Total Sputter Yield Characterization of Various Spacecraft Materials

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Abstract: Here we present experimental measurements of Xenon-impact total sputter yield (TSY) for materials relevant to flexible solar arrays: polyimide foam and a layup consisting of fiberglass mesh and Kapton FPC. Controlled sputter erosion was achieved by exposing samples to a well-characterized mono-energetic (150-350 eV) Xenon ion source for pre-determined durations. Samples were weighed before and after exposure on an analytical microbalance (with 1 µg accuracy) and the resultant mass change, along with average current and duration of exposure were used to determine the TSY. Great care was taken to correct for and quantify systematic effects, such as water absorption in samples. TSY for a baseline material, Molybdenum, is also presented and was found to be in family with historical values in the open literature. Erosion patterns for the polyimide foam showed peculiarly unexpected results, warranting further characterization using various spectroscopy methods. It was determined that elemental carbon sputtered from deeper in the foam could re-absorb near the surface of the foam.

Nomenclature

\[ c_{BG} \] = concentration of adsorbed contaminants on the sample
\[ k \] = Boltzmann’s constant in J/K
\[ M \] = atomic mass of contaminant in atomic mass unit or amu
\[ N_A \] = Avogadro’s constant
\[ N_{exp} \] = number of distinct ion doses for a sample
\[ P \] = background pressure in Pa
\[ Q \] = ion fluence in C
\[ Q_{BG} \] = ion fluence required to remove background contamination layer on sample
\[ Q_i \] = ion beam fluence
\[ s_{BG} \] = sticking fraction of background contaminants to sample
\[ T \] = temperature in K

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I. Introduction

SPACECRAFT electric propulsion devices such as Hall-effect thrusters produce thrust by accelerating and expelling high energy ions, typically Xenon ions. While the plumes of these devices are designed to have minimal divergence in order to produce the most efficient thrust, there often is a non-trivial distribution of ions with angles sufficient to impact on spacecraft surfaces. These impacts can result in sputter erosion of said spacecraft surfaces and contamination of adjacent surfaces. Accurate inputs from laboratory measurements are desirable to adequately model the lifetime effect of sputter erosion.

A system particularly impacted by sputter effects in a spacecraft with high power electric propulsion is the large solar arrays needed to support it (e.g. [1,2,13]). Erosion and contamination of surfaces on the solar array can potentially reduce their operating efficiency over the life of the spacecraft, and in some cases, can compromise their mechanical and thermal properties. A complete analysis of this effect requires accurate total sputter yield and differential sputter yield estimates for each of the solar array materials with a line of sight to the thruster.

In this study, we examine sputtering for materials are planned for use on a new light-weight flexible solar array. Specifically, we determine total sputter yield (TSY) as a function of incident energy, angle, and fluence for xenon impacting on two targets: a layup of urethane-coated fiberglass mesh on polyimide film, and polyimide foam. In addition, TSY for a baseline material, molybdenum, was measured under similar conditions. This allowed for direct comparisons with historical data to validate our approach. As discussed below, additional analysis was performed on the foam samples to understand how the surface was modified following sputter erosion.

II. Experiment

A. Sample Preparation

There were four different kinds of materials tested: fiberglass mesh, Kapton FPC, polyimide foam (Solimide TA-301), and molybdenum (as a baseline for comparison to historical results). The fiberglass mesh samples were coated with urethane, with a nominal open area of 80%. During exposure, the fiberglass mesh was placed on top of the Kapton, producing a layup with a total thickness of 0.38mm. The molybdenum was pre-cut 5 mil sheet manufactured by ESPI Metals with 3N8 purity (99.98%). Apart from the polyimide foam, each of the samples was cut into 3.8 x 3.8 cm squares. Appropriate holes were made in samples to allow mounting to sample holders. The polyimide foam had dimensions of 3.81 x 2.54 x 0.94 cm, with an approximate mass of 65 mg. To minimize any residual contaminants from production and handling, the polyimide, fiberglass, and foam samples were vacuum baked at ~55°C for at least 12 hours at 10⁻⁶ torr or better prior to weighing (the cure temperature of the fiberglass mesh was 65°C).

B. Sample Weighing

All samples were weighed before and after exposures using a Sartorius MC5 Microbalance. The precision of this microbalance was 1 μg. A weak alpha radioisotope was attached inside the weighing chamber to reduce electrostatic charging. To quantify the sample mass variation due to ambient water adsorption, an unexposed “control” sample was weighed in between each test sample. Since the masses of the test and control samples were similar, one could assume, to lowest order, that the difference between test and control masses, or “relative” test mass, should be constant with variation in humidity. Hence, total sputter yield was calculated using change in “relative” mass after ion exposure. Figure 1 shows control sample mass variation with relative humidity for the Kapton FPC and polyimide foam samples.

Each dielectric sample was weighed 3-5 times before and after each exposure (interleaved with an equal or greater number of control mass measurements). The samples were left in the weighing chamber of the microbalance until the rate of mass drift was sufficiently low. Due to the variation in dwell times, time stamps for each measurement were recorded, such that the time-interpolated “relative” mass could be determined.

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C. Exposure Setup

Sample exposures took place in an 2.6m-diameter vacuum chamber with a nominal pumping speed of 90 kL/s for air and a base pressure of $3 \times 10^{-8}$ torr. This low pressure minimized contamination/oxidation of samples during and between exposures. Figure 2 shows the simplified setup of the sputtering experiment. An array of up to 13 sample targets, on sample holders canted at defined angles, were placed on a moveable platform behind a large aluminum mask. The moveable stage allowed sequential exposure of all targets. A total of 3 sets of 12 samples were tested.

To provide accurate estimates of total ion fluence (integrated ion current) during sample exposures, ion beam current was measured on the molybdenum samples (biased -18V relative to ground to reject electrons). Each dielectric sample was paired with an adjacent molybdenum sample with identical incident angle. Hence, the current on the molybdenum sample should be nearly identical to the current on an adjacent sample (with a reduction of 1.5 for the smaller foam samples). The total fluence on the dielectric samples could then be interpolated from measurements made on the molybdenum samples every 30 minutes.

Figure 3 shows the sample holder for each type of sample. The base of the Teflon holder was exactly the dimensions of the sample, usually with chamfered edges, to prevent direct sputtering of the holder and block back-sputtered material from contaminating the back of the samples. A separate measurement verified that the temperature of the samples in these isolated holders should not exceed ~40°C, well below the thresholds where decomposition
and other undesirable processes might occur.

Care was taken to minimize the cross-sectional area of the retaining bolts and washers on the front face of the holder, to minimize spurious transfer of material from the components to the sample. When possible, low sputter yield materials were used to minimize cross-contamination. Low sputter yield Kapton washers were used to retain the fiberglass mesh. These washers also shielded the sample from contamination by the stainless steel bolts. The foam samples were retained with low-profile, low sputter yield titanium clips on either side. On the bottom edge of each foam sample, a Kapton sheet was attached as a mask. This was to prevent exposure of the leading edge of the sample to the beam for off-normal incidence conditions.

D. Ion Source & Hollow Cathode

The monoenergetic beam of Xe$^+$ ions was produced by Commonwealth Scientific 3 cm Kauffman gridded ion source. The pyrolytic graphite screen and accel grids of this source allowed for low energy beams with minimal sputter erosion of the grids (minimizing a potential source of sample contamination). Due to the high pumping speed and low propellant utilization efficiency, the charge exchange ion content from this source was negligible (as verified with a retarding potential analyzer (RPA)), as was the doubly and triply charged ion content (as verified with an ExB probe). Hence, the accelerated particles from the ion source reaching the sample all had the same velocity and charge state, and any current measured on the samples could be converted directly to particle flux.

Due to a quirk of the ion beam power supply, the ion beam was found to have a nontrivial energy distribution, particularly at low energy. This resulted in energy uncertainty in the final total sputter yield plots. The AC component of the beam power supply, measured with a digital multimeter, was found to be linearly correlated with the width of the ion energy distribution, measured with an RPA.

As discussed in [1], sample surface neutralization is critical for getting accurate low energy sputter yield measurements. Inadequate neutralization causes deceleration of incident ions, resulting in lower apparent sputter yields. Hence, the standard filament neutralizer for our ion source was replaced by a more powerful hollow cathode. This cathode, provided by NASA GRC, was a development model for the ISS plasma contactor. As such it was capable of providing orders of magnitude more neutralizing electron current to both the samples and the ion beam than is provided by the ~10 mA ion source. The plasma density at the samples due to the cathode alone is ~10$^5$/cm$^2$.

III. Molybdenum

Table 1 summarizes the results of the molybdenum exposures. Figure 4 shows plots of the data, as a function of fluence, angle, and energy. The dependence on fluence is very small, indicating that the surface morphology is not changing significantly over the range of exposure considered in this study. At 274 eV, TSY appears lightly peaked between 55 and 60 degrees. Finally, the energy dependence between 150-350 eV is nearly linear, indicating we are not near threshold. There was no obvious change in appearance of the samples after exposure.

Table 1. Molybdenum Total Sputter Yield

<table>
<thead>
<tr>
<th>#</th>
<th>Angle (deg)</th>
<th>Energy (eV)</th>
<th>Fluence (C)</th>
<th>$\Delta m_f - \Delta m_i$ (μg)</th>
<th>TSY (g/C)</th>
<th>TSY (atoms/ion)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.6 +2.3 -3.2</td>
<td>273.0 +/- 9.5</td>
<td>2.785 +/- 0.061</td>
<td>1237 +/- 11</td>
<td>4.48E-04 +/- 2.84E-05</td>
<td>0.451 +/- 0.029</td>
</tr>
<tr>
<td>2</td>
<td>4.6 +2.3 -3.2</td>
<td>272.9 +/- 9.5</td>
<td>5.879 +/- 0.231</td>
<td>2604 +/- 11</td>
<td>4.47E-04 +/- 2.91E-05</td>
<td>0.450 +/- 0.029</td>
</tr>
<tr>
<td>3</td>
<td>4.6 +2.3 -3.2</td>
<td>273.3 +/- 9.5</td>
<td>9.021 +/- 0.521</td>
<td>3876 +/- 11</td>
<td>4.34E-04 +/- 4.01E-05</td>
<td>0.436 +/- 0.040</td>
</tr>
<tr>
<td>4</td>
<td>29.8 +3.6 -4.6</td>
<td>272.9 +/- 9.5</td>
<td>5.173 +/- 0.007</td>
<td>4720 +/- 11</td>
<td>9.21E-04 +/- 3.82E-06</td>
<td>0.926 +/- 0.004</td>
</tr>
<tr>
<td>6</td>
<td>29.8 +3.6 -4.6</td>
<td>272.9 +/- 9.5</td>
<td>3.091 +/- 0.004</td>
<td>2375 +/- 11</td>
<td>7.76E-04 +/- 7.27E-05</td>
<td>0.780 +/- 0.073</td>
</tr>
<tr>
<td>7</td>
<td>44.6 +3.0 -4.3</td>
<td>273.1 +/- 9.5</td>
<td>2.926 +/- 0.005</td>
<td>2779 +/- 11</td>
<td>9.59E-04 +/- 1.12E-04</td>
<td>0.964 +/- 0.112</td>
</tr>
<tr>
<td>10</td>
<td>54.5 +2.5 -3.9</td>
<td>273.2 +/- 9.5</td>
<td>3.229 +/- 0.038</td>
<td>3215 +/- 11</td>
<td>1.01E-03 +/- 1.12E-04</td>
<td>1.011 +/- 0.113</td>
</tr>
<tr>
<td>8</td>
<td>59.5 +2.2 -3.4</td>
<td>273.5 +/- 9.5</td>
<td>2.271 +/- 0.027</td>
<td>2222 +/- 11</td>
<td>9.87E-04 +/- 1.85E-04</td>
<td>0.993 +/- 0.186</td>
</tr>
<tr>
<td>11</td>
<td>69.5 +1.6 -2.3</td>
<td>273.7 +/- 9.5</td>
<td>1.962 +/- 0.004</td>
<td>1728 +/- 11</td>
<td>8.89E-04 +/- 1.89E-04</td>
<td>0.894 +/- 0.190</td>
</tr>
<tr>
<td>9</td>
<td>74.6 +1.2 -1.8</td>
<td>273.8 +/- 9.5</td>
<td>1.429 +/- 0.004</td>
<td>1340 +/- 11</td>
<td>9.47E-04 +/- 3.28E-04</td>
<td>0.952 +/- 0.330</td>
</tr>
<tr>
<td>13</td>
<td>5.1 +3.0 -2.9</td>
<td>169.2 +/- 18.0</td>
<td>4.230 +/- 0.027</td>
<td>1067 +/- 11</td>
<td>2.55E-04 +/- 4.69E-05</td>
<td>0.256 +/- 0.047</td>
</tr>
<tr>
<td>12</td>
<td>4.4 +2.3 -3.0</td>
<td>376.3 +/- 1.2</td>
<td>11.317 +/- 0.098</td>
<td>7309 +/- 11</td>
<td>6.52E-04 +/- 2.17E-05</td>
<td>0.656 +/- 0.022</td>
</tr>
</tbody>
</table>

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<table>
<thead>
<tr>
<th></th>
<th>4.6</th>
<th>+2.3 - 3.2</th>
<th>376.5 +/- 1.2</th>
<th>9.093 +/- 0.079</th>
<th>6042 +/- 11</th>
<th>6.71E-04 +/- 6.06E-06</th>
<th>0.675 +/- 0.006</th>
</tr>
</thead>
</table>

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A. Calculation of Angle, Energy, and TSY

Note that a variety of adjustments have been made to provide corrected energy, angle, and TSY with appropriate uncertainty estimates. For the energy, the beam power supply voltage was used, with a +18 eV correction due to the accelerating bias on those samples and a correction (roughly 1.5 eV for a 250 eV beam) for the potential drop across the 1 kΩ current sensing resistor. As a result the mean energy for the molybdenum samples is slightly higher than that for the unbiased dielectric samples.

While the nominal incident angle is given by the angle of the entire target relative to the angle of the final grid of the ion source, this does not include the fact that ions exiting the source have a narrow angular distribution. Since both the source and the target are extended objects, the incident angle at each point on the sample is best described by a distribution. The effect is strongest at normal incidence, where the incident angle, on average, is 4-5 degrees due to ions traveling diagonally to the corners of the sample.

The total sputter yield, in units of g/C, is given by the following expression, where $\Delta m_i$ is the initial difference in mass between the test sample and the control sample, $\Delta m_f$ is the final mass difference, $Q$ is the fluence (time-integrated current) on the sample, and $\beta_A=0.9906$ is fraction of the sample exposed to the beam (e.g. due to masking by the bolt):

$$TSY = (\Delta m_f - \Delta m_i)/(Q\beta_A) \ (1)$$

As discussed below, each factor in this expression has an associated uncertainty, which must be added in quadrature to give the total uncertainty for TSY.

Since the currents on the samples were monitored continuously, the uncertainty in fluence was very low, <1%. Since the samples doubled as Faraday cups, each sample was exposed intermittently for 17 cycles on average. A conservative estimate of the fluence uncertainty was derived from the standard deviation of the mean of average currents from each cycle.

The uncertainty in the mass measurement was also vanishingly small, since humidity was not a factor, the samples were measured only once before and once after exposure. The variation in control mass over the entire test campaign (including error due to microbalance zero offset), 11 µg was used as a worst-case mass uncertainty.

B. Background Contamination

The above discussion would suggest that TSY uncertainty should be <1%. However, as shown in Table 1, there is a 16% variation between the two measurements at $E=273eV$, $\theta=30^\circ$. One key difference between these two measurements is that sample 4 was exposed continuously for 1 hour while sample 6 was subjected to intermittent

![Figure 4. Xe+ molybdenum total sputter yield. Plotted as a function of ion fluence at 274eV, 4.6° incidence (top); ion energy at 4.6° (middle); and average incident angle at 274 eV (bottom). Latter is compared with Kolasinski results (error bars not shown) [8].](image)
exposures every 30 minutes, spread over 7 hours, with a cumulative exposure of only 0.5 hours. A compelling explanation for the difference in TSY for these samples is background contamination. The 30 minute intermissions in principle allow for repeated accumulation of contaminants, which must be removed at the beginning of each exposure. This should reduce the net mass loss and derived TSY at the end of the test.

The background contamination flux can be calculated from kinetic theory of an ideal gas [3]:

\[ \Gamma_{BG} = \frac{8000 N_A \sqrt{2 \pi}}{3 \sqrt{mRT}} \]  

Here \( N_A \) is Avogadro’s constant, \( M \) is the atomic mass in amu of the impacting species, \( k \) is Boltzmann’s constant in J/K, \( T \) is temperature in K (assumed to be 300K), and \( P \) is pressure in Pa. With a background pressure of 3e-8 torr, \( \Gamma_{BG} = 2.7e17 \text{m}^{-2} \text{s}^{-1} \) if the contaminant is assumed to be N\(_2\) (\( M=28 \)) or \( \Gamma_{BG} = 3.3e17 \text{m}^{-2} \text{s}^{-1} \) if the contaminant is assumed to be water (\( M=18 \)). By comparison, the ion flux at 250eV is \( \Gamma_i = 6.4e18 \text{m}^{-2} \text{s}^{-1} \). This results in the following steady-state concentration of adsorbed contaminants on the sample [3,4]:

\[ c_{BG} = \frac{s_{BG} \Gamma_{BG}}{s_{BG} \Gamma_{BG} + Y_i \Gamma_i} \]

Here \( Y_{BG} \) is the TSY of background gas in atoms per ion and \( s_{BG} \) is the sticking coefficient of the contaminant. Assuming \( s_{BG} = 0.2 \) [3,5] and \( Y_{BG} = 0.13 \) [3,6] (using surface density of solid N\(_2\) and cross section at 300eV), \( c_{BG} = 0.06 \) for \textit{continuous} exposure. This meets the proposed experimental requirement of Anderson and Bay that the concentration of contaminant on the surface is less than 10% [4].

In contrast, during intermittent exposure, contaminant species can accumulate on the samples between exposures. With the assumed background flux and sticking coefficient for N\(_2\), the molybdenum sample could accumulate an additional 6.5\( \mu \)g of material in 30 minutes. While it is questionable that N\(_2\) alone could accumulate that many monolayers, other background species may contribute as well, such as water, oxygen, carbon, and assorted decomposed hydrocarbon radicals emitted from the adjacent dielectrics samples.

If we assume that the difference between samples 4 and 6 is entirely due to contamination build-up, we can calculate the empirical fluence required to remove the contaminant layer during each of the 15 intermittent exposures from the respective yields, \( Y \), of the samples:

\[ Q_{BG} = \frac{(Q_{6}/15)(Y_6-Y_4)}{Y_4} = 32.5 \text{mC}(4) \]

If we assume a contaminant sputter yield of 0.13 molecules/ion and a mean molecular mass of 28, we get a meager 1.2\( \mu \)g of contamination. If we assume a contaminant density of 0.25g/cm\(^3\), that corresponds to a mere 3 nm, which does not seem unreasonable. Lisowski et al. found contamination layers as thick as 8 nm on molybdenum sheet for scanning tunneling microscope applications [7].

With this in mind, one can calculate the fraction of fluence for each sample devoted to contamination removal, \( \beta_{BG} = Q_{BG}/Q_i \), where \( Q_{exp} \) is the number of exposures. In principle, the total sputter yield should then be enhanced by a factor of \( 1/(1-\beta_{BG}) \). The only other sample exposed continuously was sample 5, at 376eV, with normal ion incidence. This can be compared to sample 12, which was exposed intermittently. At 376eV, \( \beta_{BG} \) should become negligible due to increased fluence per exposure and presumably higher TSY for any contamination layers. Indeed, \( Y_{12}/Y_{5} \approx 1 \), consistent with expectations.

Although two data points appear consistent with this contamination hypothesis, it is not possible to entirely rule out other systematic effects, like spurious pre-exposure contamination (e.g. oil) or removal of mass by scratching or abrasion on sample 6 alone, or some other unidentified error. Furthermore, since the sputter yield of the contaminant layer as a function of incident angle and energy is unknown, it is difficult to predict how this contamination adjustment will affect other samples.

As a hedge, instead of rescaling, a fractional uncertainty factor, \( \beta_{BG}/2(1-\beta_{BG}) \), was added to error, in quadrature. This ensured that the error bars of samples 4 and 6 overlapped while generating enhanced uncertainties for samples with a low rate of mass loss. As discussed in the next section, this produced results that appeared consistent with historical values (although it is possible some of those historical values are incorrect due to contamination effects).

C. Comparison to Historical Data

The selection of molybdenum as a sputter target was motivated by the considerable historical TSY data available on this material. If the results of this study have reasonable consistency with historical data, it can be viewed as a
makes sense because at a microscopic level, the foam looks nearly identical at all incident angles. Even the energy exposed to ~50% of nominal flux but similar fluence to sample 2. The angular dependence is also fairly flat. This and energy. The fluence dependence for foam at 253eV appears fairly flat, within error bars. Note that the considerable spread in literature values is due to both methodology and the variation in initial surface condition of the samples. Also note that while the Aerospace data is nominally normal incidence, the actual average incidence angle is nonzero due to nontrivial sample size and divergence of the ion beam.

Figure 4c compares angular dependence from the present data to 300eV data from Kolasinski [8]. That latter data was collected by in-situ measurements of sputter erosion mass change from a molybdenum-coated quartz crystal microbalance. The data show good agreement, within the error bars, although the Aerospace/Maxar data tends toward slightly higher TSY values, particularly at high angles.

IV. Solimide (Polyimide) Foam

Table 2 summarizes the data for Solimide foam. Figure 6 shows plots of the data, as a function of fluence, angle, and energy. The fluence dependence for foam at 253eV appears fairly flat, within error bars. Note that sample 3 was exposed to ~50% of nominal flux but similar fluence to sample 2. The angular dependence is also fairly flat. This makes sense because at a microscopic level, the foam looks nearly identical at all incident angles. Even the energy dependence is weak, but linear, in the 150eV to 350eV range.

Table 2. Solimide (Polyimide) Foam Total Sputter Yield

<table>
<thead>
<tr>
<th>#</th>
<th>Angle (deg)</th>
<th>Energy (eV)</th>
<th>Fluence (C)</th>
<th>Δm- Δm (μg)</th>
<th>TSY (g/C)</th>
<th>TSY (atoms/ion)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.2 ± 4.5 - 1.3</td>
<td>252.8 ± 9.5</td>
<td>8.280 ± 0.466</td>
<td>101 ± 20</td>
<td>1.22E-05 ± 2.48E-06</td>
<td>0.093 ± 0.019</td>
</tr>
<tr>
<td>2</td>
<td>4.2 ± 4.5 - 1.3</td>
<td>252.8 ± 9.5</td>
<td>18.324 ± 0.715</td>
<td>221 ± 30</td>
<td>1.21E-05 ± 1.69E-06</td>
<td>0.092 ± 0.013</td>
</tr>
<tr>
<td>3</td>
<td>4.2 ± 4.5 - 1.3</td>
<td>252.8 ± 9.5</td>
<td>19.136 ± 2.990</td>
<td>286 ± 38</td>
<td>1.49E-05 ± 3.07E-06</td>
<td>0.113 ± 0.023</td>
</tr>
<tr>
<td>4</td>
<td>29.7 ± 3.7 - 5.0</td>
<td>252.8 ± 9.5</td>
<td>14.988 ± 0.067</td>
<td>202 ± 28</td>
<td>1.35E-05 ± 1.87E-06</td>
<td>0.102 ± 0.014</td>
</tr>
<tr>
<td>5</td>
<td>44.5 ± 2.9 - 4.3</td>
<td>252.8 ± 9.5</td>
<td>15.570 ± 0.051</td>
<td>199 ± 34</td>
<td>1.28E-05 ± 2.16E-06</td>
<td>0.097 ± 0.016</td>
</tr>
<tr>
<td>6</td>
<td>54.5 ± 2.5 - 4.0</td>
<td>252.8 ± 9.5</td>
<td>14.350 ± 0.912</td>
<td>225 ± 30</td>
<td>1.57E-05 ± 2.29E-06</td>
<td>0.119 ± 0.017</td>
</tr>
<tr>
<td>7</td>
<td>59.5 ± 2.4 - 3.4</td>
<td>252.8 ± 9.5</td>
<td>10.206 ± 0.496</td>
<td>170 ± 25</td>
<td>1.66E-05 ± 2.60E-06</td>
<td>0.126 ± 0.020</td>
</tr>
<tr>
<td>8</td>
<td>69.5 ± 1.6 - 2.4</td>
<td>252.8 ± 9.5</td>
<td>8.320 ± 0.056</td>
<td>158 ± 21</td>
<td>1.90E-05 ± 2.50E-06</td>
<td>0.144 ± 0.019</td>
</tr>
<tr>
<td>9</td>
<td>74.6 ± 1.2 - 1.9</td>
<td>252.8 ± 9.5</td>
<td>6.490 ± 0.048</td>
<td>119 ± 17</td>
<td>1.84E-05 ± 2.64E-06</td>
<td>0.139 ± 0.020</td>
</tr>
<tr>
<td>10</td>
<td>4.7 ± 2.5 - 3.0</td>
<td>149.1 ± 18</td>
<td>16.628 ± 0.395</td>
<td>180 ± 24</td>
<td>1.09E-05 ± 1.46E-06</td>
<td>0.082 ± 0.011</td>
</tr>
<tr>
<td>11</td>
<td>4.1 ± 4.1 - 0.6</td>
<td>357.5 ± 1.2</td>
<td>39.168 ± 1.554</td>
<td>618 ± 81</td>
<td>1.58E-05 ± 2.16E-06</td>
<td>0.120 ± 0.016</td>
</tr>
</tbody>
</table>

Figure 5. Molybdenum total sputter yield as a function of energy. Compared with selected previous studies [8-10]. Power law fit to present data is shown, with formula inset.
A. Calculation of Angle, Energy, and TSY

TSY calculation and error analysis for the foam was similar to that for molybdenum, with a few differences. The foam samples were not externally biased and their surface potential was assumed to be the floating potential of the plasma (3.6V at 250eV), resulting in a more modest correction to incident ion energy. The average incident angle for the foam samples was slightly smaller than the angles calculated for molybdenum due to the reduced sample area.

B. Comparison to Historical Data

To the author’s knowledge, there are no studies of Solimide foam sputter yield in the public domain. However, since Solimide foam is polyimide-based, Kapton should be a close analogue. Assuming the mean mass of atoms in the polymer is 12.7 amu, the TSY can be converted to units of atoms/ion.

Reference source not found.

shows a comparison of Aerospace data with that of Yalin for Kapton sheet at 250eV [1]. Note that the Yalin study used a different methodology: the mass loss was inferred by integration of extrapolated differential sputter yield data measured with a QCM. Only condensable sputter product sticks to the QCM. The lower Yalin values shown in the plot are TSY assuming 100% condensation of sputter products. The upper Yalin values include the maximum extrapolated contribution from non-condensable sputter products. Because of these approximations, these TSY results are expected to be less accurate than direct measurements of mass loss.

Measurement differences aside, the Solimide and Kapton data are clearly on the same scale (they are nearly identical at normal incidence). Differences in the angular dependence and magnitude can be attributed to the unique geometry of the foam. The many-angled microscopic facets inside the foam average away the macroscopic angular dependence in TSY. The porousness of the material means the incident ions can travel deep into the foam and a fraction of the resultant sputter products, particularly carbon, can be absorbed by the material on the way out to the surface.

C. Additional Diagnostics

As shown in Figure 7, the foam samples develop and darkened “toast”-like appearance on the surface following exposure. This dark residue appears to extend a short distance into the bulk of the material. The darkening way out to the surface.

Figure 6. Xe⁺ Solimide (polyimide) foam total sputter yield. Plotted as a function of ion fluence (top) at 253eV, 4.6° incidence; ion energy at 4.2° (middle); and average incident angle at 256eV (bottom). Latter compared with results from Yalin et al. for Kapton HN sheet at 250eV [1].

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progressively increases with erosion or exposure. Conservative calculations indicate contamination from the aluminum aperture and the carbon grids of the ion source should be orders of magnitude below background.

In order to understand the cause of darkening in the Solimide foam and its lower-than-expected TSY, a number of additional diagnostics were performed on a pristine sample and an exposed sample (sample 2, 253eV at normal incidence). Both were examined under a conventional microscope. As shown in Figure 8, both had similar porous cell structure with little obvious differences other than a “smoky” coating on the exposed samples.

Electron Dispersive X-ray Spectroscopy (EDS) was performed on both samples to determine if there were any differences in elemental content. There was a small signal attributed to silicon observed in both but no evidence of aluminum contamination. The EDS spectra collected normal to the surface of the exposed sample also had weak X-ray peaks most consistent with embedded Xenon. Overall however, there were no remarkable differences in spectra between the exposed and unexposed samples.

Figure 7. Images of front and side of Solimide foam sample #2 before (left) and after (right) Xe⁺ exposure.
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Figure 8. Microscope images of Solimide sample. Top images show the 100X magnified face of a pristine sample (left) compared to an exposed sample (sample #2, 253eV, normal incidence) (right). Bottom images show the 50x magnified edge of the same.

The last diagnostic performed was Raman spectroscopy. Figure 9 shows the spectrum of a pristine and exposed samples excited by a 785nm laser. The pristine sample exhibits sharp peaks consistent with polyimide. The exposed sample exhibits broad peaks at 1300 and 1600 consistent disordered graphitic carbon. This is matches the hypothesis that carbon sputtered off of surfaces within the foam is getting reabsorbed on the way out of the foam; hence, it also explains the lower-than-expected TSY.

Figure 9. Raman spectroscopy using a 785 nm laser. Left spectrum is from a pristine Solimide sample. Right spectrum is from an exposed Solimide sample (sample #2, 253eV, normal incidence).

V. Fiberglass Mesh

The fiberglass mesh sample is unique in that it was co-exposed to ions with a Kapton FPC backer, which would be the as-flown configuration in space. As a result fiberglass mesh sputter products can contaminate the surface of the Kapton backer and vice versa. Hence, the effective TSY of fiberglass mesh in stack-up is really a lower bound for the TSY of mesh in free space. Furthermore, the mesh is by its nature in mostly open area with zero sputter yield interspersed with strands with nonzero sputter yield. For simplicity, in this study the mesh is treated as a uniform solid. Thus, the TSY of the strands themselves can be determined by simply multiplying the mesh TSY by 5 (assuming 80% open area). Because the fiberglass mesh has a complex non-uniform composition, it was not possible to convert TSY into units of atoms/ion.

Table 3 provides the fiberglass mesh TSY data and Figure 10 plots the results as a function of fluence, angle, and energy. There appears to be a slight fluence dependence, which could be caused by a transition in sputter yield as the fibers coating is removed. The angular dependence is reasonably smooth and peaked near 70 degrees (as will be described in the next section, the large error bars on this point are due to the loss of a “flake” of material post-exposure). The energy dependence is fairly linear between 150eV to 250eV, excluding the point taken at low fluence.
Table 3. Stack-up Fiberglass Mesh Total Sputter Yield

<table>
<thead>
<tr>
<th>#</th>
<th>Angle (deg)</th>
<th>Energy (eV)</th>
<th>Fluence (C)</th>
<th>Δm= Δm (μg)</th>
<th>TSY (μg/C)</th>
<th>TSY (atoms/ion)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.6 + 2.3 - 3.2</td>
<td>252.8 +/- 9.5</td>
<td>7.980 +/- 0.016</td>
<td>162 +/- 6</td>
<td>20.9 +/- 0.7</td>
<td>--</td>
</tr>
<tr>
<td>2</td>
<td>4.6 + 2.3 - 3.2</td>
<td>252.8 +/- 9.5</td>
<td>16.790 +/- 0.000</td>
<td>244 +/- 9</td>
<td>15.1 +/- 0.6</td>
<td>--</td>
</tr>
<tr>
<td>3</td>
<td>4.6 + 2.3 - 3.2</td>
<td>252.8 +/- 9.5</td>
<td>31.730 +/- 1.388</td>
<td>433 +/- 15</td>
<td>14.1 +/- 0.8</td>
<td>--</td>
</tr>
<tr>
<td>4</td>
<td>29.8 + 3.6 - 4.6</td>
<td>252.8 +/- 9.5</td>
<td>16.767 +/- 0.045</td>
<td>262 +/- 9</td>
<td>16.1 +/- 0.6</td>
<td>--</td>
</tr>
<tr>
<td>5</td>
<td>44.6 + 3.0 - 4.3</td>
<td>252.8 +/- 9.5</td>
<td>14.148 +/- 0.076</td>
<td>262 +/- 10</td>
<td>19.1 +/- 0.7</td>
<td>--</td>
</tr>
<tr>
<td>6</td>
<td>54.5 + 2.5 - 3.9</td>
<td>252.8 +/- 9.5</td>
<td>15.363 +/- 0.045</td>
<td>327 +/- 12</td>
<td>22.0 +/- 0.8</td>
<td>--</td>
</tr>
<tr>
<td>7</td>
<td>59.5 + 2.2 - 3.4</td>
<td>252.8 +/- 9.5</td>
<td>11.142 +/- 0.043</td>
<td>243 +/- 9</td>
<td>22.6 +/- 0.9</td>
<td>--</td>
</tr>
<tr>
<td>8</td>
<td>69.5 + 1.6 - 2.3</td>
<td>252.8 +/- 9.5</td>
<td>8.642 +/- 0.109</td>
<td>242 +/- 157</td>
<td>29.0 +/- 18.7</td>
<td>--</td>
</tr>
<tr>
<td>9</td>
<td>74.6 + 1.2 - 1.8</td>
<td>252.8 +/- 9.5</td>
<td>6.941 +/- 0.024</td>
<td>188 +/- 7</td>
<td>28.1 +/- 1.0</td>
<td>--</td>
</tr>
<tr>
<td>10</td>
<td>5.1 + 3.0 - 2.9</td>
<td>149.1 +/- 18</td>
<td>16.491 +/- 0.471</td>
<td>141 +/- 5</td>
<td>8.8 +/- 0.4</td>
<td>--</td>
</tr>
<tr>
<td>11</td>
<td>4.4 + 2.3 - 3.0</td>
<td>357.5 +/- 1.2</td>
<td>40.986 +/- 0.461</td>
<td>807 +/- 29</td>
<td>20.4 +/- 0.8</td>
<td>--</td>
</tr>
</tbody>
</table>
A. Calculation of Angle, Energy, and TSY

The method for calculating energy, angle, TSY, with associated error bars is identical to that of foam. Similar to foam, one fiberglass mesh sample (sample 10) was re-weighed post-test at a different humidity. In this case mass difference was only 30µg, which is only 3.5% of the overall mass change. This error factor was uniformly included in the error calculation of all other mesh samples.

Because of the delicacy of the mesh samples, a couple additional adjustments were required for selected samples. A single fiber bundle slipped off the edge of sample 10 during the pre-exposure mounting process. The fiber bundle was preserved and weighed alongside the rest of the exposed sample at the end of the test (its mass was 5.628µg).

A more severe correction was required for sample 8. A small flake separated from this sample following exposure. This flake had the length of a single grid spacing. Unfortunately, this flake was lost during transfer. Assuming the flake was ~1/4 the cross sectional area of the fiber bundle, the flake mass was estimated to be 156µg. This value was used to offset the final mass change of the sample. The uncertainty of this estimate was conservatively estimated to be 156µg (i.e. corresponding to 0-50% of the bundle for 1 grid spacing).

VI. Kapton FPC

The Kapton FPC sheet was co-exposed to the ion beam with the fiberglass mesh. Table 4 and Figure 11 show the dependence on TSY on fluence, angle and energy. For many samples, the TSY of layup Kapton FPC was much lower than Kapton HN [1]. This suggests that sputter efflux from the fiberglass mesh was likely depositing on the Kapton FPC, adding mass and reducing the effective sputter yield. The trends in angle and energy can be understood in the context of fiberglass sputter contamination competing with Kapton FPC sputter erosion. Indeed, exposed samples of Kapton FPC exhibit a repeating pattern matching the perimeter of the mesh at the top of the layup. Presumably, the slightly darkened edges of the repeating square pattern correspond to deposition of eroded material from the mesh.

![Figure 10. Xe+ fiberglass mesh total sputter yield (as component of layup). Plotted as a function of ion fluence at 253eV, 4.6° incidence angle (top left); ion energy at 4.6° (top right); and average incident angle at](image-url)
Table 4. Stack-up Kapton FPC Total Sputter Yield

<table>
<thead>
<tr>
<th>#</th>
<th>Angle (deg)</th>
<th>Energy (eV)</th>
<th>Fluence (C)</th>
<th>Δmf - Δmi (μg)</th>
<th>TSY (μg/C)</th>
<th>TSY (atoms/ion)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.6 ± 2.3 -3.2</td>
<td>252.8 +/- 9.5</td>
<td>7.980 +/- 0.016</td>
<td>29 + 34 - 21</td>
<td>3.8 +/- 4.4 - 2.7</td>
<td>0.03 +/- 0.03 - 0.02</td>
</tr>
<tr>
<td>2</td>
<td>4.6 ± 2.3 -3.2</td>
<td>252.8 +/- 9.5</td>
<td>16.790 +/- 0.000</td>
<td>43 + 49 - 27</td>
<td>2.7 +/- 3.0 - 1.7</td>
<td>0.02 +/- 0.02 - 0.01</td>
</tr>
<tr>
<td>3</td>
<td>4.6 ± 2.3 -3.2</td>
<td>252.8 +/- 9.5</td>
<td>31.730 +/- 1.388</td>
<td>135 + 146 - 74</td>
<td>4.4 +/- 4.8 - 2.4</td>
<td>0.03 +/- 0.04 - 0.02</td>
</tr>
<tr>
<td>4</td>
<td>29.8 ± 3.6 -4.6</td>
<td>252.8 +/- 9.5</td>
<td>16.767 +/- 0.045</td>
<td>268 + 290 - 145</td>
<td>16.5 +/- 17.9 - 8.9</td>
<td>0.13 +/- 0.14 - 0.07</td>
</tr>
<tr>
<td>5</td>
<td>44.6 ± 3.0 -4.3</td>
<td>252.8 +/- 9.5</td>
<td>14.148 +/- 0.076</td>
<td>316 + 341 - 171</td>
<td>23.1 +/- 25.0 - 12.5</td>
<td>0.18 +/- 0.19 - 0.10</td>
</tr>
<tr>
<td>8</td>
<td>54.5 ± 2.5 -3.9</td>
<td>252.8 +/- 9.5</td>
<td>15.363 +/- 0.045</td>
<td>412 + 446 - 223</td>
<td>27.8 +/- 30.0 - 15.0</td>
<td>0.21 +/- 0.23 - 0.12</td>
</tr>
<tr>
<td>6</td>
<td>59.5 ± 2.2 -3.4</td>
<td>252.8 +/- 9.5</td>
<td>11.142 +/- 0.043</td>
<td>344 + 371 - 186</td>
<td>31.9 +/- 34.5 - 17.2</td>
<td>0.24 +/- 0.26 - 0.14</td>
</tr>
<tr>
<td>9</td>
<td>69.5 ± 1.6 -2.3</td>
<td>252.8 +/- 9.5</td>
<td>8.642 +/- 0.109</td>
<td>285 + 308 - 154</td>
<td>34.2 +/- 36.9 - 18.5</td>
<td>0.26 +/- 0.28 - 0.15</td>
</tr>
<tr>
<td>7</td>
<td>74.8 ± 1.2 -1.8</td>
<td>252.8 +/- 9.5</td>
<td>6.941 +/- 0.024</td>
<td>220 + 237 - 119</td>
<td>32.7 +/- 35.4 - 17.7</td>
<td>0.25 +/- 0.27 - 0.15</td>
</tr>
<tr>
<td>11</td>
<td>5.1 ± 3.0 -2.9</td>
<td>149.1 +/- 18</td>
<td>16.491 +/- 0.471</td>
<td>119 + 129 - 64</td>
<td>7.5 +/- 8.1 - 4.0</td>
<td>0.06 +/- 0.06 - 0.03</td>
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<tr>
<td>10</td>
<td>4.4 ± 2.3 -3.0</td>
<td>357.5 +/- 1.2</td>
<td>40.986 +/- 0.461</td>
<td>269 + 291 - 145</td>
<td>6.8 +/- 7.3 - 3.7</td>
<td>0.05 +/- 0.06 - 0.03</td>
</tr>
</tbody>
</table>

Figure 11. Xe⁺ Kapton FPC total sputter yield (as component of layup). Plotted as a function of ion fluence at 253 eV, 4.6° incidence (top left); ion energy at 4.6° (top right); and average incident angle at 253 eV (bottom). Results are compared with results from Yalin et al. [1] for Kapton HN (not in layup).

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Error! Reference source not found. compares the Aerospace/Maxar measurements with those of Yalin for Kapton HN [1]. Extrapolating to 150eV, it appears that the two datasets converge, suggesting that mesh contamination at that energy is reduced sufficiently that significant Kapton erosion can occur. At higher energies, contamination dominates and effective Kapton TSY grows slowly. At normal incidence, mesh contamination is particularly strong, resulting in extremely weak Kapton TSY. However, at modest angles, Kapton erosion begins to dominate and the TSY converges with the Yalin results.

The error bars for stack-up Kapton FPC are particularly large due to an unusually large uncertainty in the mass measurement. Similar to foam samples, Kapton FPC samples 4 and 10 were re-weighed at a later date. The mass loss of sample 10 doubled with a modest 5% reduction in humidity (a difference of 290ug). The mass loss of sample 4 increased by 30% with identical humidity. This suggests material is being lost well after ion exposure. It seems unlikely material is sloughing off during handling as there were no comparable mass variations during each weighing session. Perhaps the contamination layer is very slowly reacting with the air or ambient vapors and evaporating. Regardless of mechanism, a uniform error of +108%, -54% was included in addition to all the other error contributions.

VII. Conclusions

Measurements of total sputter yield were made on molybdenum, a layup of fiberglass mash and Kapton FPC, and Solimide foam as a function of exposure time, incident ion energy, and incident ion angle. The molybdenum sample results were highly repeatable and in-family with historical results, providing useful validation of the methodology. The mesh/Kapton layup sputter measurements showed clear evidence of material transfer between layers, as expected. The Kapton FPC substrate had otherwise low sputter yield, similar to unobstructed Kapton HN film. The Solimide foam had total sputter yield in-family with Kapton film (in atoms per ion), likely due to both having similar composition. The foam exhibited reduced dependence on incident angle due to its wispy microscopic surface structure. This structure also acted to recapture back-streaming elemental carbon sputtered from deeper within the foam, coating the top surface with graphitic carbon.

Acknowledgments

The authors would like to thank Taylor Matlock and Michael Nakles at the Aerospace Corporation for useful discussions. We also like to recognize Wilson Ricks, an intern at TAC who modelled the incident ion angle distributions on our samples, allowing for higher fidelity error analysis. Finally, we wish to acknowledge the technical support of Phillip Mak, Mike Worshum, Byron Zeigler, and Kevin Dorman at TAC.

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