NASA Technical Memorandum 58246

Oxygen Atom Reaction with Shuttle Materials at Orbital Altitudes

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CONTENTS

TABLE

FIGURES

SUMMARY

Surfaces of materials used in the Space Shuttle Orbiter payload bay and exposed during STS-1 through STS-3 were examined after flight. Paints and polymers, in particular Kapton used on the television camera thermal blanket, showed significant change. Generally, the change was a loss of surface gloss on the polymer with apparent aging on the paint surfaces. The Kapton surfaces showed the greatest change, and postflight analyses showed mass loss of 4.8 percent on STS-2 and 35 percent on STS-3 for most heavily affected surfaces. Strong shadow patterns were evident. The greatest mass loss was measured on surfaces which were exposed to solar radiation in conjunction with exposure in the vehicle velocity vector.

A mechanism which involves the interaction of atomic oxygen with organic polymer surfaces is proposed. Atomic oxygen is the major ambient species at low orbital altitudes and presents a flux of $\sim 8 \times 10^{14}$ atoms/cm^2-sec for reaction. Correlation of the expected mass loss based on ground-based oxygen atom/polymer reaction rates shows lower mass loss of the Kapton than measured. Consideration of solar heating effects on reaction rates as well as the high oxygen atom energy due to the Orbiter's orbital velocity brings the predicted and measured mass loss in surprisingly good agreement. Flight-sample surface morphology comparison with ground-based Kapton/oxygen atom exposures provides additional support for the oxygen interaction mechanism.

INTRODUCTION

Space Shuttle flights STS-1 through STS-3 have already provided unique information which ranges from the performance of spacecraft structures to the definition of the environment in the spacecraft vicinity. Another unique opportunity presented by Space Shuttle is the return of materials from orbital exposure free of extraneous effects generated by exposure to reentry heating conditions so that space environment effects can be assessed. In fact, all materials exposed in the Shuttle payload bay fall in this category, and examination of many of these exposed surfaces shows significant effects.

Materials have been returned for evaluation of space exposure effects in the past. Samples of thermal control points were exposed on the exterior of the command module on Apollo 9 (ref. 1), and Surveyor III parts were returned from the lunar surface on Apollo 12 (ref. 2). Examination of both sets of samples for surface effects presented problems in that the Apollo 9 samples were obscured by contamination enhanced by the reaction control engines and the Surveyor surfaces had been affected by lunar dust kicked up by the lunar module's landing. Additionally, samples of thermal control surfaces were exposed on Skylab and returned for analyses (ref. 3). Unfortunately, these samples were also heavily contaminated by outgassing from Skylab systems. Payload bay materials which were exposed during STS-1 through STS-3, therefore, represent the first return of a significant amount of materials exposed in low Earth orbit which can be examined for space effects.

Payload bay materials with significant exposed surfaces consist of Teflon-coated Beta fabric and Kapton (both used on thermal control blankets), S13G-LO and A-276 white paints and A-971 yellow paint (used as thermal control and identification paint), silver/Teflon radiator panels, and various structural materials such as grapnite/epoxy and metals. Surfaces of these materials were visually examined for orbital exposure effects after each of the three Shuttle flights. Significant changes were noticed on the Kapton, the A-276 white paint, and the A-971 yellow paint.

Kapton film used as a portion of the thermal blankets on the Shuttle payload bay television cameras exhibited a change from a glossy to a flat, light-yellow appearance. Strong shadow patterns associated with the surface appearance were noticed. After initially confusing this effect as a contaminant, it was subsequently shown to be due to surface recession of the film surface. A mechanism is proposed which involves the interaction of the Kapton film with ambient neutral oxygen atoms at the Shuttle orbital altitudes. Interaction of oxygen atoms with other organic materials will also be considered such as the apparent rapid "aging" of the white and yellow paints.

Several people assisted in providing samples of the Kapton blanket and making the necessary measurements. Ragan Edmiston provided the blanket samples, and Thomas Ballentine and Steve Jacobs made mass loss and optical property measurements on the samples. Scanning electron microscope photographs were provided by Gail Horiuchi, David McKay, and Drew Issacs. Horst Ehlers provided consultation regarding the impact of the oxygen atom reaction on other systems.

In compliance with the NASA's publication policy, the original units of measure have been converted to the equivalent value in the Système International d'Unités (SI). As an aid to the reader, the SI units are written first and the original units are written parenthetically thereafter.

FLIGHT EFFECTS

Payload Bay Materials Description

A brief description of the location and type of material exposed in the payload bay is provided as a basis for understanding shadowing effects and exposure conditions. By far, the largest exposed surface is presented by the Teflon-coated Beta fabric which is used to cover all thermal blankets in the bay except for the television cameras as shown in figure 1. This fabric is a plain weave of Beta fibers overcoated with TFE Teflon, which is, therefore, the exposed surface. The next largest surface is the silver/Teflon used on the radiator panels. Television camera blanket surface material is 0.00084 centimeter (0.00033 inch) Kapton film aluminized on the back side and reinforced with a fabric backing. The location of the film as well as camera nomenclature are shown in figure 1. Exposed metal surfaces in the payload bay are painted with either S13G-LO or A-276 white paint, and extravehicular handrails are painted yellow with A-971. Areas of exposed graphite epoxy are also shown in figure 1. A considerable number of other organic materials are exposed in the payload bay; however, these will not be specifically considered in this paper.

Postflight Examination Approach

Shortly after the payload bay door opening following flight completion, all payload bay surfaces were visually examined for gross effects. Samples of surfaces which showed an exposure effect were obtained for laboratory analysis. Several samples of Kapton blanket were obtained as well as samples of residue from the painted surfaces. Scanning electron microscopy proved to be the most useful analytical tool, since except for the initial contamination studies, surface morphology was the most revealing aspect of the exposure effect.

Description of Surfaces Affected

STS-1.- Visual examination of the materials in the payload bay after STS-1 generally revealed no unusual conditions except for the Kapton blanket surfaces of camera A. (See fig. 1.) Surfaces viewing in the same direction as the camera lens (fig. 2) showed a loss of film gloss and a slight color change from the Kapton gold color to a milky yellow. Surfaces on the other sides of the blanket exhibited little change. The surface change was rather subtle but easily noticeable after being pointed out. This surface effect was initially thought to be related to contamination; therefore, samples of the blanket Kapton layer were removed for analyses.

Two other cameras with Kapton blankets were used on this flight: one in position C and one in position D. Neither of these cameras was affected.

It was also noticed after the STS-1 flight that the yellow paint used on handrails inside the bay appeared to be aging very rapidly since yellow pigment could be removed by simply light wiping.

STS-2.- Six cameras were used on STS-2 and STS-3 as shown in figure 1. All of their blankets exhibited some surface appearance change as after the STS-1 flight. The cameras used on the remote manipulator system (RMS) elbow and wrist showed the most change. The back of the wrist camera as shown in figure 3 was heavily affected. This surface generally was perpendicular to the vehicle main axis (+X axis) during the flight, and viewing forward.

The elbow camera blanket was heavily affected on the forward-facing side and the back side as shown in figure 4. A strong shadow pattern was evident on the forward-facing side. It appeared to be associated with the support yoke position.

Blankets on cameras A and C were affected on the lens side. Other surfaces on these cameras and on cameras B and D showed minor to no observable effects.

It should be pointed out that the blanket surfaces viewing in the same direction as the lens showed changes, whereas the lens surfaces were not affected.

Aging of the paint on the handrails was again noticed after STS-2 as well as the A-276 white paint. No surfaces of S13G-LO could be easily reached for examination. Generally, affected paint surfaces lost their glossy appearance similar to Kapton; however, no change in color was noticed.

STS-3.- Essentially all blanket surfaces were heavily affected on STS-3 except for areas protected from the environment by brackets and other pieces of hardware. The surface condition of camera C as shown in figure 5 represents the effects seen on all cameras. Surfaces which had a good view in the +Z axis direction (upward out of the bay) were most affected. Painted surfaces showed progressive degradation.

SURFACE ANALYSES

Exposed Kapton surfaces were the most sensitive for definition of the effect. For this reason, most of the analyses were conducted on these surfaces and the bulk of the remaining discussion will center around this material. Analyses on other materials will be included as necessary.

Samples of affected Kapton from all three flights were obtained for further laboratory examinations. These samples consisted of the blanket from camera A for STS-1, from the RMS elbow camera for STS-2, and the blanket from camera C for STS-3. In addition, samples of the easily removable surface residue from the white and yellow paint were taken by the tape-lift method for analysis.

The initial interpretation that the Kapton was contaminated resulted in a fairly thorough chemical analysis of the surfaces involved. After surface effect (contamination layer) removal attempts with solvents proved unsuccessful, three surface analysis techniques were used. These techniques consisted of multiple internal reflectance infrared spectroscopy (MIR), scanning electron microscopy (SEM), and electron spectroscopy for chemical analysis (ESCA).

These techniques showed no significant surface contaminant. Only the substrate Kapton could be detected by MIR. Both SEM and ESCA showed slight enrichment of silicon in the affected region as compared to unaffected regions. This silicon increase is probably due to increase in surface exposure area rather than any indication of contamination.

Surface Morphology

Examination of the involved surfaces for morphological changes were the most useful in understanding the effect on the film. High-magnification examination showed that the affected surface was much more porous than the unaffected region. This characteristic is shown in figure 6. Stereographic SEM photographs at similar magnification of the two regions (affected and unaffected) showed rather clearly that the affected region was lower than the unaffected region and, therefore, Kapton film loss was indicated. Similar effects were found on STS-3 affected surfaces.

Mass Loss Determination

Mass change and thickness measurements were made to quantify the mass loss occurring. Thickness measurements made using an optical microscope indicated a minor thickness reduction in the affected region. This thickness change was too small to be reliably measured using the optical technique; therefore, mass loss measurements on the film after removal of adhesive, support film, and aluminum coating were made and yielded good results. Using controls from unaffected areas of the blanket, a mass loss of 4.8 percent was detected on the elbow camera blanket which was exposed on STS-2. A significantly affected area on the side as shown in figure 4 was used for this mass loss determination. Although a limited sample area of the blanket was available, mass loss measurements showed good agreement among the three samples selected (4.3 to 4.7 percent).

Mass loss measurements were made on the blanket for camera C which was exposed only on STS-3. Similar sampling procedures as used on STS-2 samples showed a mass loss of 35 percent for the STS-3 samples. As in STS-2, a heavily affected area was selected for evaluation; however, this type of change represented approximately 30 to 50 percent of all the blanket surface used on the STS-3 television cameras.

Optical Property Measurements

Since such a significant amount of Kapton surface was lost during flight, optical property measurements were made on several areas of the elbow camera from STS-2 and several areas on camera C from STS-3. An average change in solar absorptance α_s of 0.05 was observed on STS-2 and a change in α_s of 0.12 was observed for the STS-3 samples. An emittance ε change of 0.1 was measured for the STS-3 material.

PROPOSED ENVIRONMENT/SURFACE INTERACTION MECHANISM

All of the surface effects discussed to this point are explained well by atomic oxygen surface attack of organic materials. Average atomic oxygen concentration at 260 kilometers (STS-2 orbital altitude) is $1 \times 10^9/cm^3$.[1] Materials used on the Shuttle Orbiter, therefore, are exposed to an oxygen atom flux of 8.6×10^{14} atoms/cm^2-sec when these surfaces are in ram condition; i.e., the material surface is normal to and facing into the velocity vector of the spacecraft. This oxygen atom flux is significant and can cause loss of organic material by oxidation. Solar heating of surfaces exposed to the atomic oxygen beam, of course, results in increased reaction rates.

An approximate assessment of reaction rates of oxygen atoms with organic materials at Shuttle orbital altitudes can be made from data obtained by Hansen et al. (ref. 4) in a low-temperature asher. Organic-based samples are ashed in an oxygen atom environment produced by radiofrequency excitation of molecular oxygen. Mass loss rates for 36 organic materials were determined in that study. One was a polyimide-base sample which is used for assessment of the Kapton (polyimide) mass loss rates.

Oxygen atom concentrations in the Hansen work were not quantitatively measured but were estimated to be in the range of 10^{14} to 10^{15} atoms/cm^3. For a sample temperature of less than 343 K (70° C), mass loss rate for the polyimide sample was 4×10^{-7} g/cm^2-sec. Assuming that the oxygen atom/surface reaction rate in the Hansen experiment was not limited by oxygen concentration, then the rate is dependent upon oxygen atom surface collisions. The collision frequency C_f can be determined from the following (ref. 5) given the gas pressure P and temperature T.

$$C_f = \frac{3.5 \times 10^{22} P}{(mT)^{1/2}}$$

The total collision rate as calculated for the Hansen condition of 133.3 N/m^2 (1 torr) pressure is 5×10^{20}/cm^2-sec and the collision rate for oxygen atoms

[1] Private communication with George Carignan, University of Michigan, based on data from the MSIS model as developed by Hedin et al.

is $1.6 \times 10^{18}/cm^2$-sec if an atom concentration of $1 \times 10^{14}/cm^3$ is assumed. The reaction yield is 2.5×10^{-25} g/atom for the conditions assumed and the associated polyimide mass loss rate.

The reaction yield as derived from Hansen's data can be applied to the Shuttle conditions to estimate the Kapton mass loss rate $\dot{m}_K$.

$$\dot{m}_K = (8.6 \times 10^{14} \text{ atoms/cm}^2\text{-sec}) (2.6 \times 10^{-25} \text{ g/atom})$$

$$= 2.2 \times 10^{-10} \text{ g/cm}^2\text{-sec}$$

Using this rate, 66 hours (or 660 hours if the oxygen atom concentration was $10^{15}/cm^3$) of ram exposure time at 260 kilometers would be required to produce the 4.8 percent mass loss as measured on STS-2.

The exposure time for the STS-2 blanket (4.8 percent mass loss) is difficult to establish because of the variation of velocity vector and Sun position with respect to the vehicle. The estimated rate does seem to be too low, however, since the total orbital time on STS-2 was approximately 48 hours. The total exposure time in the velocity vector and solar condition for STS-3 (top Sun attitude) is estimated to be 5 hours.

Two other factors are important in determining actual reaction rates at orbital conditions. One factor is the polyimide temperature. If the samples in the Hansen work were considerably below the 343 K (70° C), then the reaction rate is too low to be used for the STS-2 blanket, since solar exposure produces temperatures as high as 343 K (70° C) in the blanket Kapton layer. Although the activation energies for oxygen atom reaction with organic materials are generally very low (~21 kJ/mole (~5 kcal/mole), for example see ref. 6), a temperature difference of 40 K (40° C) between the flight Kapton temperature and Hansen's polyimide sample temperature would yield approximately a factor of 2.6 increased rate for the Kapton or a required time of 25 to 200 hours. On STS-3, Kapton temperatures with an α/ε as computed from previously measured properties may have resulted in a difference in temperature of 70 K (70° C), which results in a rate increase of 6. The required exposure time for STS-3 surfaces is 11 hours for the lower oxygen atom concentrations. This exposure time is in good agreement with the estimated 5-hour STS-3 exposure. Reaction rate increases due to the thermal effect undoubtedly lead to the strong shadow patterns seen on STS-2. (See fig. 4.)

The other factor which must be considered is the reaction rate increase due to the high-energy oxygen atoms to which the Shuttle materials are exposed. For the velocity associated with an orbital altitude of 260 kilometers, the effective oxygen atom energy is 5 electronvolts. This additional energy (above thermal energies) should lead to higher reaction rates.

Considering all differences in exposure conditions for the Hansen study and orbital case, the agreement is very good and significantly supports the oxygen atom interaction proposal.

LABORATORY SIMULATION OF SURFACE MORPHOLOGY

Since, at this time, some uncertainties exist concerning activation energies, effect of oxygen atom velocity, actual materials data, etc., laboratory studies are being conducted to simulate the effect. Several problems arise however in trying to simulate the Shuttle materials effect in laboratory conditions. The largest shortcoming is not being able to simulate the oxygen atom energy associated with orbital conditions which results, as mentioned earlier, in lower reaction rates. Laboratory radiofrequency excitation experiments must of necessity be conducted at pressures in the order of 133.3 N/m^2 (1 torr), considerably greater than the orbital exposure. This increased pressure may have some effect on the removal of oxidation products, if not on the mechanism itself. It also is not possible to simulate very well the thermal conditions associated with solar exposure. Despite these shortcomings, laboratory experiments were conducted to simulate the surface morphology changes associated with oxygen atom attack.

Simulation experiments were conducted using an SPI Supplies Plasma Prep II low-temperature asher. This device produces oxygen atoms from a radiofrequency excitation of molecular oxygen and was operated at approximately 100 watts at 133.3 N/m^2 (1 torr) total pressure. Under these conditions, minimal sample heating (<313 K ($<40^\circ$ C)) occurred and reaction rates were determined for Kapton, Teflon TFE and FEP, and Mylar.

The Kapton and Mylar samples showed etching effects (loss of glossy surface) very similar to those obtained on the Shuttle materials after short exposure (<20 minutes) to the asher conditions. Mass loss rates measured under these conditions are shown in table I and generally are in good agreement with the Hansen values. A portion of Hansen's data is included for comparison of relative reactivity of a large number of organic polymers.

Samples of Kapton as etched in the low-temperature asher were examined by SEM. Surface morphology of these samples is similar to that of the orbitally exposed samples. As seen in figure 7, the surface appears very porous for the Kapton. Surfaces from the STS-2 samples have received a considerable amount of handling damage prior to SEM examination. This handling damage was simulated in the laboratory-exposed sample by contacting the affected surface with a thin film and rubbing the thin-film surface. As can be seen, the effect generated results in a surface more similar to the STS-2 surfaces.

The laboratory data as obtained in a low-temperature asher, although very limited in scope, also support the oxygen atom attack mechanism proposed. Additional studies are planned to define reaction rates as well as products of reaction as a function of temperature and pressure. This should lead to a better understanding of the orbital effect and should provide at least a relative ranking of reaction rates of various materials.

POTENTIAL IMPACTS OF ORBITAL OXYGEN REACTION

Optical Properties

Short-term orbital exposure will lead to changes in optical properties for very thin metalized films. For example, the Shuttle thermal control blanket may have to be covered with a more stable material such as Teflon-coated Beta in order to provide the necessary life. Only minor changes in optical properties should occur for thicker films except for changes in reflectivity. As observed, the organic surfaces become more diffuse with exposure time.

Long-term effects on thermal control and other surfaces are dependent upon material-peculiar reaction rate and exposure conditions. It is anticipated, however, that some materials may have limited life. Because of limitations in ground-based simulations, life determination will most likely have to be evaluated by flight experiments.

Outgassing Rate Increases

Material outgassing rates are, of course, affected by oxygen atom oxidation. For ram exposure, especially in conjunction with solar exposure, the outgassing rates may be limited by the oxygen reaction after initial ordinary material outgassing has stabilized. Outgassing rates for Kapton as preliminarily determined from STS-2 and STS-3 data may be as high as 10^{-8} to 10^{-9} g/cm^2-sec during exposure to the above conditions. Such rates may interfere with experimental measurements if not properly considered.

Increased outgassing rates of organics due to the oxygen effect, in fact, is one possible explanation for measurements of relatively large amounts of methane using the Induced Environment Contamination Monitor (IECM) mass spectrometer on STS-2 and STS-3. Preliminary laboratory experiments indicate that reaction of oxygen atoms with room temperature vulcanizer (RTV) 630 (similar to the RTV-602 used in the IECM thermal control paint near the mass spectrometer entrance) silicone produces hydrogen, carbon monoxide, and methane. These reaction products could be scattered into the mass spectrometer by ambient gases. Other sources for the methane measured by the IECM are being investigated.

Contamination-Related Effects

Contamination effects other than increased outgassing rates are possible; for example, cleaning of contaminated surfaces as pointed out by Gillette et al. This effect is generally beneficial but may affect measurements of contaminant deposition rates under certain conditions. Removal of silicone-based surface contaminants is not possible (ref. 7) and, indeed, may lead to silica-enriched contaminants which cannot be removed by heating. This effect may indeed explain the high deposition rates seen on the Skylab quartz crystal microbalance sensors. This effect could very easily be misinterpreted as radiation fixing of the contaminant.

Oxidation of surfaces will also produce pigment-rich surfaces in case of paints and a porous surface on some polymer films as pointed out earlier. Release of these pigment or polymer particles could occur because of vehicle vibrations or even by sputtering from ambient gases. Detailed flight measurement will have to be made to determine whether particle production and release due to this mechanism is important.

CONCLUDING REMARKS

Significant surface effects have been observed on organic materials exposed in the Shuttle Orbiter payload bay on the first three orbital flights. These effects are associated with removal of surface matter as measured from mass loss determinations on Kapton films used as a component of Orbiter thermal blankets. A mechanism for this Kapton mass loss is proposed and involves the reaction of oxygen atoms available at low Earth orbital altitudes with organic polymers. Shadow patterns seen on some surfaces indicate accelerated reaction rates due to solar exposures. Although additional studies are necessary to confirm the oxygen attack mechanism, it is presented at this point for consideration of such an effect on other programs.

Lyndon B. Johnson Space Center
National Aeronautics and Space Administration
Houston, Texas, April 27, 1982
953-36-00-00-72

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TABLE I.- MASS LOSS RATES IN ATOMIC OXYGEN

Type of polymer	Rate, g/cm^2-sec	
	Ref. 4	This report
Polypropylene	12×10^{-7}	-
Chlorinated high-density polyethylene	17	
Polytetrafluoroethylene	2.1	1.0×10^{-7}
Polyimide	4.1	4.6
Polycarbonate	8.8	
Polyethylene terephthalate	6.2	
Nylon 610	11.2	
Formaldehyde polymers	19.8 to 27.0	
Polyester (Mylar)		8.0
Poly(tetrafluoroethylene co-hexafluoropropylene)		1.1

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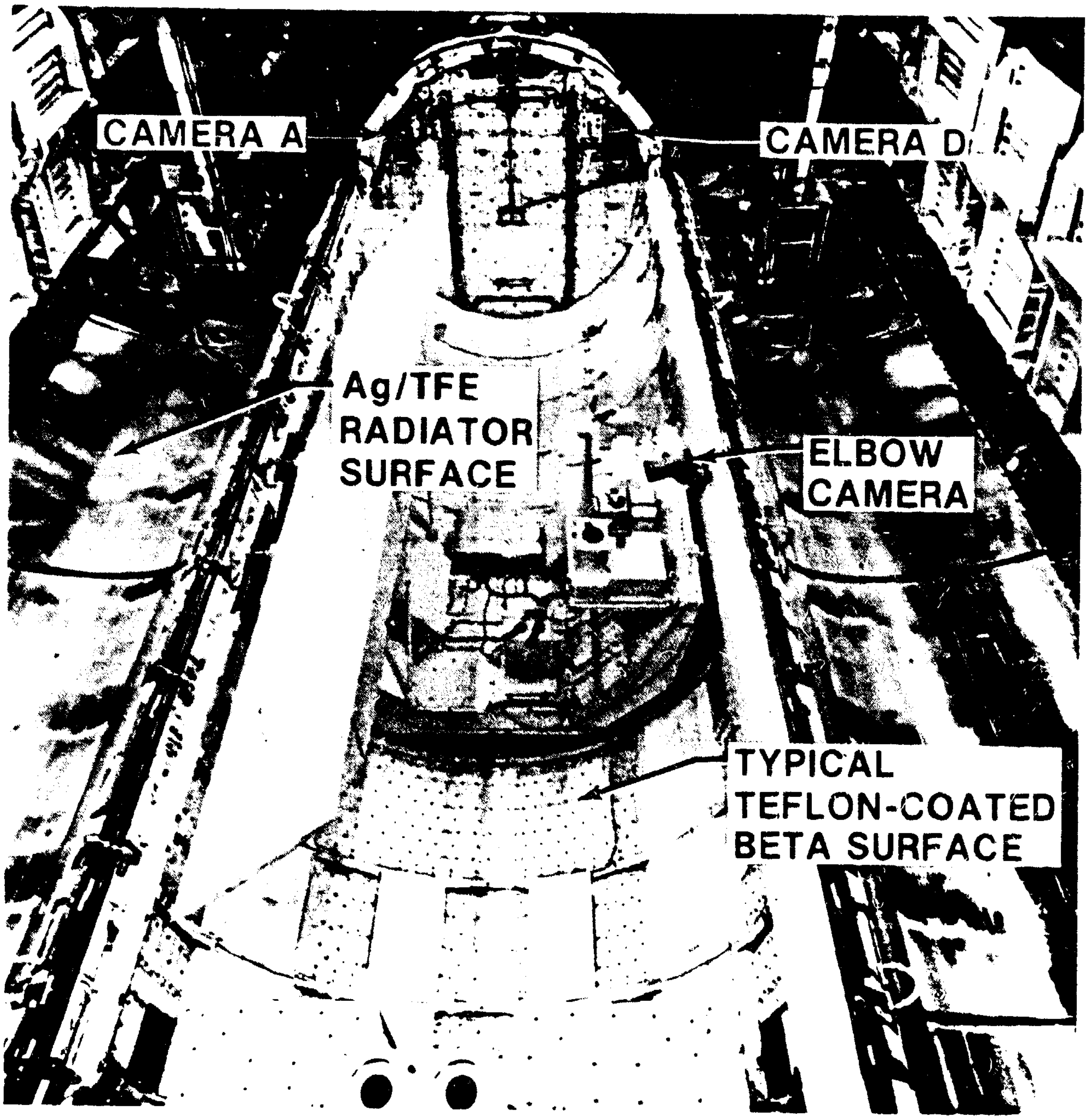
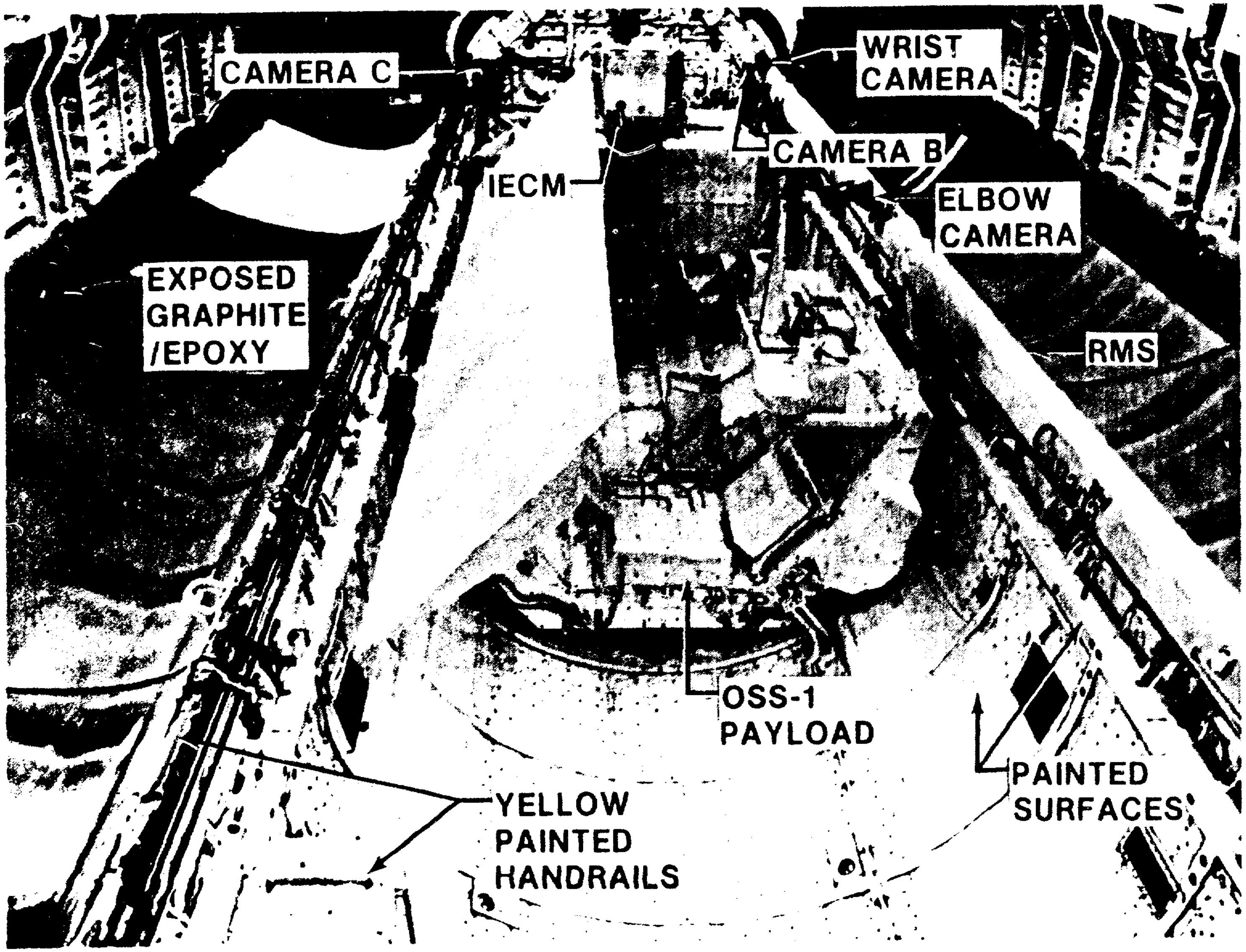


(a) View looking forward.

Figure 1.- Payload bay.



(b) View looking aft.

Figure 1.- Concluded.

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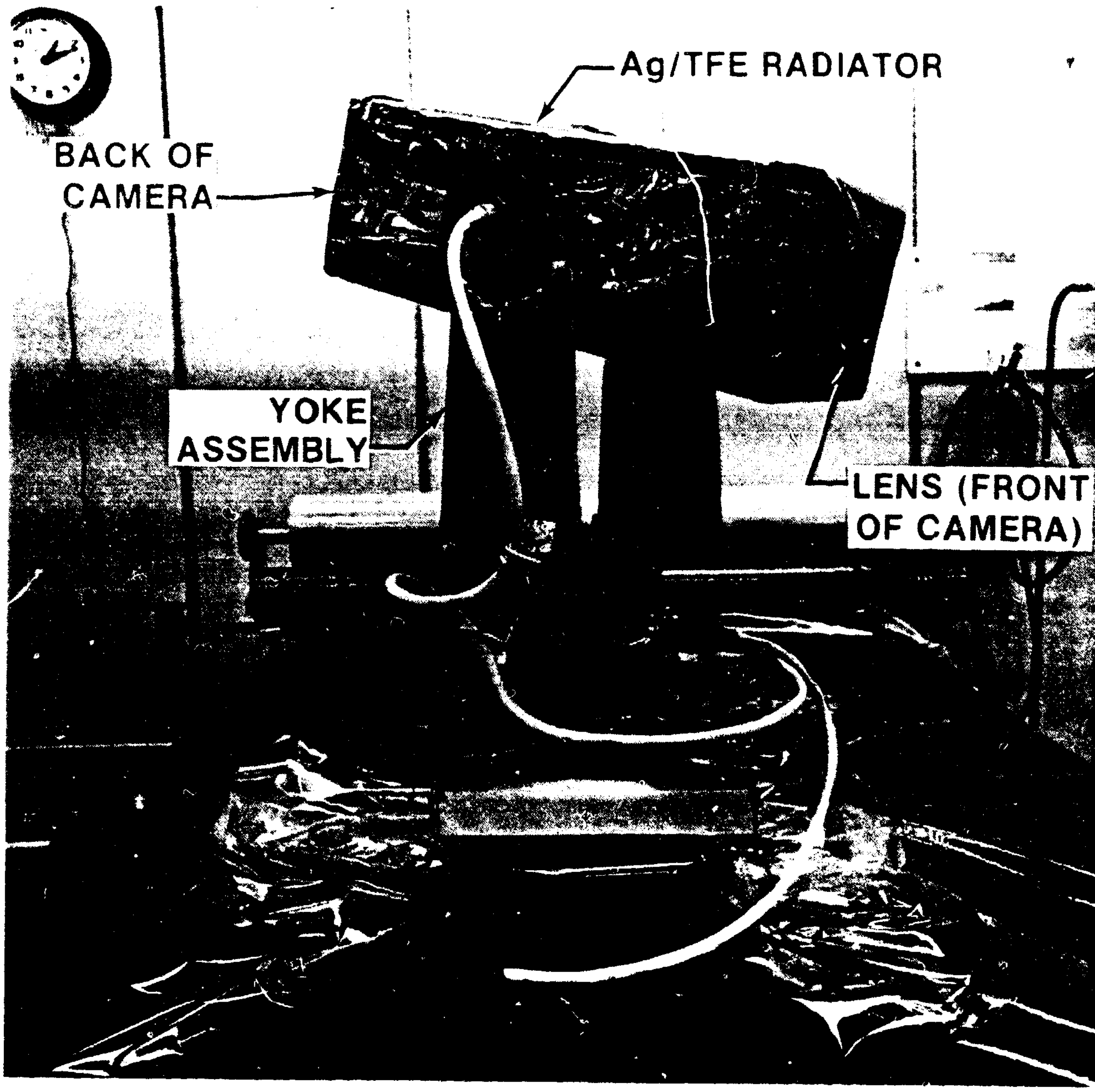

Figure 2.- General view of camera configuration.

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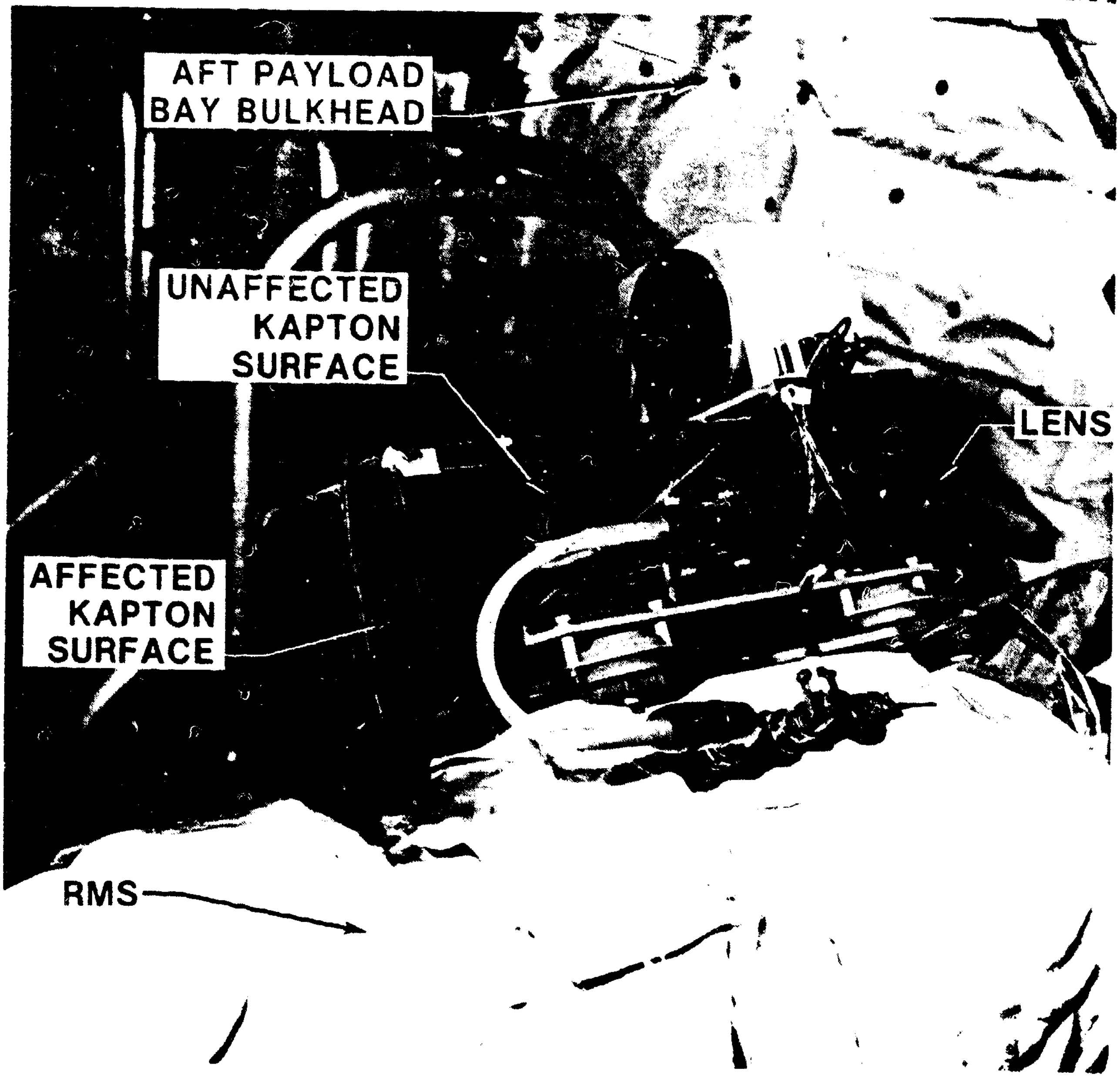

Figure 3.- RMS wrist camera after STS-2 flight.

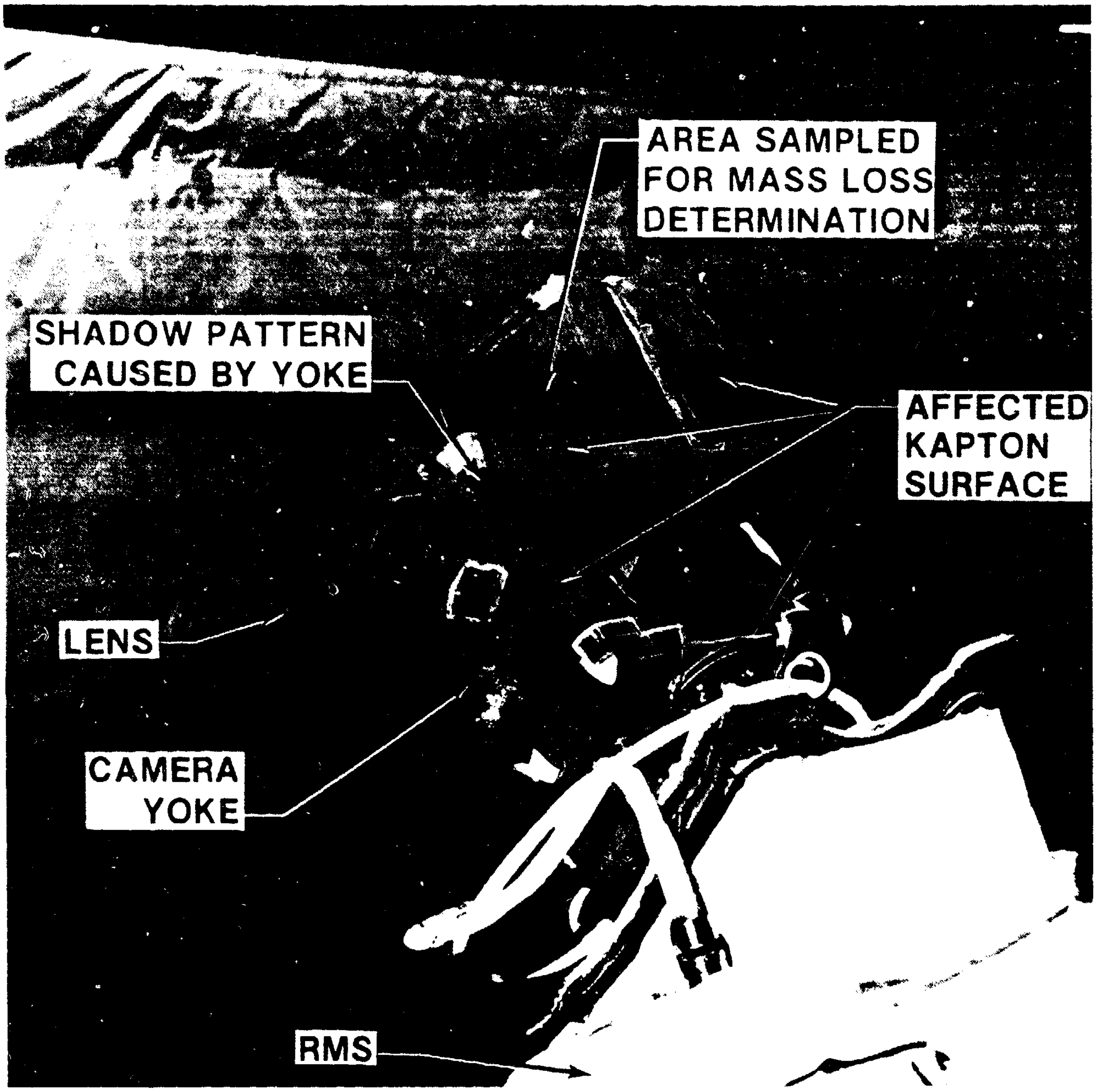

Figure 4.- RMS elbow camera after STS-2 flight.

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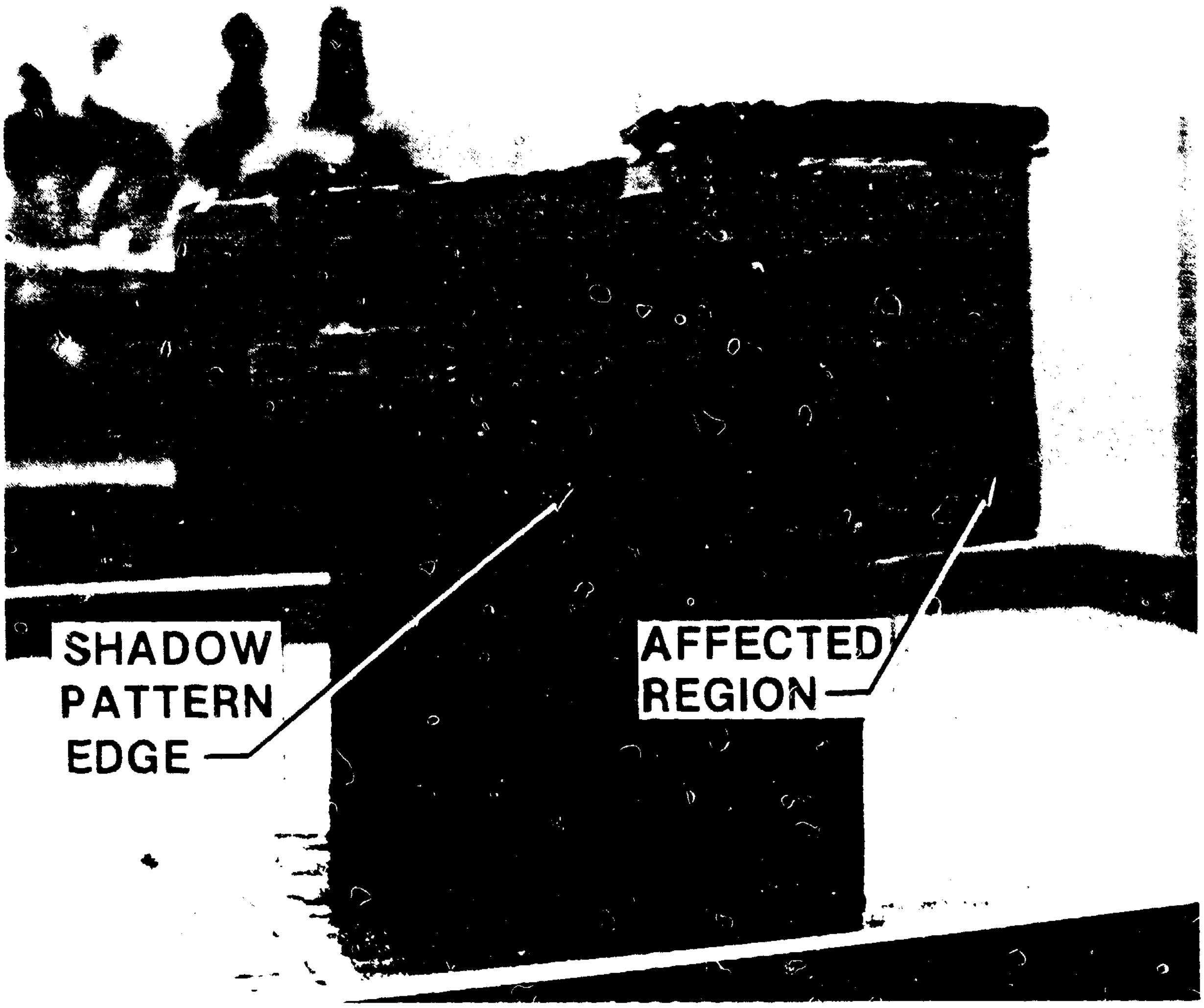

Figure 5.- Camera C after STS-3 flight.

UNEXPOSED
KAPTON SURFACE

STS-2
EXPOSED SURFACE

STS-3
EXPOSED SURFACE

Figure 6.- SEM photographs of flight-exposed Kapton surfaces.

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ASHER
EXPOSED SURFACE

ASHER EXPOSED
SURFACE WITH SOME
HANDLING DAMAGE

Figure 7.- SEM photographs of low-temperature-asher Kapton surfaces.

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