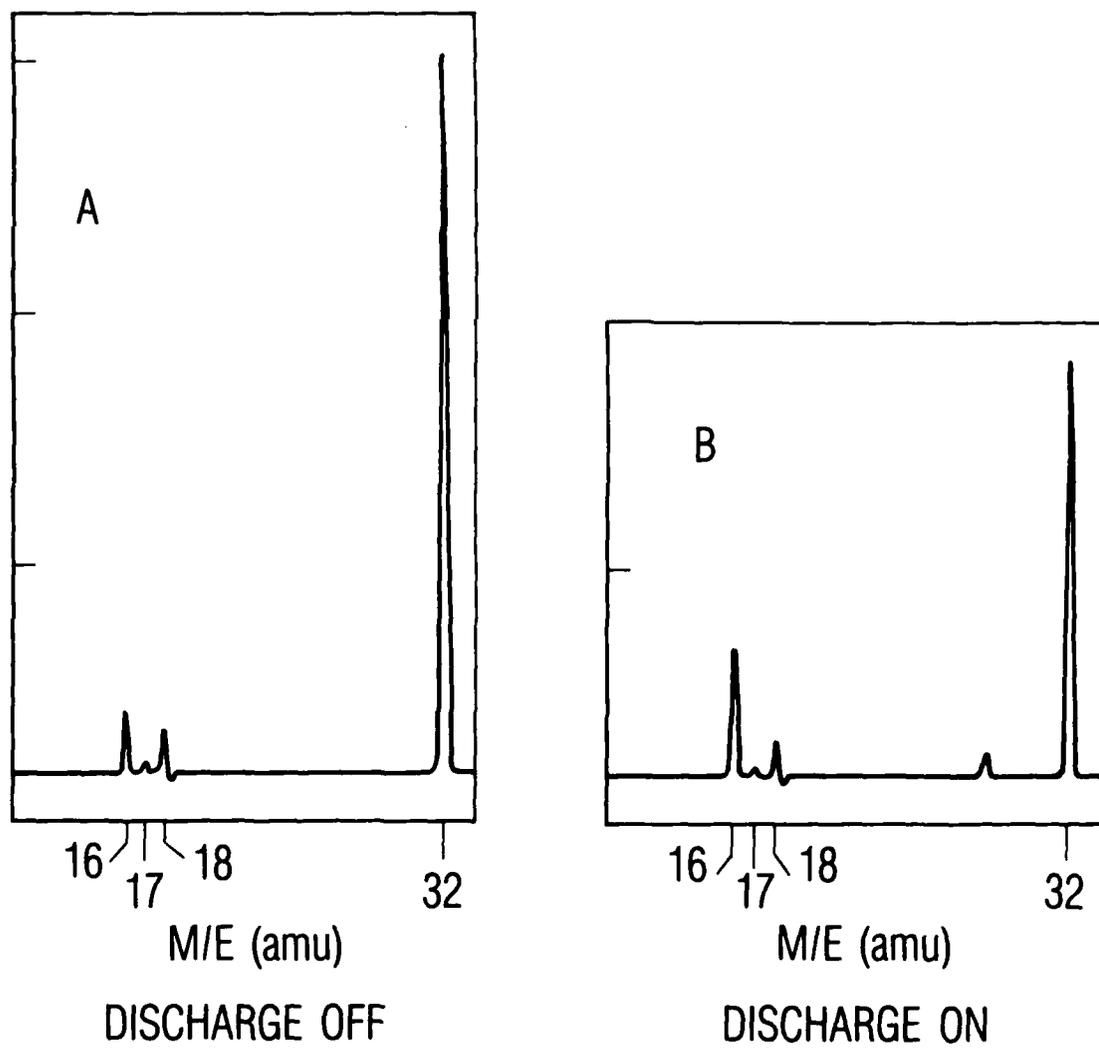
Fig. 3. Mass Spectrum of Atomic Beam. (a) Discharge off, (b) discharge on. (M/e values of 16, 17, 18, and 32 correspond to O^+, OH^+, H_2O^+, and O_2^+, respectively.)

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O_2 in the beam source. Treating the source, at low pressures, as essentially effusive allows one to compute an effective beam density at the ionizer.

These two approaches agreed to within a factor of five for the current facility's geometry, with the static gas addition implying a greater sensitivity. The direction of this discrepancy is intuitively satisfactory, since one should expect the ionization volume of the mass spectrometer to be greater for a static gas, entering the ionizer from all directions, than for a directed beam.

Once the calibration of the mass spectrometer to O_2 was known, the atomic oxygen density in the ionizer could be computed absolutely from the relative number-density measurement described above. The beam flux at the target was then computed by multiplying the apparent number density at the mass spectrometer by the nominal beam velocity and by the appropriate factor to account for the locations of the source, target, and mass spectrometer. The typical lower limit to the O atom flux measured this way was 2×10^{15} cm^{-2} sec^{-1} at the target.

The approximate computational scheme of Lam[24] was used to estimate the atomic oxygen flux as well. In this scheme the properties of the mixed-gas jet are modeled by assuming the gas is a single component with thermal and collisional properties equal to the weighted average of those of the constituent gases. This computation, assuming perfect alignment of the system and no interaction between the beam and the skimmer, indicates that the atomic oxygen flux should be approximately 1.6×10^{16} cm^{-2} sec^{-1}. Because this calculation ignored the shielding effect of the skimmer, it overestimates the flux by a factor of approximately two.[25]

It is assumed that these computed and measured values of flux bracket the true flux value. It is further assumed that the preponderance of the O atoms are in the ground (^{3}P) state.

Table 1 summarizes the operating conditions of the source for these experiments.

Table 1. Microwave Discharge Beam Source Characteristics

Source Feed Gas:	
Composition	$He/O_2/H_2O:90/10/1$
Pressure	10 Torr
Microwave Excitation:	
Frequency	2450 MHz
Power	70 Watts
Performance:	
$[O]/[O_2]$	1.8–2.2
Dissociation	50%
Velocities	
O	1.30 km sec^{-1}
O_2	1.12
He	1.45
O Flux on Target	$>2 \times 10^{15}$ cm^{-2} sec^{-1}

C. SAMPLE PREPARATION

The samples that were bombarded were disks of 5-mil-thick Kapton, 0.9 in. in diameter. Each film disk was cleaned with sequential rinses of trichloroethene, acetone, methanol, and deionized water. The solvents were Baker Analyzed Regent Grade. Following the rinses, the samples were blown dry with nitrogen. The polymer disks were bonded to 1-in.-diameter fused-silica flats, with Dow Corning 93-500 space-grade encapsulant as the adhesive. The siloxane material was mixed according to manufacturer's instructions and vacuum degassed before use. The adhesive was allowed to cure for at least seven days at room temperature before the samples were bombarded.

III. RESULTS AND DISCUSSION

Three samples were bombarded, two for a total of approximately 6 hr, and one for 3 hr. Thus, the samples received an atomic oxygen fluence[*] in excess of 2×10^{19} cm^{-2} or 4×10^{19} cm^{-2}. After the bombardments, the samples were removed from the vacuum system and examined, by the naked eye and by optical and electron microscopy.

In no case was there any sign of reaction. If the reaction had been proceeding at the rate observed for 1-eV oxygen atoms,[8] then at least 0.3 μm of Kapton would have been removed and the characteristic surface roughening caused by the etching reaction would have been readily visible.

Interpretation of a negative result such as this one is always difficult. One must consider whether, through some unrecognized error in experimental procedure, the reaction was inhibited. One possibility is that there was some source of contamination in the sample exposure chamber which deposited on the Kapton surface and effectively prevented reaction. It has been shown through flight and laboratory experiments that thin siloxane or metal films can inhibit oxidation of polymers.[2,6]

To test for this problem, a thin silver film was exposed to the oxygen beam for 2 hr, following the same procedure used in the Kapton bombardments. Both low-velocity atomic beam experiments[26] and shuttle-borne experiments[4] have shown silver to be rapidly oxidized by $O(^3P)$. A characteristic black spot in the area where the beam struck the silver film was observed, confirming that oxygen atoms were striking the surface and reacting.

The fact that no significant reaction of atomic oxygen with Kapton was observed in the experiments reported here, together with the results of Tennyson et al., drive one to the conclusion that the hypothesized strong positive dependence on incident translational energy of the reaction of $O(^3P)$

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[*]"Fluence" is the time integral of flux, and therefore has units of particles per unit area. This quantity is sometimes called the integrated flux, or dose.

with polyimide films is real. In this characteristic, the gas-solid reactions of $O(^3P)$ with organics appear similar to the analogous gas-phase reactions.

It is useful to inquire whether the data now available allow one to infer a value for a dynamic threshold (in incident translational energy) for the reaction of atomic oxygen with Kapton. An examination of a simple model for the dynamics of O atom etching reactions can provide a basis for such an inquiry.

Figure 4 portrays a dynamic behavior for the probability, P, that an O atom striking a polymer surface will react; P can be summarized as

$$P = P_0 \qquad E_t \geqslant E_0$$

$$P = 0 \qquad E_t < E_0 \qquad\qquad (2)$$

where E_t is the incident translational energy of the O atom, and E_0 is the threshold energy.

If one were to perform a series of reaction probability measurements with a perfectly monoenergetic beam, with variable energy, then those measurements would map out precisely this behavior (assuming the model to be the correct one). However, if one were to expose a reactive polymer to a gas containing atomic oxygen in thermal equilibrium, then one would measure an effective reaction probability of

$$P_{eff} = P_0 \times F(E_0:T) \qquad\qquad (3)$$

where T is the temperature of the gas, and $F(E_0:T)$ is the fraction of O atoms that strike the polymer surface with translational energy greater than E_0.

Under the assumption that the gas always remains in thermal equilibrium, $F(E_0:T)$ can be calculated from simple kinetic theory to be given by

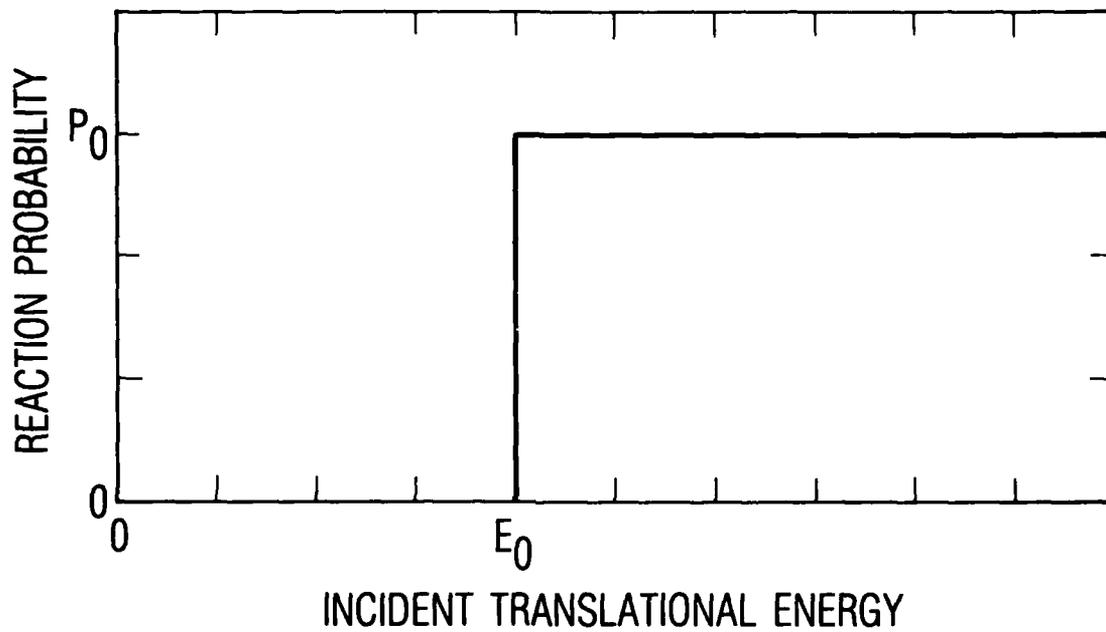$$F(E_0:T) = \Gamma(2, E_0/kT) \qquad\qquad (4)$$

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Fig. 4. A Simple Model for Threshold Behavior in O Atom Reactions with Solids

where k is Boltzmann's constant and $\Gamma(x,y)$ is the incomplete gamma function of the second kind.[27] Figure 5 shows a plot of $\Gamma(2,y)$ for $0 < y < 10$.

The experiments of Hansen et al.,[7] mentioned above, can be analyzed under the assumption of thermal equilibrium.[16] Such an analysis yields the result that the rate of reaction for 340-K O atoms is approximately 1.2% of the rate for orbital velocity (5 eV) O atoms. If one takes the orbital rate to be P_0, then Eq. (4) implies a threshold energy of approximately 0.2 eV. This value is consistent with values observed for gas-phase reactions of $O(^3P)$ with organic molecules.[10-14]

The experimental work reported in this paper used an atomic oxygen beam having a most probable energy of 0.14 eV. However, the distribution of velocities from this source is very narrow. A calculation of the cumulative distribution of O atom velocities one would expect from this source suggests that only approximately 5% of the O atoms emanating from the source have translational energies in excess of 0.2 eV. Therefore, the fact that no reaction was observed in this work is not inconsistent with a dynamic threshold of 0.2 eV. Any more precise definition of that threshold, if indeed the simple behavior postulated is correct, would require additional experimentation.

It would not be unreasonable to suppose that oxidation of other organic polymers, including paint binders, would exhibit a similar dependence on O atom translational energy. This suggests that oxidation of organic materials on spacecraft surfaces by O atoms _reflected_ from another surface, at lower than orbital velocities, will not be as great a problem as oxidation by direct atmospheric impingement.
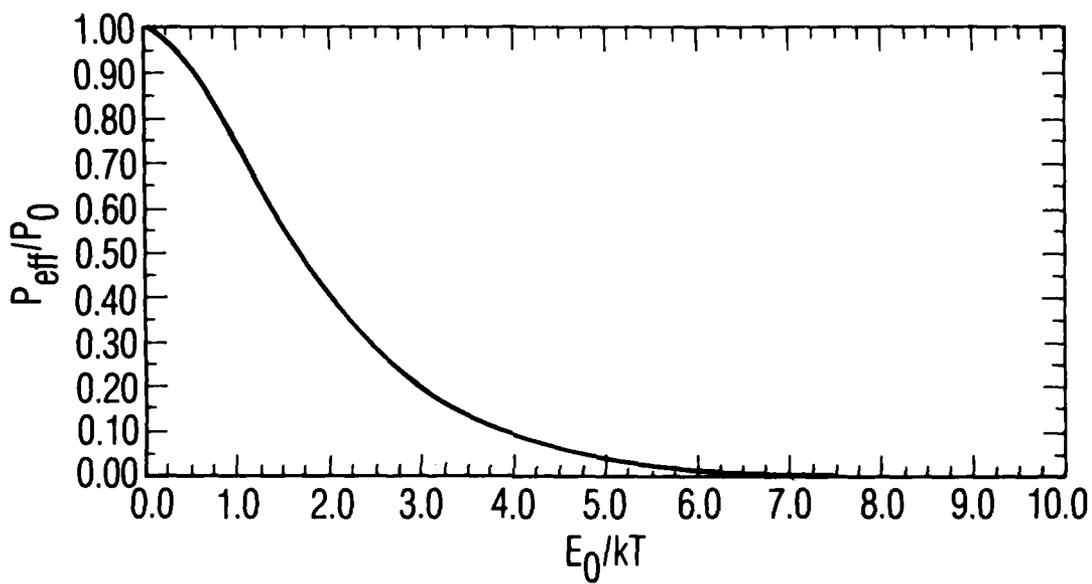
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Fig. 5. Plot of P_{eff}/P_0, the Function $\Gamma(2, E_0/kT)$, vs. E_0/kT

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