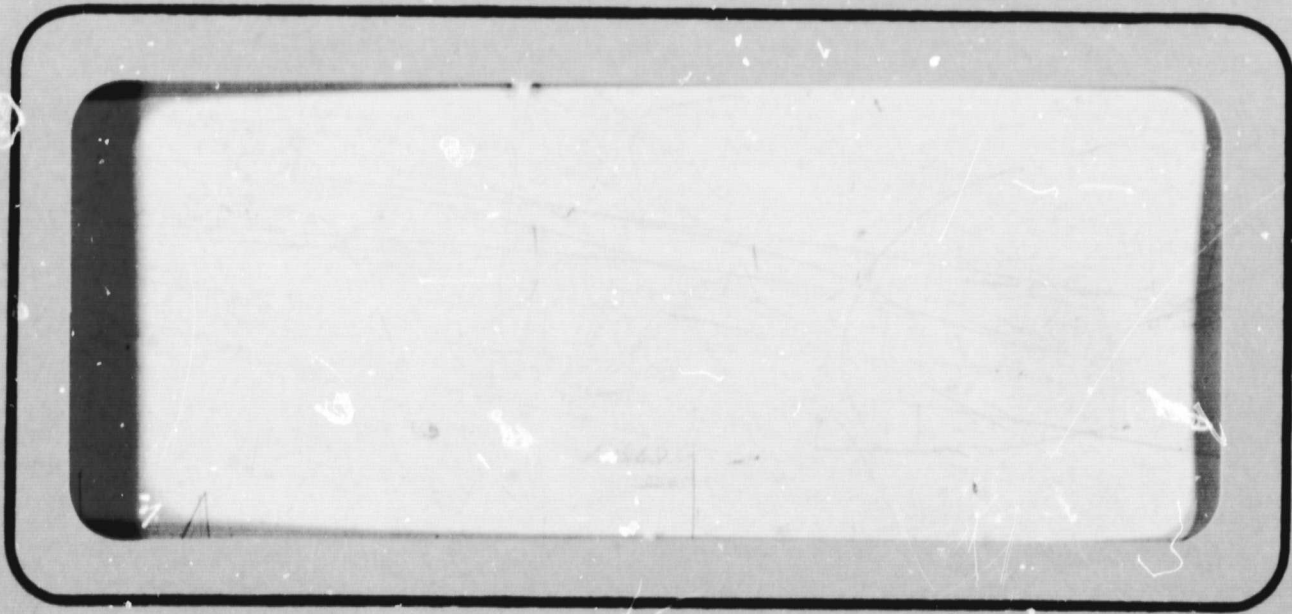


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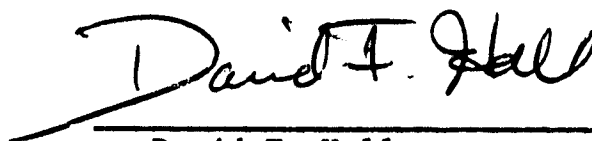
FACILITY FORM 602

ELECTROSTATIC PROPULSION BEAM DIVERGENCE EFFECTS
ON
SPACECRAFT SURFACES

Quarterly Technical Report

25 July - 23 October 1970

Contract No. 952350



David F. Hall
Program Manager

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TRW SYSTEMS
One Space Park
Redondo Beach, California

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ABSTRACT

The third phase of a program to develop understanding of an tolerance-level criteria for the deleterious effects of electrostatic rocket exhaust (Cs , Cs^+ , Hg , Hg^+) impinging on typical classes of spacecraft surfaces is underway. Phase I was completed under Contract No. NAS 7-575, while Phase II was completed under the present contract. This phase will include additional experiments and analysis leading to a modeling of contamination effects.

The theoretical optical properties of semitransparent films of typical electrostatic rocket accelerator grid materials are presented. Preliminary results of 3 KeV Hg^+ bombardment of 16 spacecraft surface materials indicate that Teflon FEP has a much higher erosion rate than the other materials tested, including several other organics. Immersion of S13G, PV100, and Z93 white paints in Cs at room temperature for 48 hours had little effect; however 3M Velvet black paint became a mottled grey. Immersion of Kapton in Cs increases its electrical conductivity by at least a factor of 10^5 .

TECHNICAL CONTRIBUTORS

Electric Propulsion

D. F. Hall

Thermophysics

W. Miller and E. E. Luedke

Chemistry

E. Wilson and R. A. Meyers

Metallurgy

R. A. Mendelson

I. INTRODUCTION

A. Background

Electrostatic rockets emit particles into at least 2π steradians. Spacecraft (S/C) designers therefore require tolerance-level criteria for the almost inevitable interception of propellant and thruster particles by S/C surfaces. During Phase I of this program, under Contract NAS7-575, a systematic analytical study was made of the deleterious effects of Hg, Hg⁺, Cs, and Cs⁺ on spacecraft surfaces.¹ Erosion of non-metallic surfaces by sputtering, degradation of thermal control coatings, chemical degradation of non-metallic surfaces, and propellant condensation on solar cell cover glasses were identified as likely to pose the most restrictive design constraints. Of the above areas, quantitative constraints were generated for condensation subsequent to the adsorption of the first monolayer; the others were at the qualitative stage and required experimental study.

In Phase II of this program the necessary experimental fixtures were fabricated and exploratory experiments were performed.² These experiments provided a basis for dismissing portions of some potential problem areas, suggested definitive experiments in other areas, and showed that still other areas require more complete exploration. Additional analysis of potential problem areas demonstrated that sputtered accelerator grid material is an important thruster effluent.

Portions of the work completed to date have been summarized in presentations to the electric propulsion community^{3,4} and in the archival literature.⁵

B. Program Goal

The goal of the present phase is to accumulate additional experimental data necessary to develop physical and/or empirical models describing propellant effects on spacecraft surfaces.

C. Program Approach and Scope

Table 1 summarizes the potential problem areas of degradation of spacecraft components by propellant interactions. Since the fields of solar electric spacecraft design and component construction are constantly evolving, a series of engineering material tests in each of these areas is not indicated. Rather, physical models of degradation effects and extrapolatable data are needed to provide the bases for tradeoff calculations and consideration of new materials and designs. It is the goal of this multi-phase program to provide these models and data with respect to cesium and mercury ion thrusters. For both of these propellant species, the effects of neutrals, ions, and mixtures of neutrals and ions are important.

Table 1. Potential degradation of spacecraft components from impinging propellants.

SPACECRAFT COMPONENTS AFFECTED DEGRADATION MECHANISM	THERMAL CONTROL COATINGS	OPTICAL ELEMENTS AND COATINGS	STRUCTURAL MATERIALS	INSULATORS AND ELECTRODE GAPS	CONDUCTORS	ADHESIVES	MOVING JOINTS	SOLID STATE COMPONENTS
PROPELLANT CONDENSATION AND/OR THRUSTER MATERIAL DEPOSITION	X	X		X				
CHEMICAL REACTION	X	X	X	X		X	X	
METALLURGICAL REACTION	X		X		X		X	
SPUTTERING EROSION	X	X	X	X	X	X	X	X
RADIATION DAMAGE (DEPOSITION OF ENERGY WITHIN SOLIDS, E.G. IONS)	X							X

X POTENTIAL PROBLEM AREA

The program contains four work units; Metallurgy, Chemistry, Thermophysics, and Electric Propulsion. The metallurgy group is charged with elucidating reactions between rocket efflux and S/C metals. The chemistry group has similar responsibility with respect to non-metallic S/C materials. The thermophysics group determines the surface thermal changes which occur in various thermal control coatings. The electric propulsion group is responsible for sputtering experiments, operation of the primary facility in which most experiments are performed, and program management.

D. Program Status

Analysis of the optical effects of thin films of sputtered accelerator grid material deposited on thermal control coatings and solar cells is well underway. A set of exposures of some 16 spacecraft materials to a mercury ion beam is also underway, and early, qualitative results are interesting. Progress has been made in defining an appropriate experiment regarding the effect of mercury on soft solder. Immersion of S13G white paint, Z93 white paint, 3M velvet black paint, and PV100 white paint and Kapton in cesium is complete.

E. References

1. Hall, D. F., "Evaluation of Electric Propulsion Beam Divergence Effects on Spacecraft," 08965-6013-RO-00, Final Report Contract No. NAS7-575, Sept. 1969, TRW Systems, Redondo Beach, Calif.
2. Hall, D. F., "Electrostatic Propulsion Beam Divergence Effects on Spacecraft Surfaces," Final Report, Vol. I, JPL Contract No. 952350, August 1970, TRW Systems, Redondo Beach, Calif.
3. Hall, D. F., Newnam, B. E., and Womack, J. R., "Electrostatic Rocket Exhaust Effects on Solar-Electric Spacecraft Subsystems," AIAA Paper No. 69-271, Williamsburg, Va., 1969.
4. Hall, D. F., and Kelley, L. R., "Experimental Techniques to Determine Electrostatic Rocket Exhaust Effects on Spacecraft Surfaces," AIAA Paper No. 70-1144, 8th Electric Propulsion Conference, Stanford University, September 1970.
5. Hall, D. F., Newnam, B. E., and Womack, J. R., "Electrostatic Rocket Exhaust Effects on Solar-Electric Spacecraft Subsystems," Journal of Spacecraft and Rockets, Vol. 7, No. 3, March 1970, pp. 305-312.

II. THERMOPHYSICS

A. Optical Effects of Thin Films

As discussed in Sect. VII A of Vol. I of the Final Report, sputtered grid material is an important high angle thruster effluent. Thin films of grid material deposited on transparent components, thermal control coatings or electrical insulators may produce significant changes in those materials. Three separate pieces of information are required to assess the magnitude of the problem:

- 1) The bulk thermophysical properties of accelerator grid materials,
- 2) The minimum film thickness at which bulk properties are obtained, and
- 3) The functional dependence of substrate properties on effective surface properties.

Preliminary calculations have been made on the properties of thin films of the following materials:

Molybdenum
Niobium (Columbium)
Aluminum
Aluminum Oxide
Silicon Dioxide

These materials are representative of both conventional and dielectric accelerator grids.

1. Bulk properties. Solar absorptance and 300°K emittance values were obtained from data available in the TRW Systems Thermophysics Section for the bulk (opaque) properties. These values are presented in Table 1.

Table 1. Measured values for bulk samples

	Solar Absorptance	300°K Emittance
Aluminum	0.12	0.04
Molybdenum	0.36	0.04
Niobium	0.56	0.13
SiO ₂	0.02	0.80
Al ₂ O ₃	0.02	0.60-0.80

2. Thin film properties. Calculation of the optical properties of thin films is discussed in detail in The American Institute of Physics Handbook¹. The complex equations given there may be used to calculate theoretical values of the spectral reflectance, absorptance and transmittance of thin films as a function of index of refraction, n , and extinction coefficient, k . The case (II) of a thin absorbent film which lies between an incident dielectric medium and a massive absorbent substrate is applicable to the problem of accelerator grid material deposited on spacecraft surfaces. The equations for this case were coded in the BASIC programming language. Given a list of film thickness, the refractive indices of the media, and the incident wavelength, the program prints a table of reflectance R , transmittance T , and absorptance A versus thickness. The program extends the list of thicknesses, if necessary, to achieve a value for T less than 1%.

Optical properties were calculated to a first approximation for various thicknesses of the metals molybdenum, niobium and aluminum using n and k values from the literature. The procedure required for each metal depended upon the amount of data available. The values of n and k at $\lambda = 0.75$ for aluminum ($n = 1.8$, $k = 7.12$) were used to calculate solar reflectance, transmittance and absorptance. This wavelength was used since it is near the spectral center of the solar irradiance. The results are shown below in Table 2.

Table 2. Aluminum -- solar region -- on glass
calculated from $n = 1.8$, $k = 7.12$

Thickness	Reflectance	Transmittance	Absorptance
1 Å	4.3%	94.4%	1.3%
10	9.0	79.6	11.4
30	25.0	52.3	22.7
100	62.9	14.2	22.9
300	76.3	10.0	13.7
1000	87.7	0	12.3

The 1000 Å thickness value compares well with a typical solar absorptance of vacuum deposited aluminum of 0.12, as shown in Table 1.

It is reported in the literature that very thin films (< 100 Å) do not obey the simple theory. The invalid assumption of the theory is that the thin films are uniform and continuous. Actually in the initial stages of the film growth, the atoms cluster in nuclei. The geometry of these nuclei depends upon the various parameters of the deposition such as the rate, angle of incidence, kinetic energy of atoms and the conditions of the substrate such as surface roughness and temperature among others. In general, the nucleation produces films having a higher transmittance and lower reflectance than uniform, continuous films of equal average thickness (assuming a highly absorbing metal film). Thus, the thin films will actually have higher transmittance and lower reflectance than the simple theory predicts. The effect upon absorptance is not so straightforward, and it is here that experimental data would be most welcome.

In order to illustrate the deviation of actual films from the simple theory, Table 3 presents some experimental data from W. Walkenhorst² reprinted in Reference 3.

Table 3. Aluminum -- solar region -- on Fluorspar
measured - Walkenhorst

Thickness	Reflectance	Transmittance	Absorptance
1 Å	4%	95%	1%
10	6	85	9
100	66	12	22
1000	88	0	12

These results compare remarkably well with the calculated values of Table 2. The films were probably prepared under ideal conditions selected to produce smooth continuous films.

Values of n and k as a function of wavelength for niobium are much more scarce than those for aluminum. The computer program was used to calculate the properties with one available set of data: $n = 1.8$, $k = 2.11$, $\lambda = .579\mu$. Using this data, the calculation gave an absorptance for a nearly opaque layer almost the same as the solar absorptance of a sample of bulk niobium. At first glance, this seems strange since 0.579μ is considerably below the center of the solar irradiance function. The spectral reflectance of niobium, however, remains fairly constant over a large part of the solar spectrum including 0.579μ , thus providing usable input data to perform calculations which will yield usable engineering property values. The results are presented below in Table 4.

Table 4. Niobium -- solar region -- on glass
 $n = 1.8$, $k = 2.11$

Thickness	Reflectance	Transmittance	Absorptance
1 Å	4.3%	95.2%	0.5%
10	5.3	89.8	4.9
100	16.8	53.2	30.3
200	27.9	32.6	39.5
1000	41.9	1.0	57.1

The 1000 Å thickness value compares well with the data for bulk niobium: $\alpha = 0.56$.

The same procedure was tried with short wavelength data on molybdenum. This time, however, the absorptance value for the opaque layer did not agree with the solar absorptance of bulk molybdenum. Therefore the values of n and k were extrapolated until an opaque film absorptance was obtained which agreed reasonably with that of bulk molybdenum. These data are presented in Table 5.

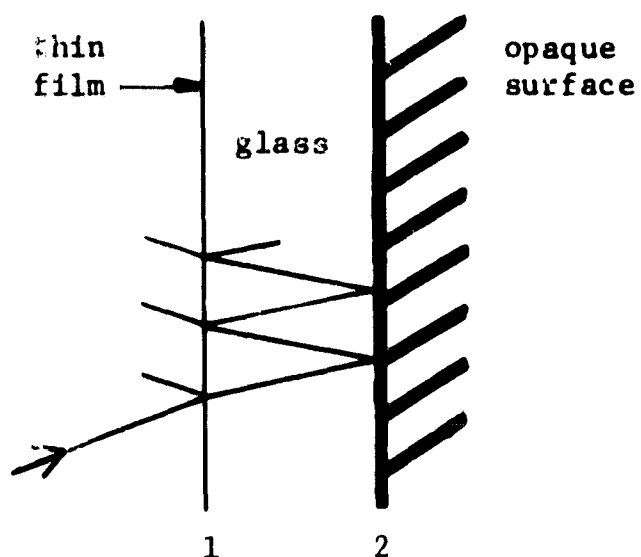
Table 5. Molybdenum -- solar region -- on glass
 $n = 3, k = 4$ (extrapolated values)

Thickness	Reflectance	Transmittance	Absorptance
1 Å	4.2%	94.6%	1.2%
10	6.5	83.2	10.2
30	12.3	64.1	23.6
100	30.7	30.8	38.5
300	57.0	7.4	35.6
1000	62.6	0.1	37.3

The 1000 Å thickness value has been made to compare well with the data for bulk molybdenum: $\alpha_s = 0.36$.

The data presented in Tables 2 and 5 are for the films on glass. This information is especially useful for evaluating the transmittance of thin films. However, evaluation of the effective value of solar absorptance and infrared emittance of a composite surface, consisting of a semi-transparent film on an opaque substrate, requires that the properties of both the film and the substrate be taken into account. The following describes the required calculational procedures.

3. Composite surface properties. The foregoing calculations gave the solar absorptance, reflectance and transmittance for thin films of the metals aluminum, molybdenum and niobium on glass. These results have been combined with the properties of solar cells to obtain the composite properties. The following general equations were used to calculate these values:



R_1, T_1, A_1 : reflectance, transmittance and absorptance at Surface 1 (the thin film).

R_2, A_2 : reflectance and absorptance at Surface 2 (the opaque substrate).

Surface 1 and 2 is completely transparent, i.e., $T_{\text{glass}} = 1.0$. Then the total reflectance of the composite surface is

$$R_T = R_1 + T_1^2 R_2 + T_1^2 R_2^2 R_1 + T_1^2 R_2^3 R_1^2 + \dots \quad (1)$$

$$= R_1 + T_1^2 R_2 \left[\frac{1}{1 - R_1 R_2} \right] \quad (2)$$

since the sum of a geometric progression is:

$$1 + X + X^2 + X^3 + X^4 + \dots = \frac{1}{1-X} \quad \text{for } |x| < 1 \quad (3)$$

Hence, the total absorptance is given by:

$$A_T = 1 - R_T = 1 - R_1 - T_1^2 R_2 \left[\frac{1}{1 - R_1 R_2} \right] \quad (4)$$

The total absorptance may also be expressed as the sum of the absorptances of the individual elements of the composite surface. Since A_1 of the light is absorbed when it passes through the thin film in either direction, the total absorptance of the film in a composite is:

$$A_{1 \text{ Total}} = A_1 + T_1 A_1 R_2 [1 + R_2 R_1 + R_2^2 R_1^2 + \dots] \quad (5)$$

$$= A_1 [1 + T R_2 M] \quad (6)$$

where $M = \frac{1}{1 - R_1 R_2} \quad (7)$

On the other hand, the total absorptance of the opaque substrate (Surface 2), including that arising from multiple reflections within the glass, is:

$$A_{2 \text{ Total}} = T A_2 [1 + R_1 R_2 + R_1^2 R_2^2 + \dots] \quad (8)$$

$$= T A_2 M \quad (9)$$

The total absorptance of the composite must be the sum of the absorptances of its elements:

$$\begin{aligned} A_{\text{Total}} &= A_1 + A_{2 \text{ Total}} = A_1 + T_1 M (R_2 A_1 + A_2) \\ &= \alpha_{\text{composite}} \end{aligned} \quad (10)$$

The following results were calculated with Equation (10) using a solar cell value of $\alpha = .78$ for A_2 :

Table 6. Niobium -- solar region -- on solar cell

α_{cell}	Thickness	α_{Nb}	T_{Nb}	$\alpha_{\text{composite}}$
0.78	0 Å			0.780
	1	.005	.952	0.756
	10	.049	.898	0.767
	100	.303	.532	0.770
	1000	.571	.010	0.581

Table 7. Molybdenum -- solar region -- on solar cell

α_{cell}	Thickness	α_{Mo}	τ_{Mo}	$\alpha_{\text{composite}}$
0.78	0 Å			0.780
	1	.012	.946	0.759
	10	.102	.832	0.759
	100	.385	.308	0.671
	1000	.373	.001	0.374

Table 8. Aluminum -- solar region -- on solar cell

α_{cell}	Thickness	α_{Al}	τ_{Al}	$\alpha_{\text{composite}}$
0.78	0 Å			0.780
	1	0.13	0.944	0.759
	10	0.114	0.796	0.768
	100	0.229	0.142	0.366
	1000	0.123	0	0.123

Calculations of the infrared emittance of the composite aluminum on quartz were made at two wavelength, 7 and 12 microns. These wavelengths were selected as being representative for quartz which has a restrahlen at 9 microns. Quartz is representative of solar cell cover glass, and, since $T \approx 0$ in the infrared, also glass covered solar cells, optical lenses, etc.

Table 9. Aluminum -- infrared region -- on quartz

Normal Emittance, ϵ_N

Film Thickness	$\lambda=7\mu$	$\lambda=12\mu$	Total*
1 Å	.93	.81	.84
3	.83	.71	.74
10	.64	.46	.50
30	.24	.23	.23
100	.08	.07	.07
300	.03	.03	.03
1000	.02	.02	.02

*Based on a weighted average of 7 and 12 micron data of 25% and 75%, respectively.

Since no infrared data was found for other metals of interest, further calculations have not been made. However, the present data provide general information which can be used in a qualitative examination of problem areas.

4. Dielectric thin films. The possibility of Al_2O_3 or SiO_2 coatings on thruster grids raises the possibility of thin films of these dielectrics being deposited on the spacecraft components.

When these materials are deposited under conditions favoring low contamination, they are non-absorbing in the solar region. In other words, deposited layers of pure SiO_2 or Al_2O_3 would not affect the solar absorptance, at least under ideal conditions of deposition. If the actual conditions of deposition introduced impurities, as would be the case with dielectric grid sputtering, it is possible that solar absorptance would be affected synergistically. Experimental data would be required to assess the magnitude of such an effect.

The emittance of these dielectrics is high, and would not adversely affect solar cell cover glasses or paints. However, if they were deposited on metals used for their low emittance characteristics, these surfaces would be affected as dielectric thickness increased, since the emittance would increase.

B. General Remarks

The calculations performed to date allow several comments related to the effect of thruster grid materials on spacecraft components. These observations will be used to define future work in this area.

1. Solar arrays. The output of the solar array is a function of two factors controlled by thruster effluents: cover glass transmittance and cell temperature. As shown in Table 5, a 10 Å film of molybdenum reduces the transmittance to the cell by approximately 10%. The cell solar absorptance is essentially unchanged for this thickness, but the emittance is considerably reduced. Thus, the temperature of the array would be increased significantly. Of course, in this case the effect is the same whether the

deposit is on the front or the back of the array. Thicker films of molybdenum would even more drastically increase the temperature of the array due to rapid increase in emittance as a function of molybdenum thickness. The next step of the analysis will be to compute solar array performance degradation as a function of film thickness, and determine the thickness regimes over which transmittance and temperature effects predominate.

2. Thermal control coatings. Although specific calculations for grid thruster effluents on white coatings have not been made to date, trends can be projected based on the calculations for solar cells. Once again the emittance will change more rapidly than absorptance. For white coatings it is expected that the absorptance will not dramatically change, since both the coatings and the metals have low to moderate absorptance. The emittance, however, is expected to change in a manner similar to that shown in Table 9. Thus, 100 Å of aluminum (and presumably molybdenum) on a white coating would result in a α/ϵ in the 5-10 range. This would obviously result in a high surface temperature.

3. Future work. The data which has been generated to date will be used to evaluate effects on both solar array output and temperature as a function of coating thickness and spacecraft orbit/location. The quantitative results of these calculations will guide future work. If thin films in the range of 10 to 100 Å are deemed on a major problem area, future work must be done to obtain or generate more accurate data. This could result in an experimental effort to obtain basic data; probably some limited experimental work will be desired to substantiate the general conclusions reached to date.

C. References

1. Hass, G. and L. Hadley, "Optical Properties of Metals," Chapter 6g, American Institute of Physics Handbook, 1963, pp 6-103 to 6-121.
2. Walkenhorst, W., Z. Techn. Phys. 22 (1941) 14.
3. Heavens, O.S., Properties of Thin Solid Films, p. 167 (1955).

III. ELECTRIC PROPULSION

A. Facility Improvements and Beam Diagnostics

1. Accel power supply. The new accelerator grid power supply was installed in the control console. It is far superior to the supply it replaces in both operating range and reliability. The limit on the magnitude of the potential applied is no longer set by the supply itself, and screen-to-accel grid arcs, which occasionally occur when operating with large accel potentials, no longer cause circuit breakers to actuate and shut down the thruster. Broader, more uniform and dilute ion beams are associated with higher accel potential.

2. Sample shutter probes. A second major improvement in the spacecraft surface contamination experimental facility was fitting the sample shutter with the array of five ion current density probes as shown in Figure 1. These j_+ probes provide a direct measurement of the ion beam intensity and uniformity prior and subsequent to sample exposures. The probes have 1/4-inch diam. entrance apertures, and are of the gridless type described previously. Installation of the probes provided the occasion for fabrication and installation of a master instrumentation patch panel on the console.

Ion current density profile data was obtained with this array. Figure 2 is a composite of the pertinent information. At the top, the j_+ probe array is shown and data point symbols assigned. The profiles displayed at the center of this figure represent the normalized current density along the perpendicular diagonals of the 5 probe array. Data taken with the smallest (-2.5 Kv) and the largest (-5 Kv) accelerator grid potentials employed is shown. It is seen that beam uniformity is quite good at -2.5 Kv and, as expected, is improved when greater accel grid potentials are applied. The inset at the bottom displays the relationship between grid potential and beam uniformity quantitatively. Its ordinate is the average value of the j_+ probe currents at the four corners of the array divided by that of the center probe.

Beam uniformity averages less than $\pm 5\%$ at -2.5 Kv, improves rapidly with increasing potential to about -3.5 Kv, and then more slowly until a $\pm 1\%$ value is obtained at -5 Kv. No attempt was made to exceed -5 Kv, since under these conditions the potential difference between the closely spaced screen and accel grids is 8 Kv and severe breakdown is imminent.

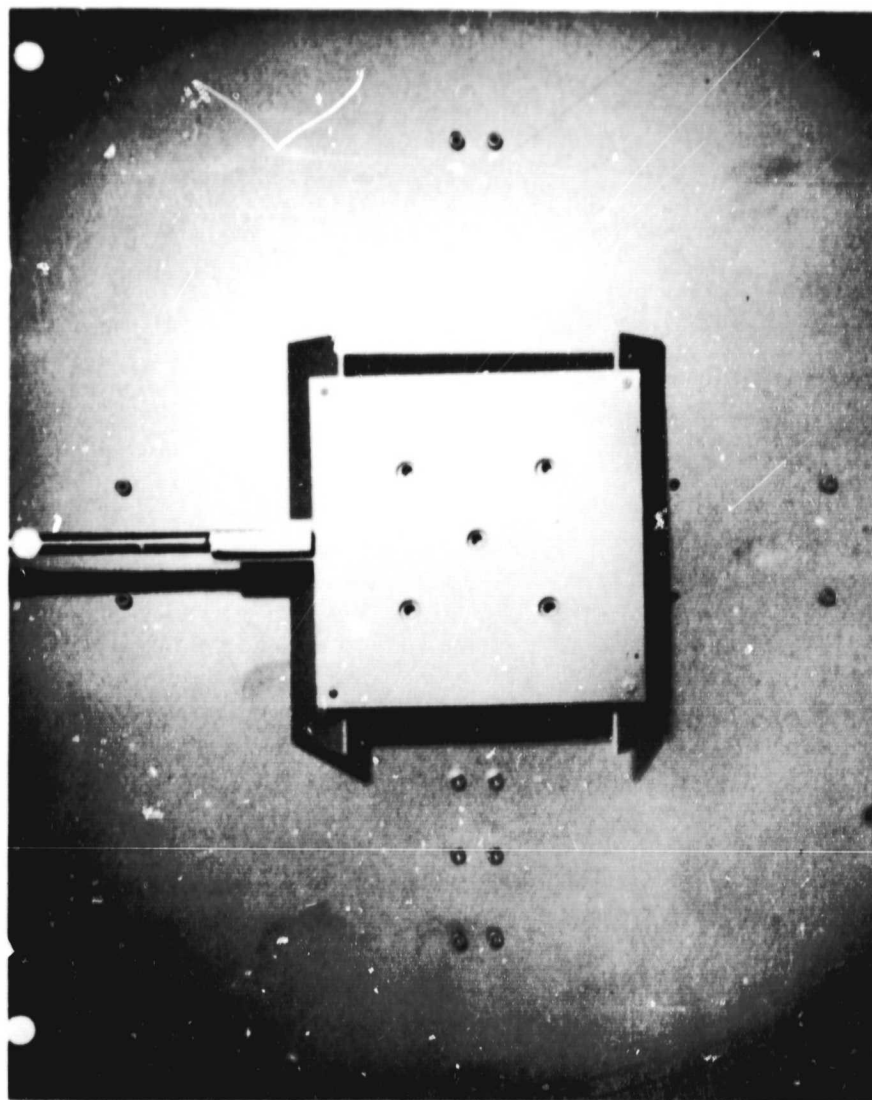


Figure 1. Photograph of sample shutter in "closed" position in front of collector cutout, showing array of 5 ion current density measuring probes. Probe spacing indicated in Figure 2.

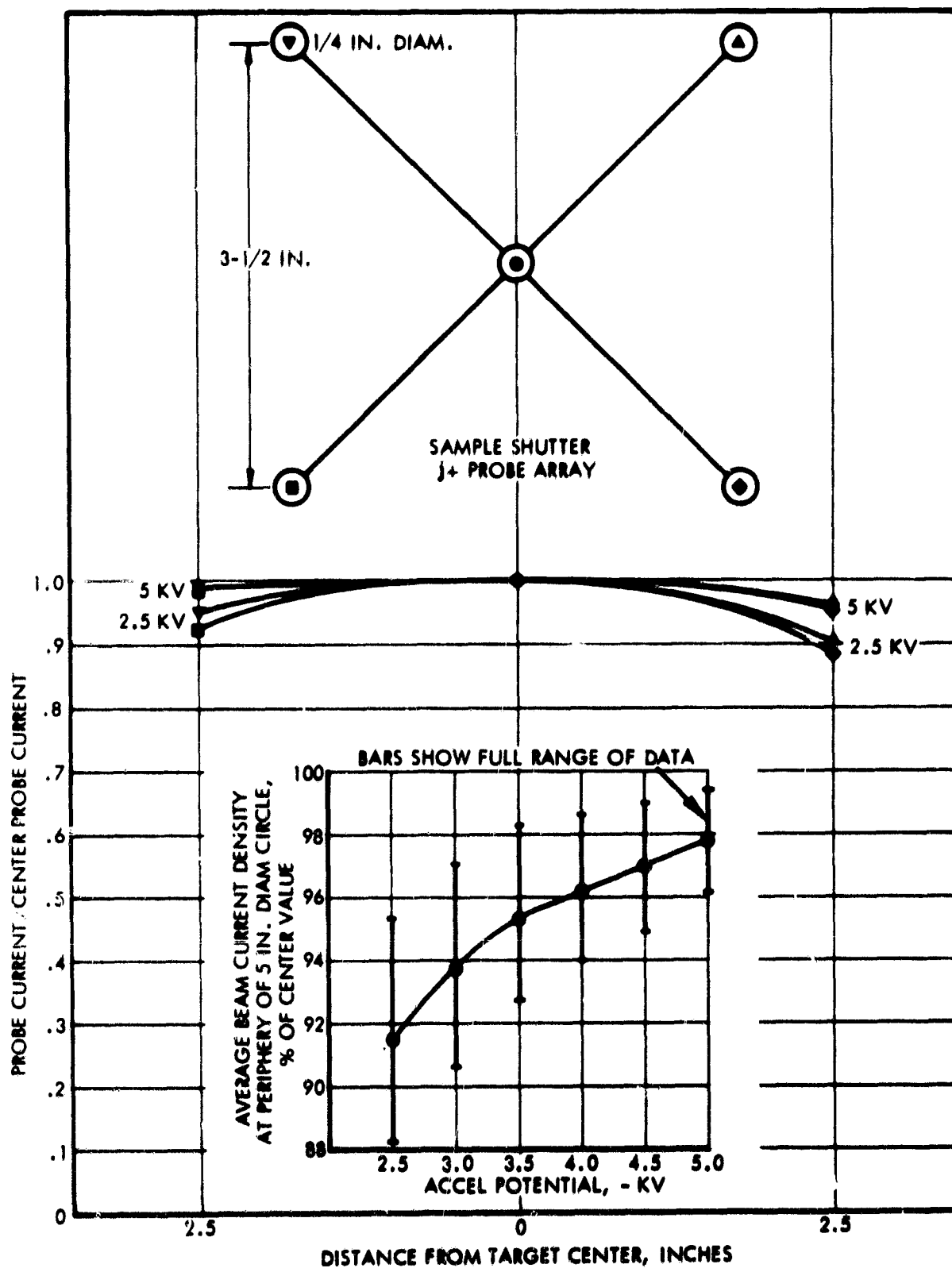


Figure 2. Ion beam current density profiles at sample plane. Top shows sensor array; center shows normalized profiles for two accel potentials; bottom shows average normalized current density profile at periphery at a 5 in. diameter circle vs accelerator potential.

B. Mercury Ion Beam Exposures

The exposure of samples of some 16 different spacecraft materials on the multipurpose sample holder to 3 KeV Hg^+ is underway. Before loading, each sample was weighed to the nearest 1/10 milligram and (with the exception of delicate Z93 and S13G) the sample thicknesses were measured at the center and at each end with a micrometer to the nearest 1/10 millinch. A similar set of measurements subsequent to beam exposure will supplement the measurement of erosion depth from the fiducial surface shielded by the heads of the sample mounting screws. RTV-60 was used as an interface compound between the sample substrates and the sample holder pipes to assure good thermal contact. Temperature is held at approximately 21°C during the exposure by passing city water through the sample pipes.

Figure 3 shows the multipurpose sample holder after initial loading with samples. The pipes are numbered from top to bottom. Figure 4 shows Pipe 4 more closely, and Figure 5 the left end of Pipe 1. Figure 6 illustrates insertion of the sample holder into the test facility, and Figure 7 shows the sample holder in the exposure position through the cutout in the titanium ion beam collector.

The first ion beam exposure was two hours in duration at approximately $1/4 \text{ ma/cm}^2$ ion current density. This exposure duration was chosen in anticipation that a significant portion of the approximately 1 mil thickness of the silver plated sample would then be eroded away. (Silver has a high sputtering yield among metals.) This expectation was fulfilled, but the condition of the 5 mil thick Teflon FEP samples were not anticipated. These samples were heavily eroded; in fact, the 0°, 30° and 45° samples were eroded through to the underlying adhesive! No other sample, metallic or organic, exhibited an erosion rate of this order of magnitude.

Figure 8 is a photograph of the samples after this two-hour exposure. When this photograph is compared with that of Figure 3, two major changes in visual appearance are apparent: The Teflon FEP samples appear to have turned black, and the reflectance of the Z93 and PV100 white paints has been

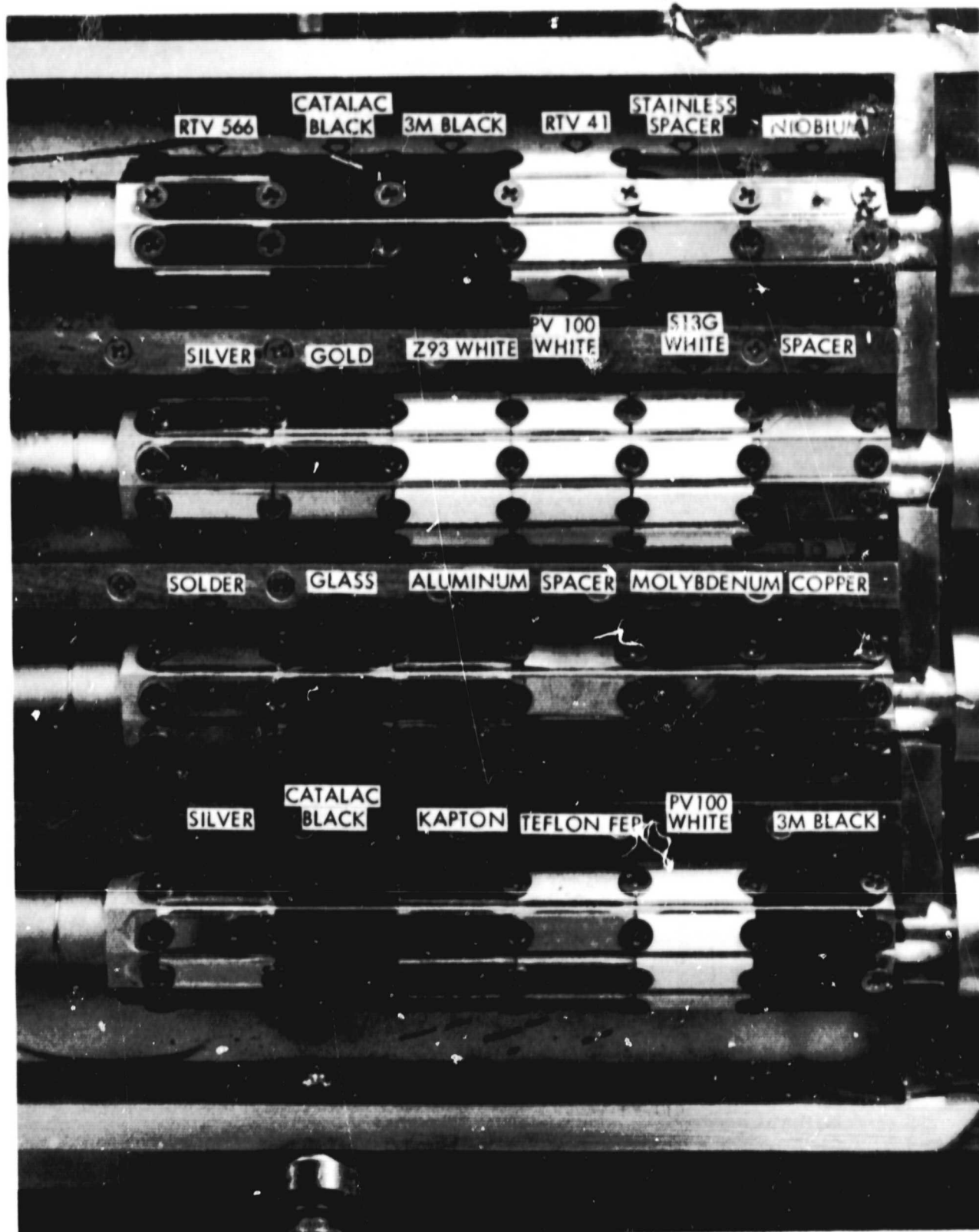


Figure 3. Loaded multipurpose sample holder prior to first exposure. Each set of 5 samples forms 75, 60, 45, 30, and 0° angles between their normals and the beam axis.

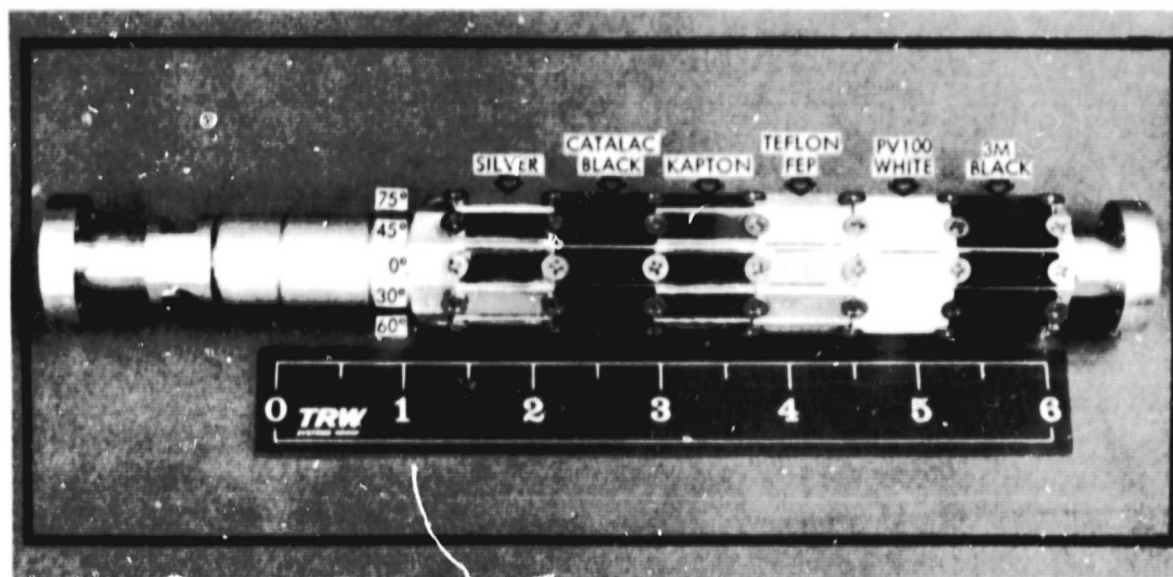


Figure 4. Closeup view of Pipe 4 before mounting in the holder.

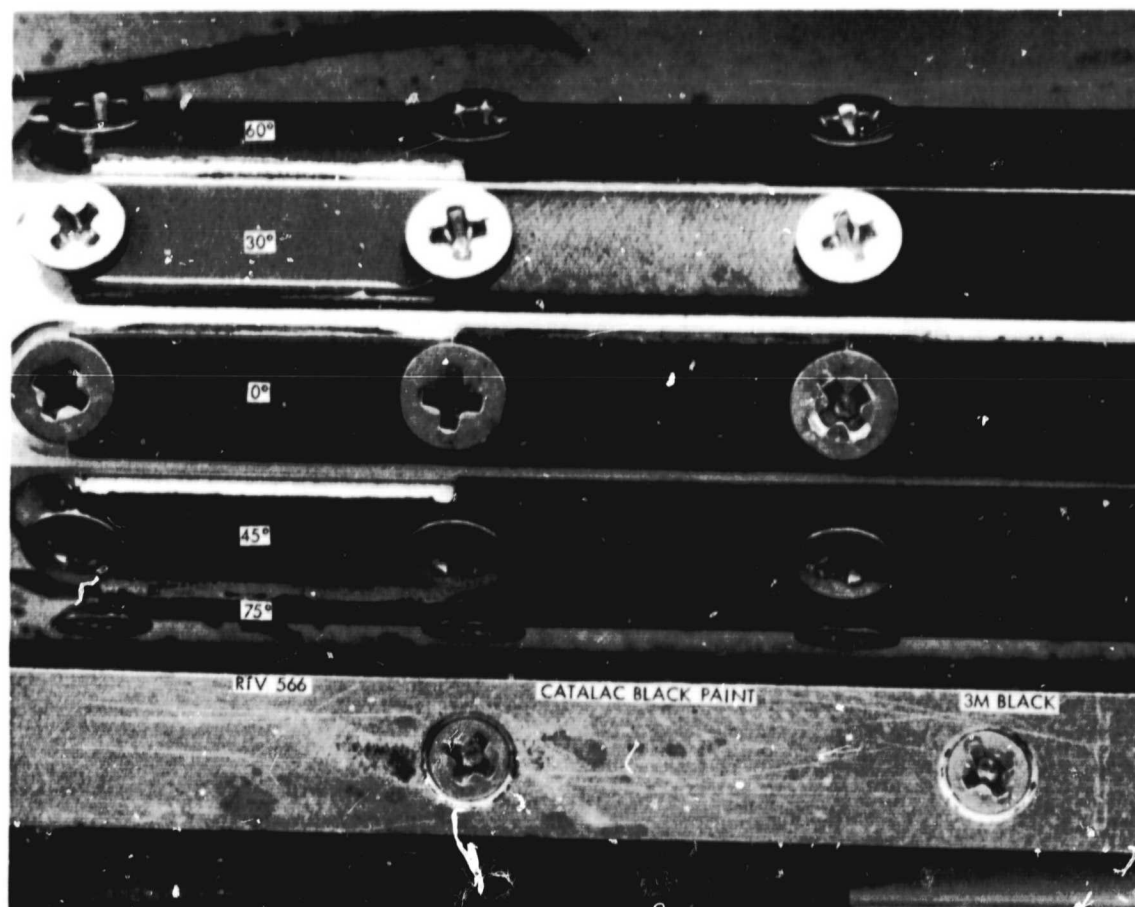


Figure 5. Closeup of Pipe 1 mounted in holder. The screw heads shield a portion of each sample from the beam to provide a fiducial surface.

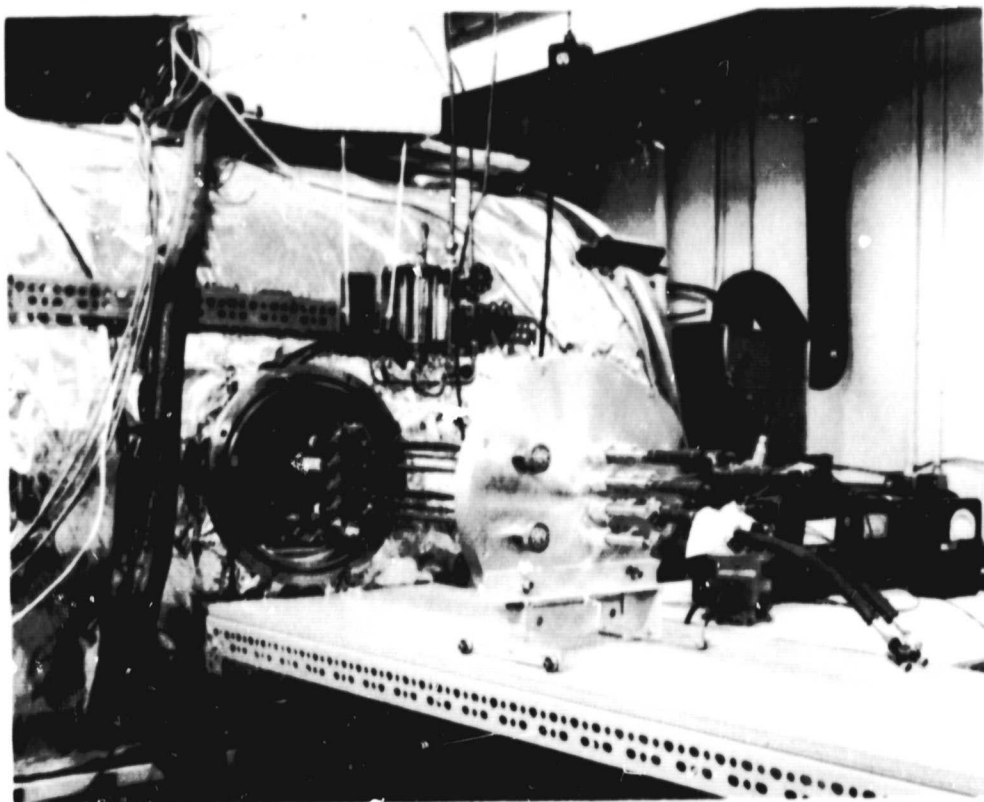


Figure 6. Insertion of the loaded multipurpose sample holder into the 4 x 8 foot vacuum facility prior to the first exposure.

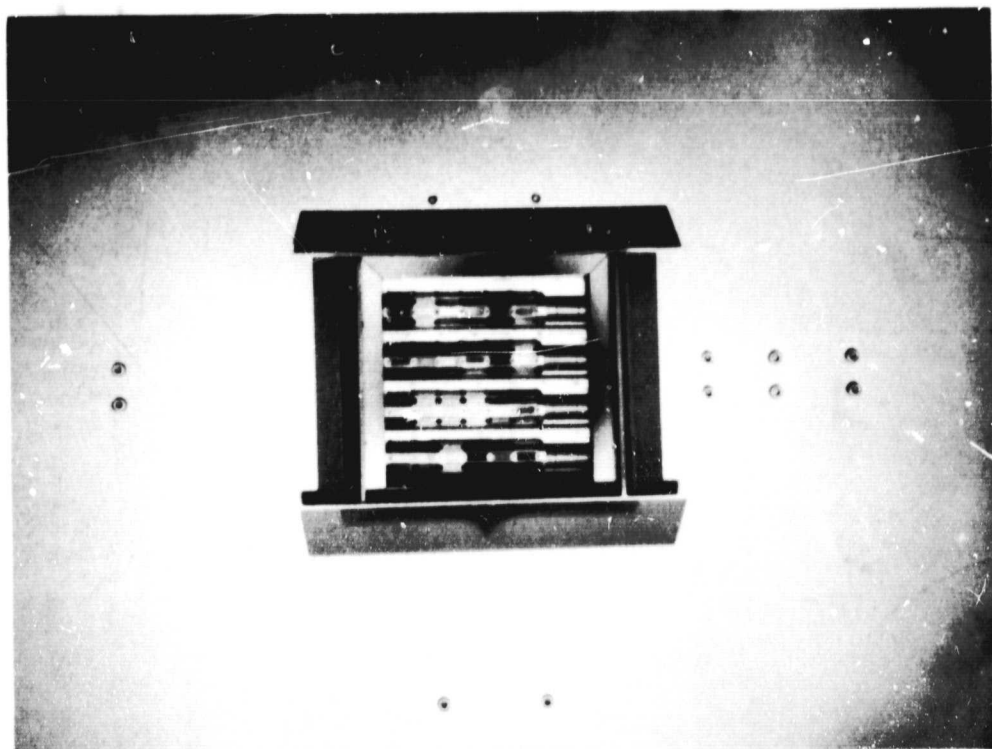


Figure 7. Sample holder in the exposure position as viewed from the ion beam source. Knife edged masks block the arrival of metal sputtered from vacuum chamber walls.

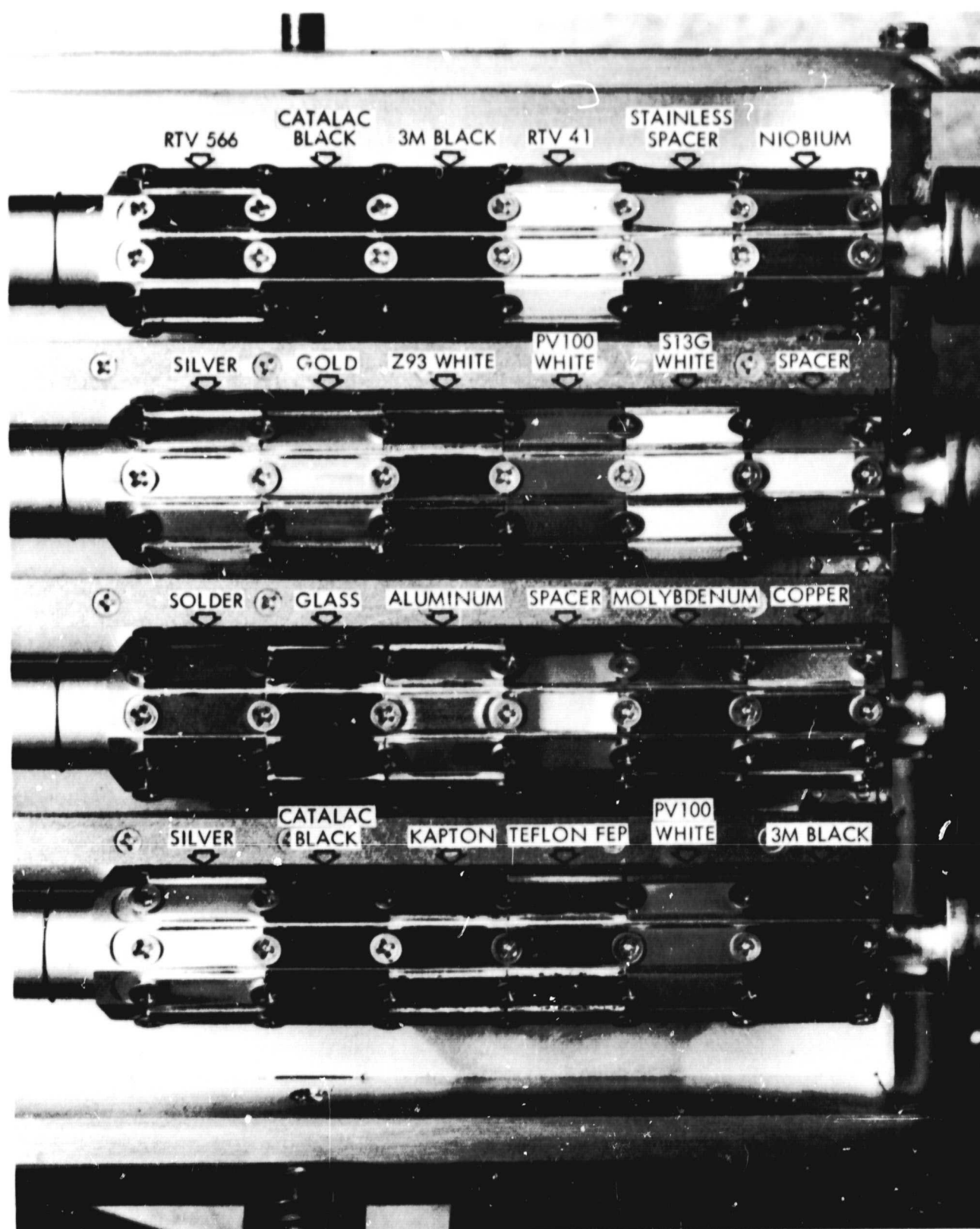


Figure 8. Multipurpose sample holder following 2 hours exposure to 3 KeV Hg^+ at approximately $1/4 \text{ ma/cm}^2$.

markedly reduced. Figure 9 is a closeup of Pipe 4 showing the Teflon samples. Actually, this black material is polyvinyl acetate, the adhesive used to cement the Teflon to the aluminum substrate. Before ion bombardment the adhesive is transparent, but, as later experiments proved, 3 KeV Hg^+ turns jet black. Microscopic examination of the 0°, 30°, and 45° samples showed a modest number of fine needles of Teflon projecting from the surface along the ion beam axis, some of which were 5-mils high.

A much greater density of needles was formed on the 75° degree sample. The larger diameter needles are visible in Figure 10 as bright lines. Also visible is the fiducial area of Teflon which was shadow shielded by the mounting screw. The reason that the beam-exposed regions are translucent in contrast to the transparent fiducial area, is found in the microstructure of the surface. Careful visual examination of the sputtered surface at approximately 100X reveals that it consists of densely packed, tiny needles.

The formation of straight needles or spikes on a surface by ion bombardment is not an unusual occurrence, and the growth process is believed to be understood. However the topography of the 60° Teflon sample was quite unusual. Its surface looked like wet sheep's wool; it had thickly packed, long, curved, very small diameter "hair". Figure 11 is a closeup of the sample. More will be said about this surface structure following additional sample exposures and chemical evaluation of the exposed samples.

The variation in degree of ion damage to the reflectance of Z93, PV100, and S13G white paints and the white RTV41 silicone rubber is graphically illustrated in Figure 12. It is seen that S13G is the least affected, next RTV 41, next PV100, and Z93 the most. This qualitative observation is consistent with the in situ quantitative measurements reported in Vol. I of the Final Report.

C. Future Work

During the forthcoming reporting period, exposures of approximately 20°C samples on the multipurpose sample holder to 3 KeV Hg^+ will continue.

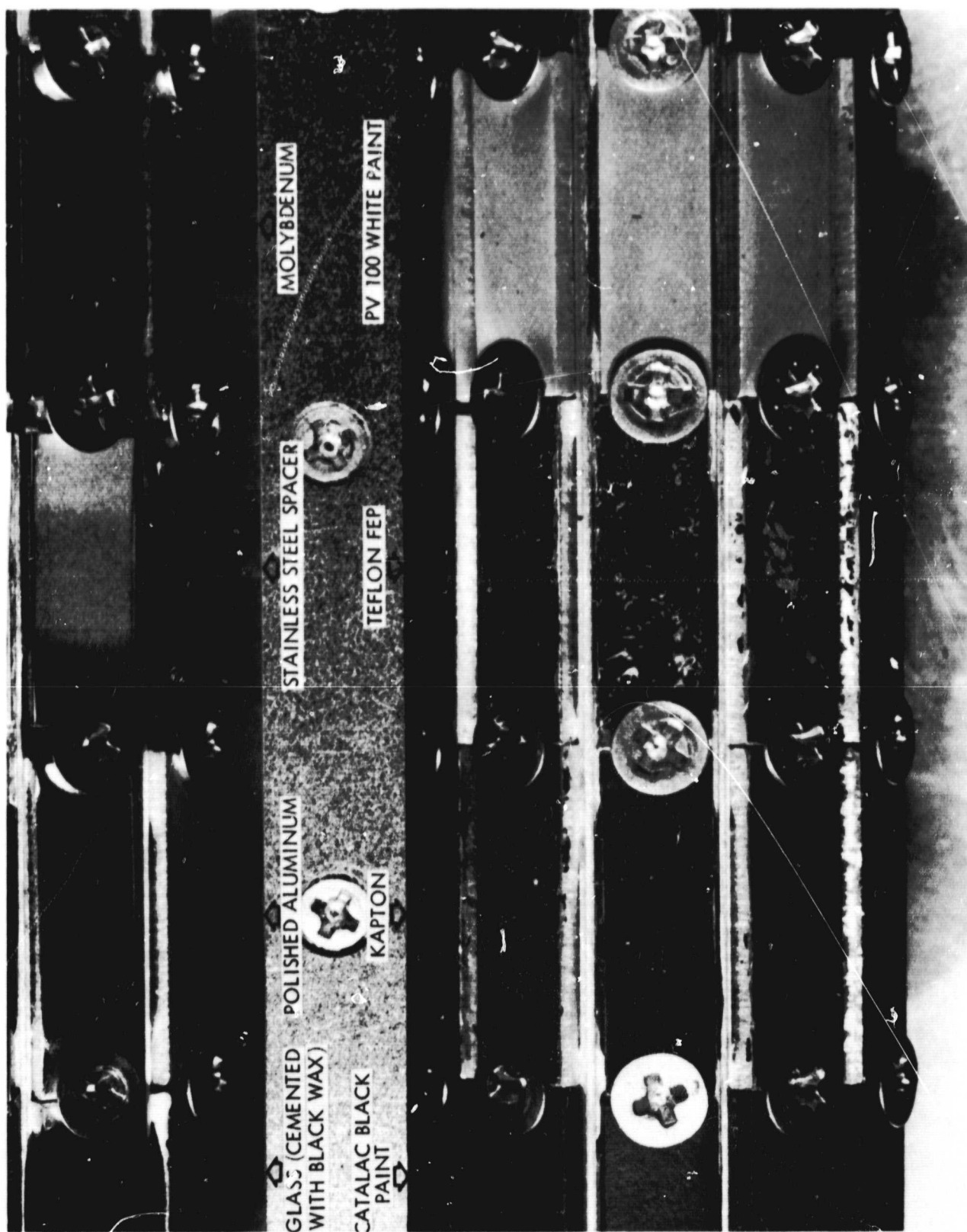


Figure 9. Closeup of Pipe 4 after first exposure showing completely eroded Teflon samples and PV100 white paint turned grey. Other visible samples, including Kapton, appear unaffected by the exposure.

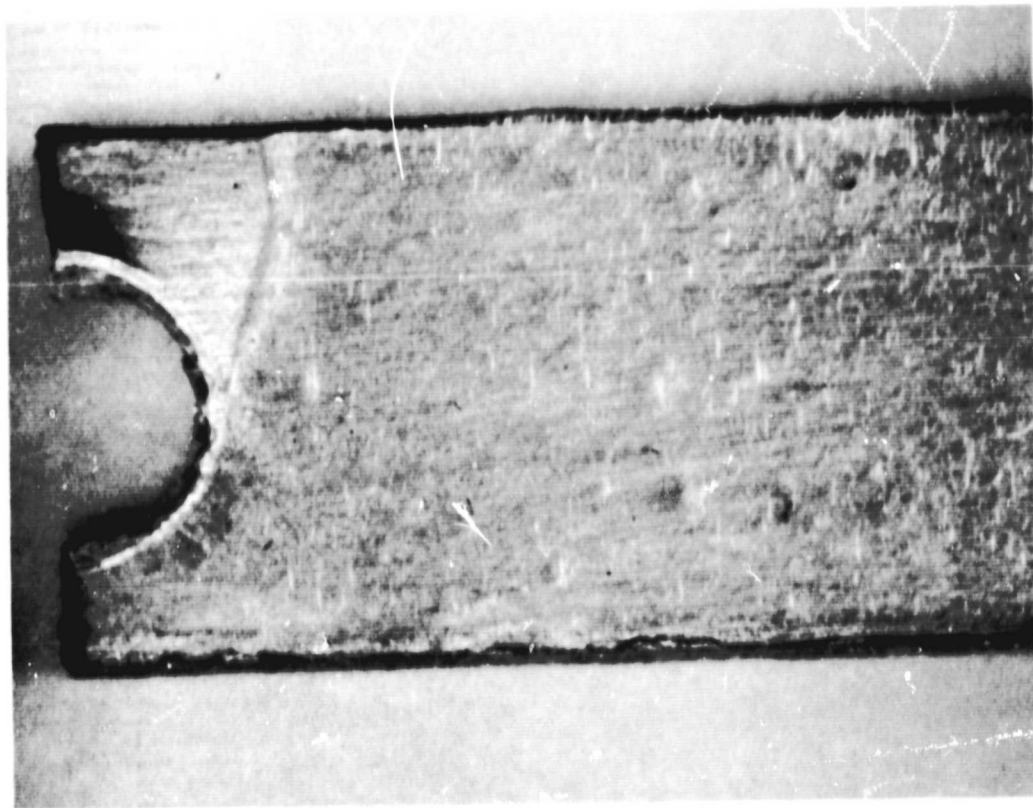


Figure 10. Closeup of 5 mil thick Teflon FEP sample cemented to aluminum substrate and exposed to Hg^+ with 75° between beam axis and sample normal. 10X.

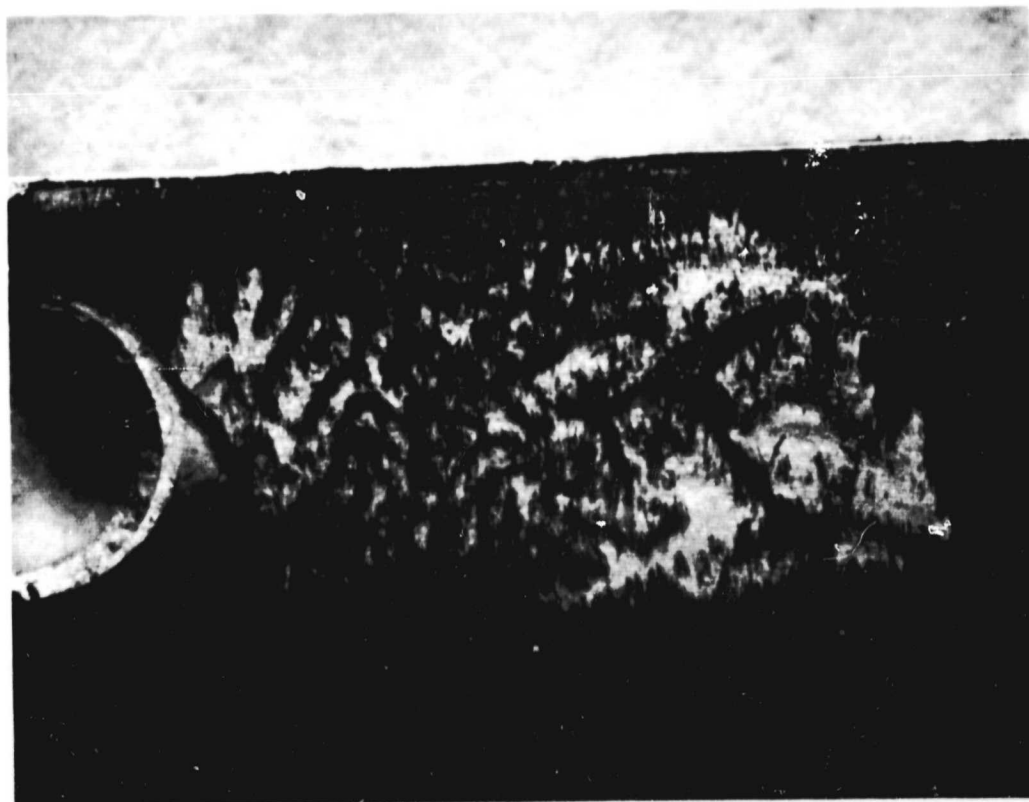


Figure 11. Closeup of 60° Teflon sample. 10X.

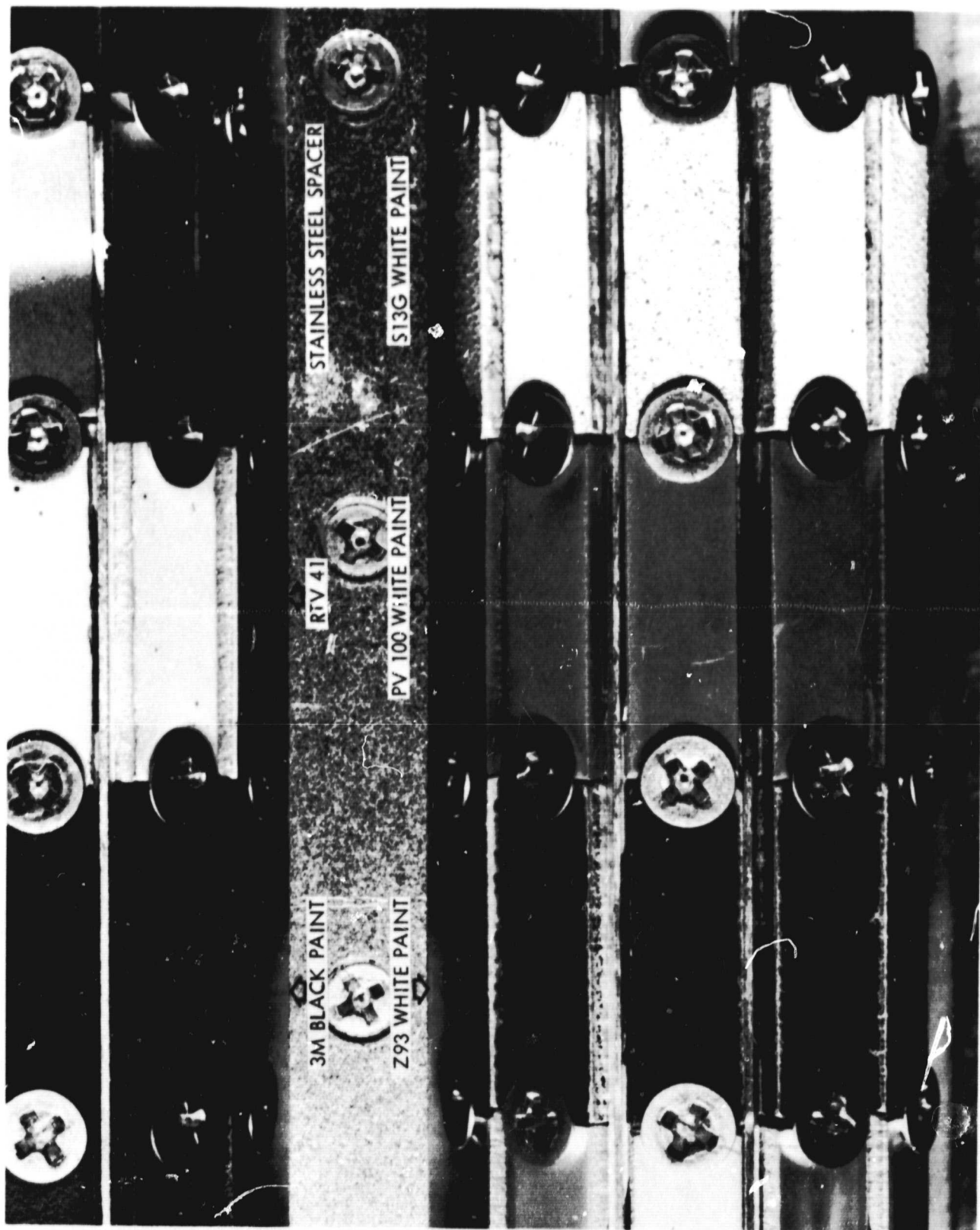


Figure 12. Closeup of white paint and RTV silicone rubber samples after ion beam exposure.

These exposures will be made in increments so that samples of varying sputtering yields and/or thicknesses may be removed from the holder and processed after appropriate exposure durations. Of course, this requires removing the sample holder from the vacuum chamber between exposure increments. This procedure will cause a thin (approximately 21 Å) oxide layer to form on the aluminum samples each time they are exposed to air, but the time required to sputter off this film will be infinitesimal compared with the duration of exposure. No other samples to be tested will form oxides at room temperatures.

IV. METALLURGY

In Volume I of the Final Report it was reported that the mechanical properties of solder were markedly affected by immersion and surface coating of samples with mercury. Since the solder "failed" these relatively severe tests, it was recommended that further tests be devised which 1) more closely simulated the conditions of exposure in space, and 2) tested, either directly or indirectly, the effect of mercury on the critical properties of solder in its most common spacecraft surface application, namely, in solar arrays. Exposure to atomic or ionic beam of mercury will satisfy the first mentioned requirement. A brief review of the uses and failure modes of solder in solar arrays was undertaken as the first step in satisfying the second requirement.

Soft solder is used extensively in bonding solar cells to solar cell interconnects. The most common cause of failure of these bonds on deployed solar arrays is differential thermal contraction between silicon and the metal interconnects during temperature cycles, particularly during cool down. However, it is not the practice of light weight solar array engineers to specify the required or desired strength of cell-to-interconnect solder bonds, apparently because stress levels are neither conveniently calculated nor measured on these tenuous structures.

Nevertheless, solder bond failures do occur, and our previous tests suggest that the mechanical properties of solder are sensitive to even small quantities of mercury. Therefore, a simple test of the effects of a mercury beam exposure to solder will be designed.

V. CHEMISTRY

During the quarter additional cesium immersion tests were performed in accordance with the recommendations in Volume I of the Final Report. The immersion of the paints were low-cost screening tests which permit informed selection of those materials deserving of more detailed study. The immersion of Kapton was to permit further study of its previously reported reaction with cesium.

A. Cesium Immersion of Thermal Control Coatings

Weighed samples of PV100, Z93, S13G, and 3M Velvet black coated on aluminum were each exposed to distilled Cs at room temperature for 48 hours. Each sample was quenched in a benzene - 10% methanol solution and washed with fresh methanol and then dried in vacuum. After drying each sample was reweighed and the surface was inspected for visible damage. Control samples were also given the quenching, washing, and drying treatments to determine their effect on sample weight.

The results are summarized in Table 10. With the exception of the 3M Velvet sample there was no visible damage. This sample shows some increase in reflectance (as determined visually) and small pits in one small area of the sample. Cesium did not wet the surface of the Z93 sample.

The large weight loss incurred by the S13G sample is very likely due to drying of the inorganic pigment. There was no evidence of surface darkening nor degradation of the coating itself.

During the quenching operation on the PV100 sample, the coating was lifted from the aluminum substrate. The same result was obtained when the control sample was subjected to the quenching operation, so the cesium exposure had nothing to do with this result. The samples of this coating were weighed separately and a weight loss based on coating weight alone was determined. This weight loss (associated with cesium exposure) is only 2.74% and is probably also due to drying of the coating pigment. However, the possibility of leaching of TiO_2 pigment* from the surface of the coating should not be discounted completely.

*As suggested in Chemical Effects Table II-2, Vol. I of the Final Report.

Table 10.
Cesium Exposure of Thermal Control Coatings

Sample	Initial Weight g.	Final Weight g.	Weight Change g.	% Change ¹	
S13G	0.2829	0.2718	-0.0111	-3.92	No visible surface damage
Control	0.1481	0.1478	-0.0003	-0.20	
Z93	0.1829	0.1802	-0.0027	-1.48	No surface damage; Cs did not wet surface
Control	0.1975	0.1971	-0.0004	-0.20	
3M Velvet	0.3906	0.3834	-0.0072	-1.84	Some whitening and small p's formed
Control	1.1804	1.1752	-0.0052	-0.44	
PV100	0.3684	0.3660	-0.0024	-0.65 ²	No visible surface damage. Sample lifted from substrate by quench solution.
Control	1.1968	1.1945	-0.0023	-0.19 ²	

1. Based on total weight of sample
2. % weight losses were 3.93% for the exposed sample and 1.19% for the control sample based on substrate-free coating weights of 0.0611g and 0.1928g, respectively.

The results obtained from the immersion of the S13G and Z93 samples in cesium indicates that darkening of the coatings is not to be expected and entries for these materials in the chemical effects tables may be changed to "no effect" due to cesium exposure.

An additional sample of the 3M Velvet black paint was exposed to metallic cesium at room temperature in the manner above. Quenching and handling was also the same. This time, however, sample weight prior to drying was also recorded. The sample was dried by maintaining its temperature at 100°C for one hour in vacuum.

<u>Sample</u>	<u>Initial Weight, g</u>	<u>Wt. Dried g</u>	<u>% Change</u>	<u>Wt. After Exposure, g</u>	<u>% Change</u>
3M Velvet	0.3130	0.3105	- 0.80%	0.3006	-3.19
Control	0.2934	0.2912	- 0.75%	0.2912	0

As observed in the previous exposure, the sample surface had changed color, becoming a mottled grey. No pitting was evident this time, however. A small amount of coating had flaked off of the substrate, possibly accounting for the increase in weight loss.

In view of these results, this material should be exposed to the cesium atom beam.

B. Cesium Immersion of Kapton

1. First series. Two sets of five 1" x 1" samples of 2 mil thick Kapton film were exposed to distilled Cs for 48 hours at room temperature and at 100°C, respectively. The test vessel of the room temperature samples was back-filled with dry helium for the reaction period, whereas the 100°C samples remained under vacuum.

The samples exposed at room temperature were removed and quenched in a helium filled glove bag. Because the samples were touching each other in the test vessel, only one sample was obtained which was completely purple. It was subsequently lost due to exposure to air or moisture after being

placed in a holding jar. Therefore, additional room temperature Kapton samples were exposed, as reported below.

The samples exposed at 100°C had an unexpected property. When the samples were removed from the cesium in the glove bag, they broke into small fragments. Further, several small detonations occurred while trying to handle these fragments with tweezers. Sample handling was suspended for safety reasons. Since the glove bag had been flushed with helium several times prior to opening the test vessels, it is believed that very little oxygen was present. Furthermore, by the time the 100°C sample vessels were opened, bulk cesium from the other samples was in an open container in the bag. The appearance of the cesium surface gave no indication of oxygen present, and of course it was acting as an oxygen "pump."

The three hypothesis listed below have been formulated to explain the detonations. However, no further effort has been expended on understanding this phenomenon.

- o Cesium formed a complex with the Kapton film which is much more sensitive to the small amount of O₂ remaining in the glove bag.
- o Cesium formed a complex with the film which is shock and/or friction sensitive.
- o Cesium degraded the film and formed organo-metallic compounds which are shock sensitive.

2. Second series. In view of the aforementioned difficulties, an additional set of Kapton samples were immersed in cesium. Five samples of 0.5 in x 1.5 in, 2-mil-thick Kapton were placed in separate tubes. Cesium was distilled onto each sample under vacuum and then each tube was back-filled with helium gas and capped. These samples were held at room temperature for 48 hours and then each was quenched in a benzene-5% methanol bath after initially cleansing in benzene. During the quenching operation, two of the five samples were partially destroyed when they caught fire inside of the helium filled glove bag. They were not used. Each sample was then dried in vacuum and sealed in an evacuated tube.

The previously postulated one electron oxidation-reduction reaction would result in a reaction product with increased electrical conductivity. As a test of this hypothesis, one of the vacuum ampules was transported to Dr. A. Rembaum of the Jet Propulsion Laboratory, a specialist in the measurement of the electrical properties of organic materials. He measured the film's resistivity in a nitrogen filled dry box. It was 10^{13} ohm-cm, as compared with the normal Kapton resistivity of 10^{18} ohm-cm.

This 10^5 increase in electrical conductivity is believed to be conservative, since quenching the film in the methanol and benzene solution immediately following its removal from the cesium very likely depleted the surface of the film of current carriers. Thus, the possibility exists that the resistivity before quenching could have been one to several orders of magnitude lower.

Another of the exposed samples was used in a measurement of electron spin resonance (ESR) spectrum. An extremely strong signal was measured at 3325 gauss and 9.36 GHz, with no associated fine structure. Two possible sources of such a signal are organic free radicals and trapped electrons.

Both the resistivity and ESR measurements support the hypothesis that the deep purple-red reaction product is formed by one electron oxidation-reduction, or a kind of solid state solvation of the electron from cesium.

C. Future Work

During the forthcoming reporting period post exposure analysis of samples from the Hg^+ beam exposures is expected to begin.

VI. CONCLUSIONS

A. Thermophysics

Theoretical calculations of the optical properties of thin films indicate that transmission in the solar region is markedly reduced by 100 Å or less thickness of aluminum, molybdenum, and niobium. This thickness on a solar cell will also have a marked effect on its infrared emittance but not its solar absorptance, so cell power output will be reduced by increased temperature as well as reduced light. White painted surfaces will also increase in temperature. Unfortunately, it is for thicknesses less than 100 Å that the assumptions of the theory are least likely to be correct. Further analysis is indicated.

B. Ion Bombardment

Teflon FEP has a very high erosion rate in comparison to metals and other spacecraft surface organics tested.

C. Metallurgy

A test of the effects of mercury on soft solder which is pertinent to the strength of a silicon-interconnect bond is needed.

D. Chemistry

Immersion of S13G, Z93, and PV100 white paints in cesium for 48 hours at room temperature had little effect; they probably need not be tested further. 3M Velvet black became mottled grey and should receive additional attention. The electrical conductivity of cesium immersed Kapton is increased approximately 10^5 over unexposed Kapton. This and the ESR spectrum support the one electron oxidation-reduction hypothesis. Kapton immersed at 100°C fragmented and detonated during handling in a glove bag, suggesting that additional study of the effects of cesium on Kapton might be fruitful.

VII. RECOMMENDATIONS

Recommendations for future work are contained in the several technical sections.

V. NEW TECHNOLOGY

None.