19951174 91

N95-23911

WHISKER/CONE GROWTH ON THE THERMAL CONTROL SURFACES EXPERIMENT #S0069

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SUMMARY

An unusual surface "growth" was found during scanning electron microscope (SEM) investigations of the Thermal Control Surface Experiment (TCSE) S0069 front thermal cover. This "growth" is similar to the cone type whisker growth phenomena as studied by G. K. Wehner (Univ. of Minn.) beginning in the 1960's. Extensive analysis has identified the most probable composition of the whiskers to be a silicate type glass. Sources of the growth material are outgassing products from the experiment and orbital atomic oxygen, which occurs naturally at the orbital altitudes of the LDEF mission in the form of neutral atomic oxygen. The highly ordered symmetry and directionality of the whiskers are attributed to the long term (5.8 year) stable flight orientation of the LDEF.

INTRODUCTION

During scanning electron microscope (SEM) investigations of the front thermal control cover on the LDEF Thermal Control Surface Experiment (TCSE) S0069 an interesting "growth" was discovered on its exposed Teflon surface. The authors of the paper were utilizing a field emission scanning electron microscope to investigate the atomic oxygen (AO) damage to the silver Teflon coating as a function of AO incident angle. This "growth" has lead to a great deal of interest within the LDEF material investigator community and by various news media sources[1,2,3,4], of which some publications were of a rather dubious accuracy and intent[5,6,7].

EXPERIMENT DESCRIPTION

Objective

The overall objective of the TCSE on the LDEF was to determine the effects of the combined near-Earth orbital environment including the LDEF induced environment on space-

craft thermal control surfaces. In flight optical measurements were performed to evaluate optical changes in thermal control coatings as a function of time on orbit. This unique data also enabled the development of lifetime prediction models for these materials.

Mission Summary

On April 7, 1984 the LDEF was placed in a 463 km (250 N miles), 28.5 degree inclination low Earth orbit by the Space Shuttle *Challenger*. After 5 years 10 months in space, on January 12, 1990, the LDEF was retrieved by Space Shuttle *Columbia* at an altitude of 330 km (178 N miles).

Figure 1 is a schematic of the LDEF in orbit. The LDEF was a gravity-gradient stabilized and mass loaded so that one end of the spacecraft always pointed at the Earth and one side always pointed into the velocity vector or RAM direction. Spacecraft deployment was as planned with the TCSE located on the leading edge (row 9) of the LDEF and at the Earth end of this row (position A9). Actual LDEF orientation was slightly offset from this planned orientation. As shown in Figure 2, the LDEF was rotated about the long axis where row 9 was offset from the RAM direction by about 8 degrees[8,9]. This LDEF/TCSE orientation resulted in total exposures of 8×10^{21} atoms/cm^2 atomic oxygen[10], 1×10^4 ESH solar ultraviolet[11], 3.3×10^4 thermal cycles[12], and 3×10^5 rads of particulate radiation[13].

Hardware Description

The TCSE was the first experiment to measure the optical properties of thermal control surfaces in space in the same way they are routinely measured in the laboratory[14]. The configuration of the experimental system consisted of a completely self-contained experiment package providing its own power, data system, reflectometer, and preprogrammed controller for automatically exposing, monitoring, and measuring the sample materials.

The primary in-space measurement was provided by a total hemispherical reflectometer recording total reflectance as a function of wavelength at 100 wavelength points from 250 to 2500 nm. The reflectometer design consisted of two light sources, tungsten and deuterium lamps, used with a scanning prism monochrometer with selectable slit widths to provide the monochromatic energy for the spectral measurement. The 115 mm (4.5 inch) diameter integrating sphere collected both the specular and diffuse reflected monochromatic light from a wall mounted sample to provide the angularly integrated measurement capability. Kodak barium sulfate ($BaSO_4$) was used for the sphere coating. Reflected energy was measured with a UV enhanced silicon photodiode detector and a lead sulfide detector.

FRONT THERMAL CONTROL COVER

Summary of Previous Results

Optical Properties of Front Thermal Control Cover

The front thermal control cover has a Sheldahl 0.05 mm (0.002 inch) thick Ag/FEP thermal control material applied with 3M 966 acrylic adhesive to an aluminum substrate of 6061 alloy, 1.6 mm (0.063 inch) thick. The Ag/FEP is composed of an outer Teflon support

layer, a silver high reflective layer deposited on the backside of the Teflon, an inconel protective layer deposited on the silver, and 3M 966 acrylic pressure sensitive adhesive. The silver layer provides the high visible reflectance, while the Teflon layer provides a high far-infrared emittance value, providing an excellent surface for minimizing average surface temperatures during orbital sunlight/shadow cycles.

Covered areas that were protected from AO and solar ultraviolet radiation have no apparent damage and are still highly specular[15]. Areas exposed to the space environment are clearly delineated and have a diffuse, whitish appearance with brown discoloration. This brownish discoloration varies from light brown to dark brown. Changes in Ag/FEP visual appearance are the result of two damage mechanisms: AO erosion and internal damage associated with cracking of the silver/inconel layer during application[15].

Exposed regions on the front cover without the brownish discoloration experienced only small increases from the original solar absorptance value from 0.08 to 0.10. The worse case brownish area had a solar absorptance as high as 0.49. Thermal emittance was unchanged in the covered areas whereas the exposed regions degraded from 0.68 to 0.48. Change in emittance resulted from loss of Teflon by AO erosion [15,16].

Atomic Oxygen Erosion Effects

AO erosion of the exposed Ag/FEP surface, sample S1 in Figure 3, is typical of that observed on other LDEF experiments[17,18]. Erosion of the exposed Teflon surface creates a nonuniform etching pattern . This results in a roughened surface which scatters incident light in a manner similar to a sandblasted piece of glass, providing an overall diffuse whitish color. SEM photographs of the Teflon surface on either side of the whisker/cone growth region are also shown in Figure 3. The covered region of sample T51 appears to show contamination deposits or layers. Apparent AO erosion on sample T51 in the exposed region near the growth shows a radical variation in the normal Teflon erosion surface morphology. All three images are at approximately the same magnification, yet erosion near the growth area is composed of roughly parallel ridges. These ridges are parallel to the local overlapping covers. Close inspection of the sample T51 image for the exposed region near the growth reveals what appears to be growth on the top of the central ridges, as would be expected if the ridges provide the nucleation site for growth initiation. Crystal or dendritic growth always initiates (all other parameters being equal) at surface defect sites or on sharp pointed protrusions.

Brownish Streaking Effect

Optical microscope investigations of the silver Teflon covered area showed that the silver/inconel layer is cracked[15]. When the Ag/FEP material is stressed, the silver/inconel layer cracks. Uniform parallel cracks are formed during normal application. A shattering type crack pattern is formed when the Ag/FEP is bent around protrusions or when air bubbles are squeezed out.

An overall surface contamination was not found on the brownish discolored areas; only very localized surface contamination occurred along overlapping panels and vents. The brownish discoloration observed is actually a result of the silver/inconel cracks. The closer grouped cracks give the appearance of a long dark smeared brownish streak when viewed without magnification. The overall brownish streaking on the front cover is the result of a series of events, starting with the initial cracking of the silver/inconel layer during application to the TCSE front thermal control cover. Subsequent long-term exposure to thermal cycling and solar ultraviolet caused the brownish discoloration of the acrylic adhesive, exposed by the silver/inconel cracks to the solar UV transmitting through the Teflon. The intensity of the

brownish discoloration is a direct function of the crack density which was caused by what we now know to be inappropriate application and handling techniques.

Location of Growth

Figures 4 and 5 show the location on the front thermal control cover where the whisker/cone growth was found. The arrow in Figure 4 points at a gap between two of the front thermal control covers. Within this gap a dark contamination deposit is apparent on the Teflon surface in Figure 5 on which the growth was located. This dark deposit, in contrast to the brownish streaks, was found to be a surface contamination deposit.

All regions on the front cover having overlaps had a very narrow contamination deposit within their gaps. This deposit was ~3 mm in width by ~50 mm in length. Other growth areas may exist on the front panel, but this is the only one found. In fact, a front cover sample section immediately below the growth region was provided to Dr. Stuckey/The Aerospace Corp. for analysis but no growth was found on the contamination deposit on this sample.

WHISKER/CONE GROWTH CHARACTERISTICS

The growth region is oriented parallel to the front thermal panel gap shown in Figure 4. SEM images in Figure 6 show that the growth is well ordered, oriented, directional, localized and varies in whisker concentration across the growth region. The growth surface shows no indication of surface facets. There are several stages of growth apparent in the SEM pictures. The base of the growth is a thin brittle dark layer on the Teflon which can be easily removed. In places the base layer has separated from the Teflon substrate. Individual whisker/cones are translucent when viewed with a visible wavelength optical microscope.

There are two major growth orientations: one is normal to the surface aligned with the LDEF major axis; the other is parallel to the surface facing inward on one side and outward on the other side. The overall growth pattern appears to have some of the characteristics of a dendritic type growth with nucleation occurring along defect sites. The growth dimensions are on the order of 7 microns height and a fraction of a micron in diameter. A few growth units are larger, many are smaller. Individual growth units have a hollow tube down their center and have an inverted cone morphology. The growth surface does not appear eroded by the space environment, including atomic oxygen exposure.

ANALYSIS

Biological Viability Testing Results

Analysis performed to date has indicated that the growth is not a standard fungus or mold type growth or contamination that could have occurred on the ground after flight.

Biological testing results were negative in that the unknown growth material did not respond to culturing on a nutrient agar. In addition, the acridine orange direct count epi fluorescence tests, which stain DNA to determine if the growth material is biological, were also negative. These tests were repeated for two different samples, one of which had not been exposed to the SEM vacuum and electron beam irradiation. Results were negative in both cases.

Electron Microprobe Elemental Analysis

Elemental analysis was performed on three areas of sample T-51. The "interior area" was shielded from the exterior LDEF environment. The "growth area" was located in a gap between the front panel covers (Figure 4) which formed a vent path from the experiment interior and was partly shielded from atomic oxygen and solar ultraviolet. The so called "no growth" area was exposed to the full space environment during the LDEF mission. Results of these analyses are shown in Figures 7, 8, and 9.

EDAX data for the interior location defined as the "unexposed/covered no growth region" on Figure 7a shows the presence of carbon (C), oxygen (O), fluorine (F), and silicon (Si). Although sulfur (S) is identified on the scan, it is very weak and may be questionable.

In comparison, the EDAX data for the "no growth" exposed region on Figure 7b shows strong peaks for fluorine (F), and very weak peaks for silicon (Si) and no sulfur (S). A small hint of carbon (C) can be seen on scan, although it is not labeled. The small silicon peak may be from residual growth or contamination not obvious during the initial viewing of the SEM images. This data is consistent with a Teflon surface eroded by atomic oxygen.

EDAX data for the growth region was based on focusing the SEM beam on a grouping of the inverted cone whiskers. EDAX scans for this region are shown in Figure 8a and b from two different instruments. A very strong silicon (Si) peak is detected, along with sulfur (S), oxygen (O), fluorine (F), and magnesium (Mg), when using the Hitachi S-4000. Notice that the fluorine (F) is greatly reduced from the "no growth" regions, and is probably background from the Teflon substrate. Also, notice that the carbon peak virtually disappears on exposed as compared to the unexposed regions. This same sample was run in the Cambridge 250 Mark II, having a higher energy analysis capability in order to confirm the magnesium and sulfur peaks. Surprisingly the magnesium peak turned out to be arsenic (As). Both peaks for arsenic are present as shown in Figure 8b. Therefore, the peak in Figure 8a identified as Mg is most likely As.

In order to determine if the fluorine peak in Figure 8a was from the growth or was background scatter from the Teflon substrate, another series of EDAX scans were made, carefully focusing on the thickest growth area. Results of these scans are shown in Figure 9a. The fluorine (F) peak is now totally eliminated, proving that fluorine is not a typical component of the growth and the previous fluorine peak was from background scatter. Silicon (Si) is still the main peak and assumed to be the main component. Sulfur (S) shows as a clean peak, indicating its presence. Oxygen (O) still shows but is weak. Interestingly, carbon (C) now shows up, but is also very weak. The arsenic (As) peak also is present, but is very weak.

Another set of scans were taken of the underside of the base material layer on which the growth is located. This layer is the darker material located at the base of the inverted cones as can be seen in Figure 6. On sample T51, some of the growth was disturbed while taking samples for the biological tests, thereby exposing the underside of some of the base material. Figure 9b is the result of a series of EDAX scans of this material. Most of the previously identified elements remain at the same ratio, except that the sulfur (S) peak is greatly reduced and the fluorine (F) peak now returns. This data indicates that some fluorine (F) is incorporated in the base material but is not incorporated in the whisker/cones. Also, it appears that sulfur was principally incorporated into the whisker/cone growth during the growth process and not from the base material.

FTIR; Total Attenuated Microprobe Analysis

Infrared analysis of this phenomenon has been very complicated because of the complex chemistry across the sample surface and the very small size of the dendritic growth. The problem of size has been alleviated through the use of an infrared microprobe system, but the complex chemistry across the sample still remains. For the purposes of this analysis, the scope will be limited to the Teflon substrate and the whisker/cone growth material.

Molecular microanalysis utilizing a scanning infrared microprobe microscope was performed by Nicolet Instrument Corporation in Stamford, Connecticut. Figure 10 shows the infrared spectra for Teflon flight control, Teflon flight sample exposed with no growth, and the whisker/cone growth (labeled dendritic growth). Teflon's characteristic absorption bands can be seen for both flight and control samples. The infrared spectra for the whisker/cone growth show almost no structure. The large absorption band at 1057 cm^{-1} indicates a strong presence of Si-O-Si. The small absorption bands at 3601, 3628, 3705, and 3732 cm^{-1} indicate a weak presence of Si-OH. This data indicates that the whisker/cone growth is primarily a SiOx glass type material.

In addition to the infrared spectra, contact with the probe crystal during surface probing indicates that the whisker/cones are hard and brittle in comparison to the surrounding fluorocarbon polymer. Again, this is consistent with a glassy or silicate type material.

CONTAMINATION SOURCES

Sulfur

During post flight investigation, when the S0069 cover was removed, very odiferous fumes were detected. Gas samples were taken using organic (activate charcoal) vapor monitors located inside the S0069 TCSE instrument with the shipping cover in place. Three monitors were located inside the instrument and one control was placed outside. Analysis identified the gas as dimethyldisulfide. Batteries were then removed and double bagged. Gas samples were taken from the bag using a vacuum bottle technique and analyzed at MSFC. As before, the gas was identified as dimethyldisulfide.

Figure 11 is a mosaic of photographs showing the returned flight batteries in the S0069 experiment tray (four batteries were used; one is hidden under the carousel). One of the batteries is shown with and without its lid in place. Individual cells are potted in the battery case, as can be seen in Figure 11. For these batteries it was found through investigations at MSFC* that after approximately three years (even in cold storage) the individual nickel cell safety pressure release would rupture. Figure 11 shows an individual cell with the pressure release ruptured. Dimethyldisulfide gas is vented by these cells when they rupture. In conjunction with the cell rupture, the battery case seal leaked when the o-rings (ethylene propylene) failed from compression set. Figure 11 clearly shows the magnitude of the compression set. A cross section of the flight o-ring compared to an unused o-ring shows that the flight o-ring has taken the shape of its groove indicating 100% compression set. The dimethyldisulfide gas vented from the batteries is the most likely source of the sulfur detected in the growth.

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*Private communication with M. Martin/EB11 and M. Mendrek/EH24 MSFC.

Silicon/Silicone/Silicate

Silicone contamination has been identified at several locations on the LDEF [19,20,21]. Although no principle source of silicone has been identified internal to the S0069 instrument, it does appear from the data that a silicone source existed. Silicone under exposure to atomic oxygen converts to a silicate[22].

The possibility that the silicone source was external to the S0069 experiment has to be considered. Sources have been identified both internal to the LDEF and from the Shuttle[23]. The S0069 experiment did not have a direct line-of-sight to these sources and at orbital pressures the mean free path for molecular collisions is several kilometers, which would make it unlikely that the localized thick (several microns) silicone deposits found could have formed from returned flux. In addition there appears to have been a flow of contamination from the interior of the S0069 experiment to the exterior. If the internal contamination deposits were from external sources, then the molecules would have to enter through vents and gaps in the front thermal panels normal to the RAM direction.

Rantanen[24] has performed calculations using the Integrated Spacecraft Environments Model (ISEM) which indicates potentially significant backscatter of outgassing contamination from the LDEF, back onto its RAM facing side. Further analysis is required to resolve this issue, but it may be that the silicone contamination layers originated from sources both internal and external to the S0069 experiment.

Oxygen/Carbon/Fluorine/Arsenic

The principal source of oxygen is presumed to be from orbital atomic oxygen. Fluorine identified in the growth base material could originate from the Teflon eroded by atomic oxygen. Carbon is reasonably plentiful, located both in the Teflon and from practically all non-metallic materials used in the TCSE instrument. Thermal vacuum bakeout of the S0069 instrument prior to flight should have reduced hydrocarbon sources to very low levels, consistent with ASTM E595[25]. The potential source of Arsenic has not been identified.

DISCUSSION

Dr. R. Warner*, Univ. of Minn., suggested that the growth may be similar to that reported by his colleague, Dr. G. K. Wehner. In Wehner's 1985 survey paper[26] he discusses the conditions for cone formation during whisker growth by ion bombardment of metal surfaces. One of the earliest reported observations of cone formations is by Guenterschulze and Tollmien[27] in their 1942 paper where they reported formation of cone forests on cathodes.

There are several growth characteristics reported by Wehner that are necessary for whisker and cone formations to occur. Most of the experiments reported were performed in a low energy ion sputtering high vacuum environment. Ion energies less than 500ev were capa-

*Private communication with Dr. R. Warner, in October 1992.

ble of achieving whisker growth with dissimilar metals. When the ion energies neared the sputtering threshold, whisker growth was observed. Whisker growth occurs when dissimilar metals are physically close (seed and substrate). Redeposition of sputtered materials results in unique formations of long thin and short thick cones. Inverted cones can also be formed. Whisker/cone orientation is not related to ion impact direction at low ion energies.

For the LDEF growth, the sputtering source was the orbital atomic oxygen (AO), which, being neutral, is capable of "sputtering" or erosion of materials at much less energy than the sputtering threshold, although the rates are very slow[28,29]. Atomic oxygen impacts the RAM face of the LDEF (Figure 2) at ~8 Km/sec or with a kinetic energy of ~5 ev. Wehner found that for metals, whisker growth occurred at low energies only when surface temperatures were elevated. Surface temperatures on the front cover were measured to be below ambient during the first 18 months and thermal model predictions indicated low temperatures throughout the mission.

The major difference between these ground experiments and the flight growth is that metals were used in the studies reported by Wehner, while the growth found on the TCSE experiment is non-metallic and initially polymeric which tends to undergo conversion at low temperatures. As a consequence, cones reported by Wehner were mostly faceted as one would expect, while the TCSE cones were not faceted. Interestingly, both hollow and inverted cone formations were reported, but no inverted hollow cones were reported by Wehner.

Taking into account all the growth conditions between flight and ground experiments makes the growth mechanism less likely to be the true "ion sputtering" related phenomena as reported by Wehner, but a very similar low energy neutral atomic oxygen related erosion phenomena.

CONCLUSIONS

Proposed Growth Scenario

After LDEF orbital insertion, exposure to atomic oxygen initiates surface erosion of the Teflon surface on the front thermal control cover. Since the LDEF was inserted in a high orbit and the solar cycle was in a low period, the AO flux was also low. Therefore the erosion rate was low. At the vent/gap interface where growth occurred, the Teflon surface erosion was in the form of roughly parallel ridges versus the normal peak and valley hill type surface texture found in the exposed areas. In time, outgassing molecules of silicone and other contaminants reached this narrow gap between the front thermal covers, which provided a vent, resulting in deposition of thin layers of contamination. Solar ultraviolet photons incident on the Teflon interacted with the thin silicone contamination layer to form longer chain, lower vapor pressure, silicone materials. This photodeposition process continued, resulting in a thick, brown varnish type layer. Away from the gap, the silicone contamination flux was dispersed to such low levels that the AO erosion of Teflon dominated and no silicone buildup could be sustained.

As the atomic oxygen level increased, resulting from the LDEF orbital decay and solar cycle heading towards a high period, the silicone was transformed to a silicate[20,22]. At about 3 years into the mission the battery cells ruptured, venting a continuous source of dimethyldisulfide gas. At this period in time, all growth elements existed. Figure 12 is a schematic of the growth environmental conditions and associated hardware orientation.

Initially, hollow whiskers are formed which slowly form inverted cones as growth

progresses. Growth is driven by redeposition of silicate contamination by atomic oxygen erosion or "sputtering". Simultaneously, dimethyldisulfide outgassing molecules react with AO freeing sulfur, possibly in the form of a sulfate (SO_3), which is then incorporated into the whisker/cone growth. Sulfate has a solubility in silica of $<0.6\%$[30]; therefore, the sulfur concentration should be low, as was found from the EDAX data.

The proposed scenario fits the existing data and knowledge of events. As other observations of similar growth are reported and analyzed[31] a better understanding of the growth process can be developed. With a better understanding of this intriguing whisker/cone growth phenomena, intentional growth can be performed, thereby providing a means to process material on a microscale with unique surface morphologies and physical characteristics.

ACKNOWLEDGEMENTS

The authors would like to recognize the interest and support of the following individuals: scanning electron microscope and EDAX analysis by James Coston/MSFC; biological tests by Dr. E. Rodgers/MSFC and Dr. D. Obenhuber, Sverdrup Inc.; and battery offgasing sampling and analysis by R. Crowder, SEA Inc. and J. Perkins/EH32 MSFC. Technical assistance was provided by Dr. John Reffner of Nicolet Instrument Corp., and Dr. Pamela Martoglio of Spectratech Applied Systems for the total attenuated microprobe FTIR analysis.

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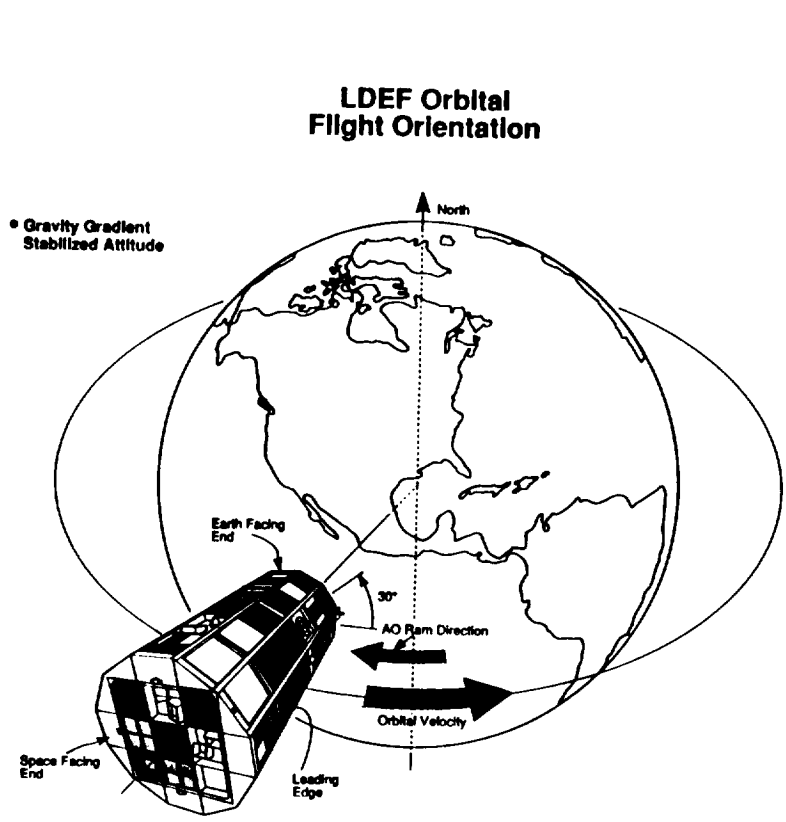


Figure 1. Schematic of the LDEF in Earth Orbit.

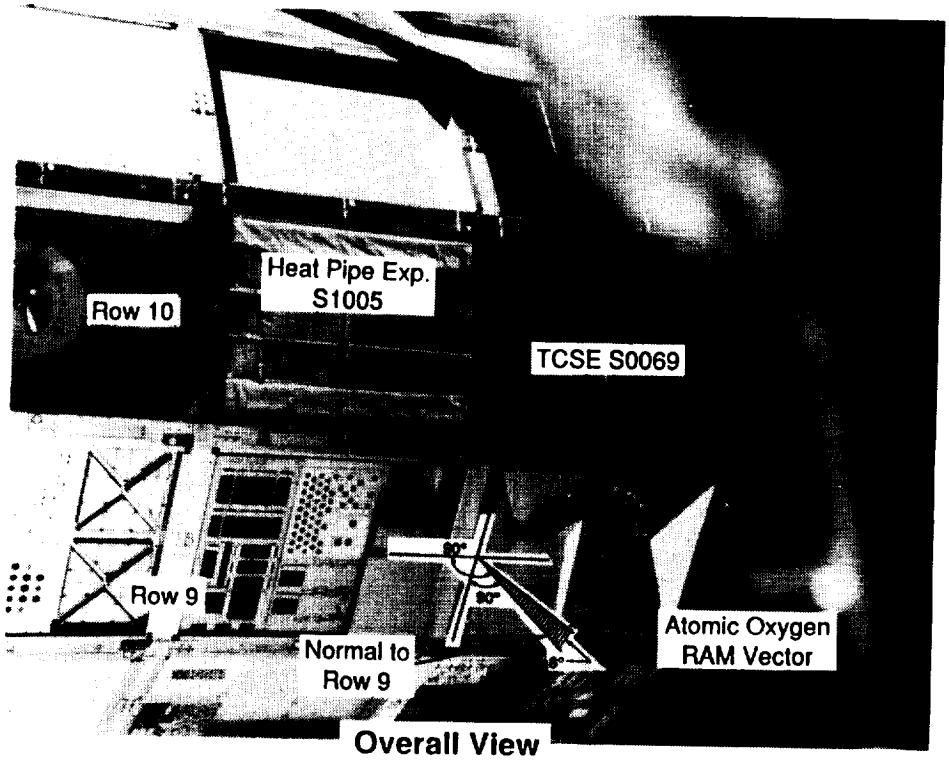
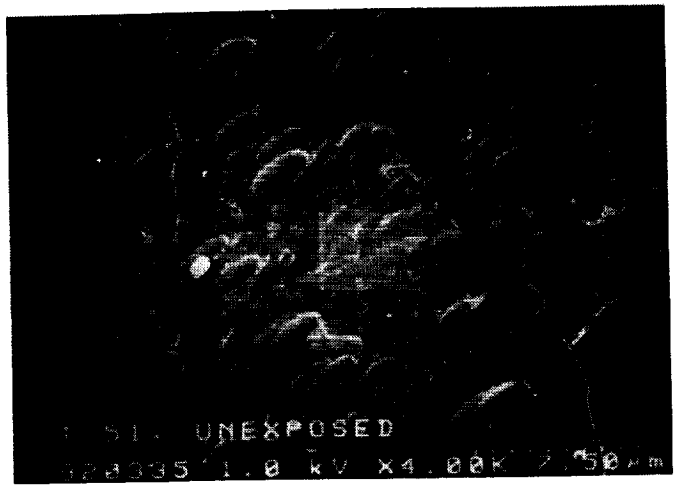
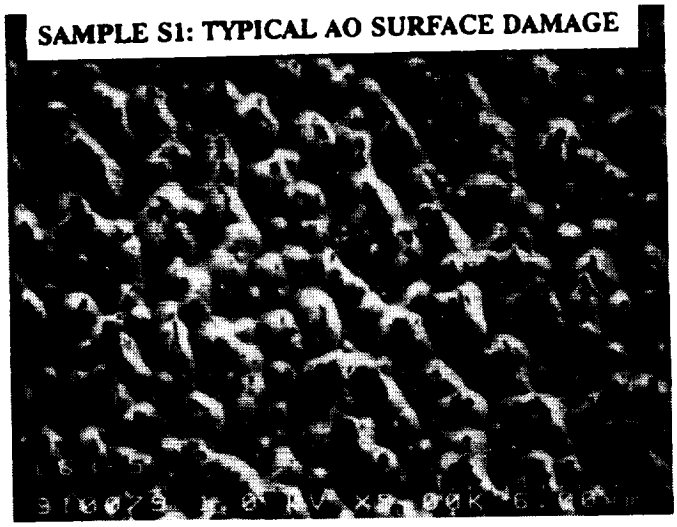


Figure 2. S0069 TCSE Experiment on LDEF During Retrieval

SAMPLE T51: COVERED REGION NEAR GROWTH



SAMPLE S1: TYPICAL AO SURFACE DAMAGE



SAMPLE T51: EXPOSED REGION NEAR GROWTH

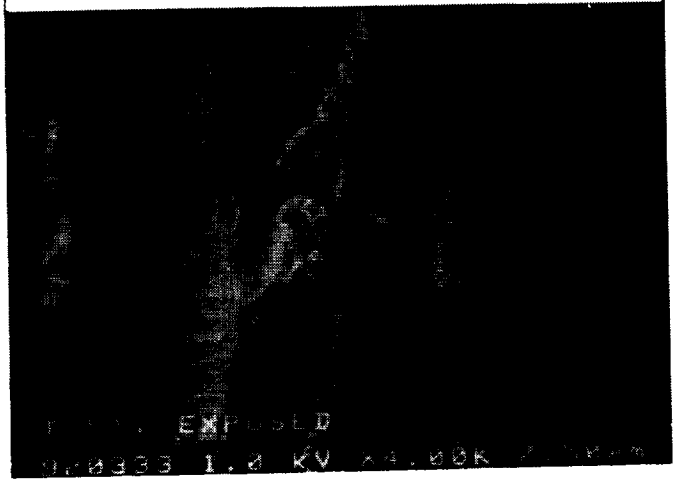


Figure 3. SEM Images of Teflon Surfaces on S0069 for covered and exposed locations.

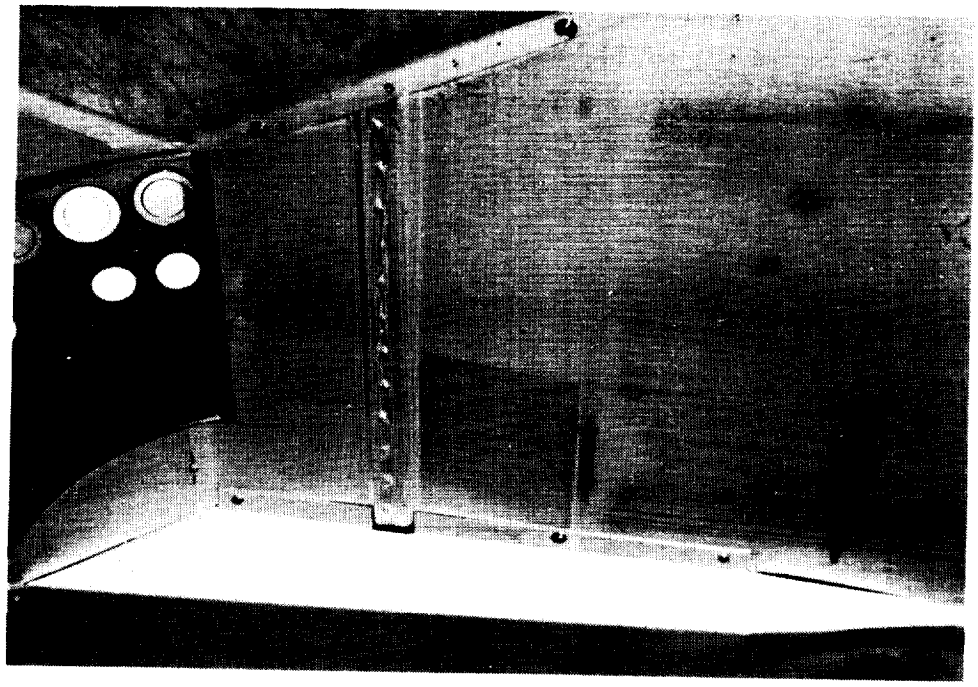


Figure 4. Front Surface of S0069 TCSE Instrument in the Laboratory after Retrieval Showing the Brown Streaks and the Gap (vent) between the Front Covers.

Figure 5. Front Thermal Control Cover Removed from the S0069 Instrument Showing Covered Regions, Exposed Regions, and location of Whisker/Cone "Growth".

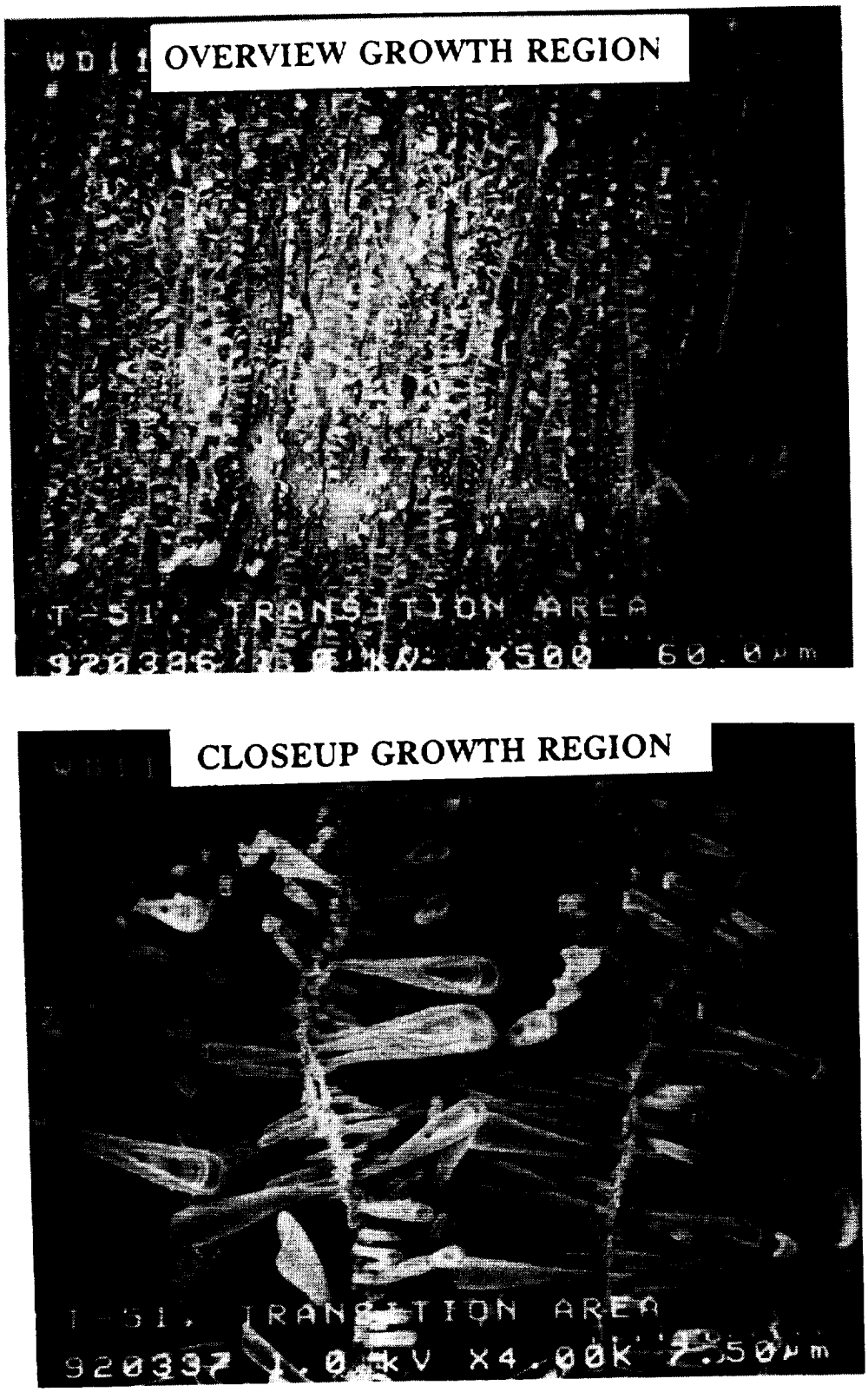


Figure 6. SEM Images of the Whisker/Cone Growth.

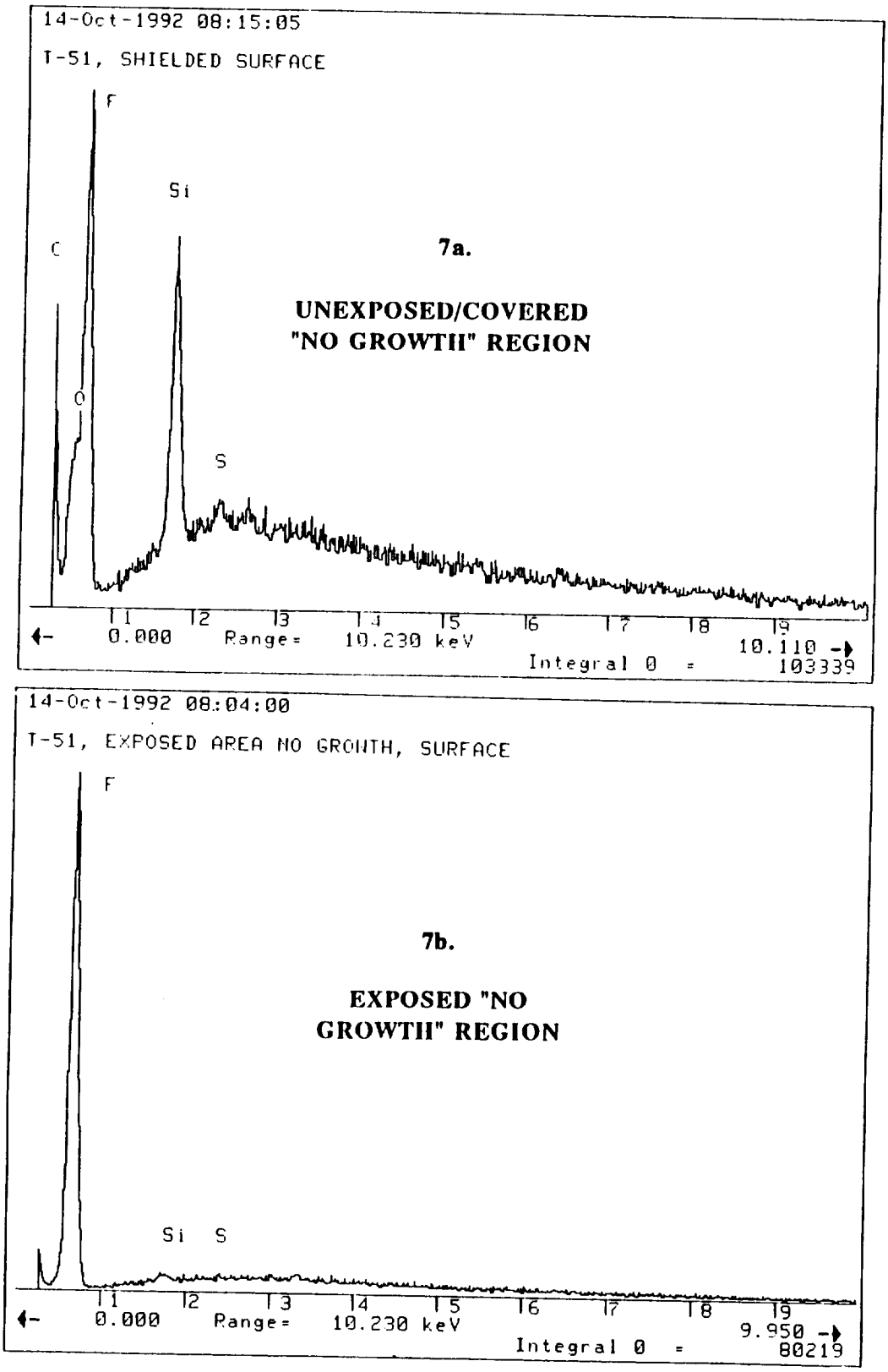


Figure 7. EDAX Data for the Silver Teflon Surface of Sample T51.

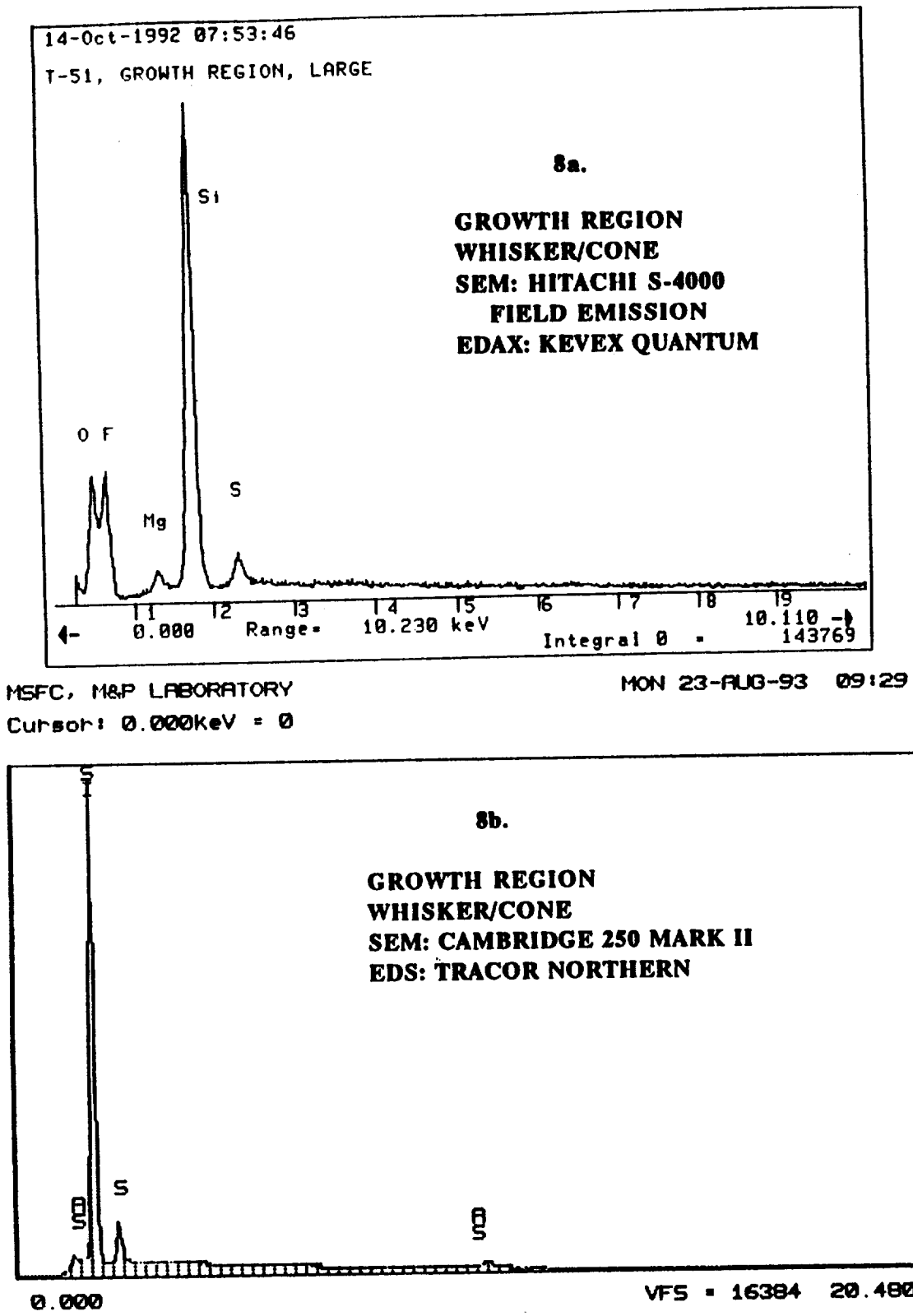


Figure 8. EDAX Data for the Whisker/Cone Growth on Sample T51.

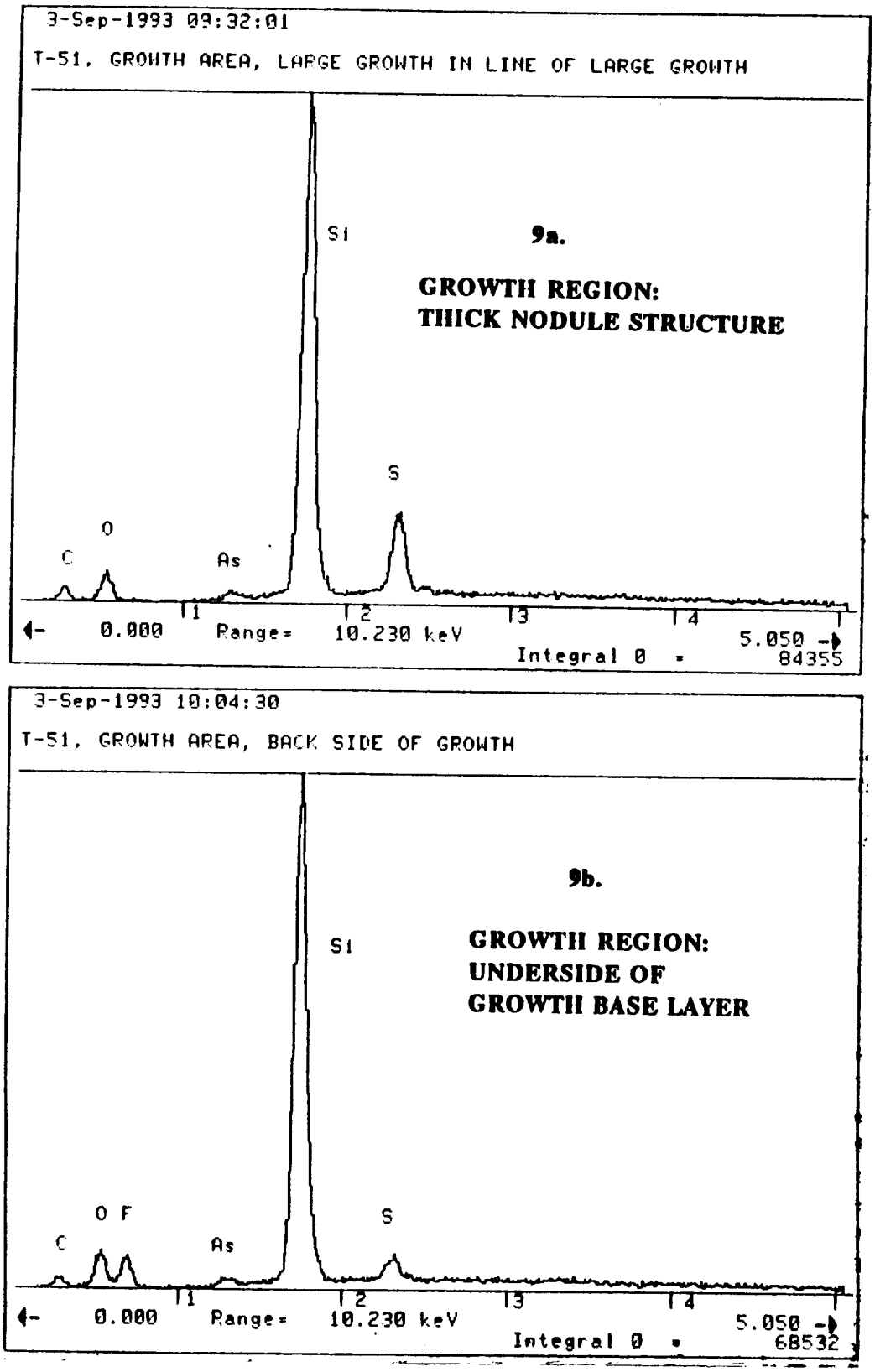


Figure 9. Comparison of EDAX Data for Whisker/Cones and Growth Base Layer.

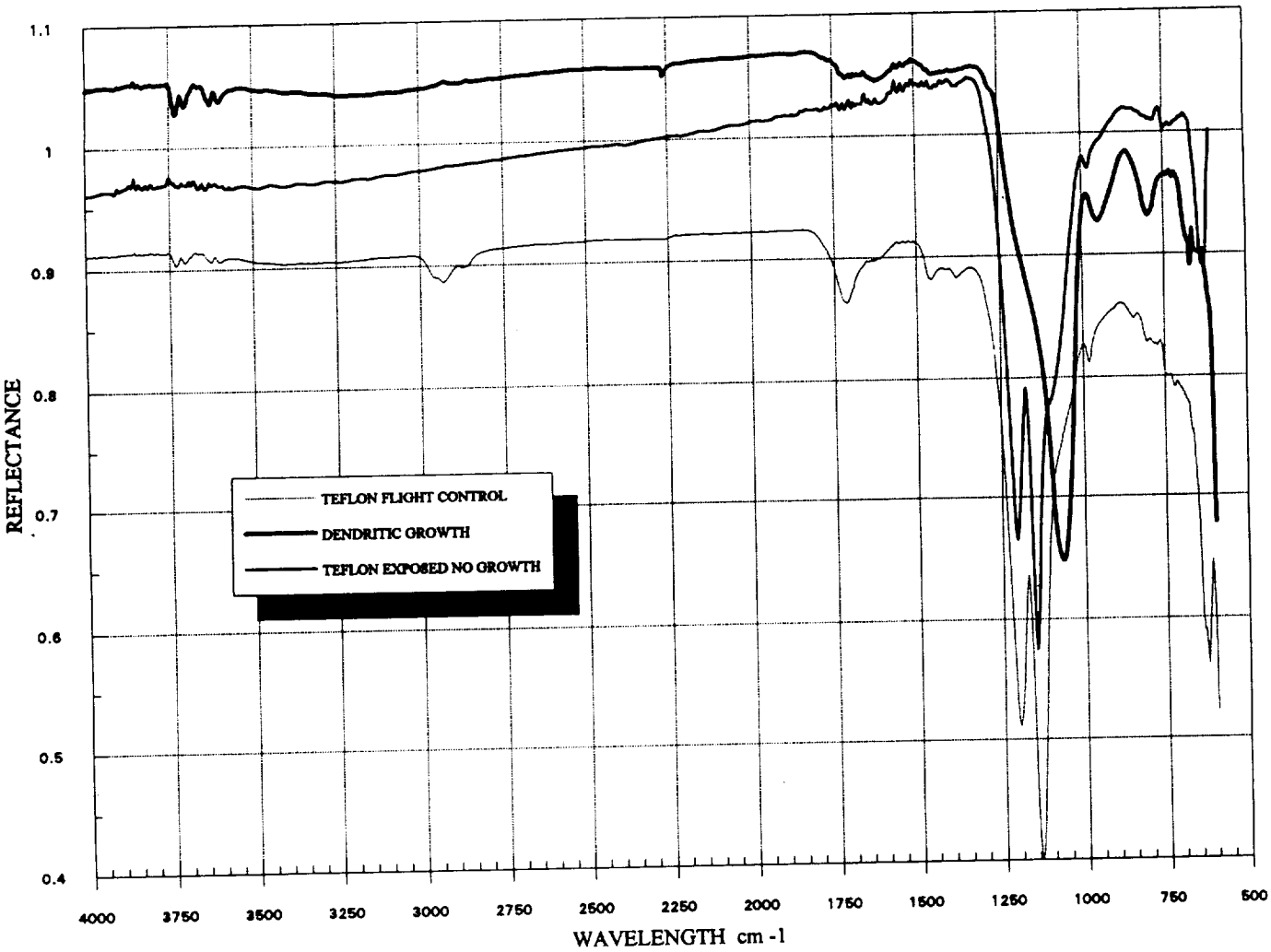


Figure 10. FTIR, Total Attenuated Microprobe Analysis Data for Sample T51.

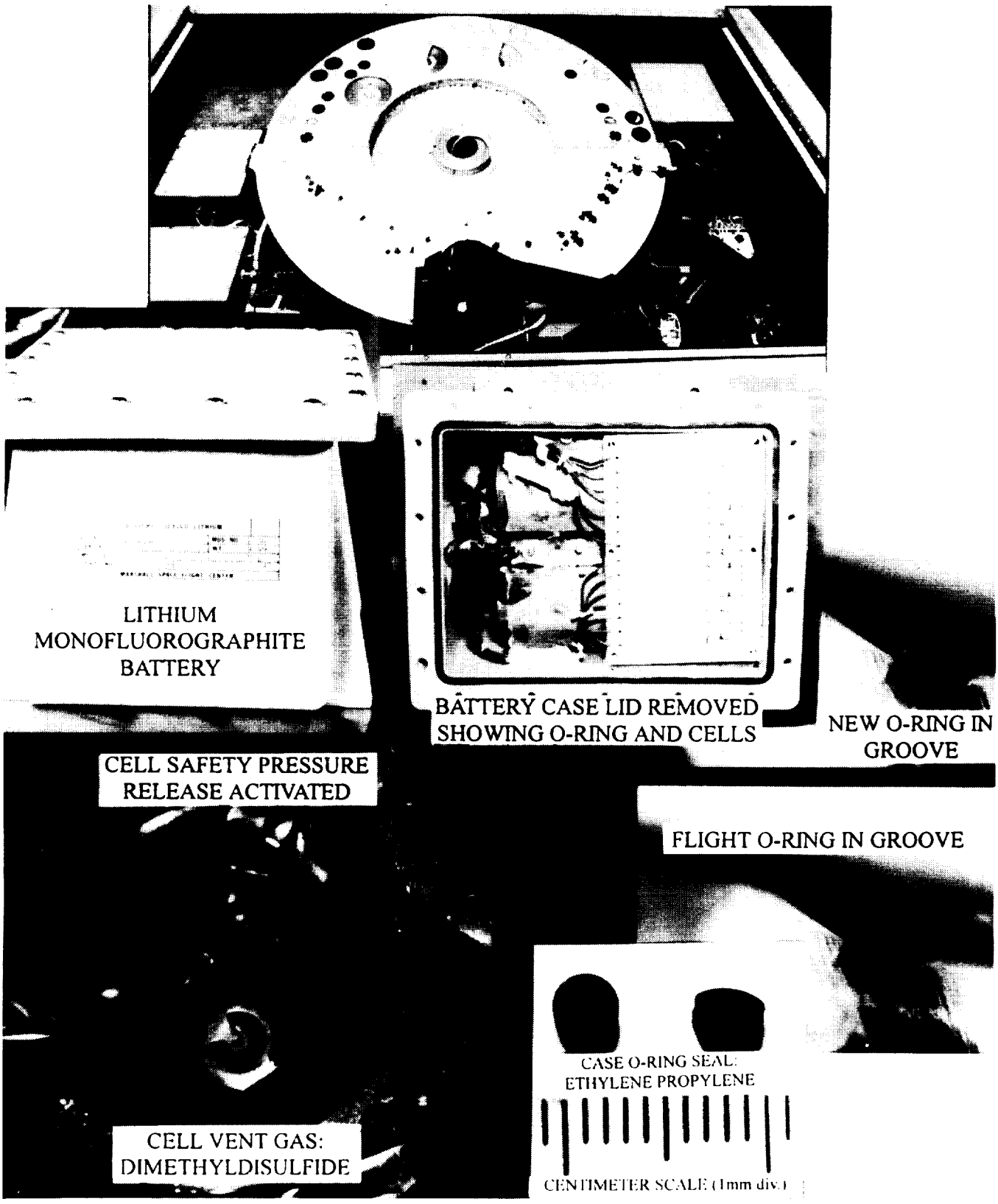


Figure 11. S0069 Lithium Monofluorographite Batteries and Leakage of Dimethydisulfide Gas.

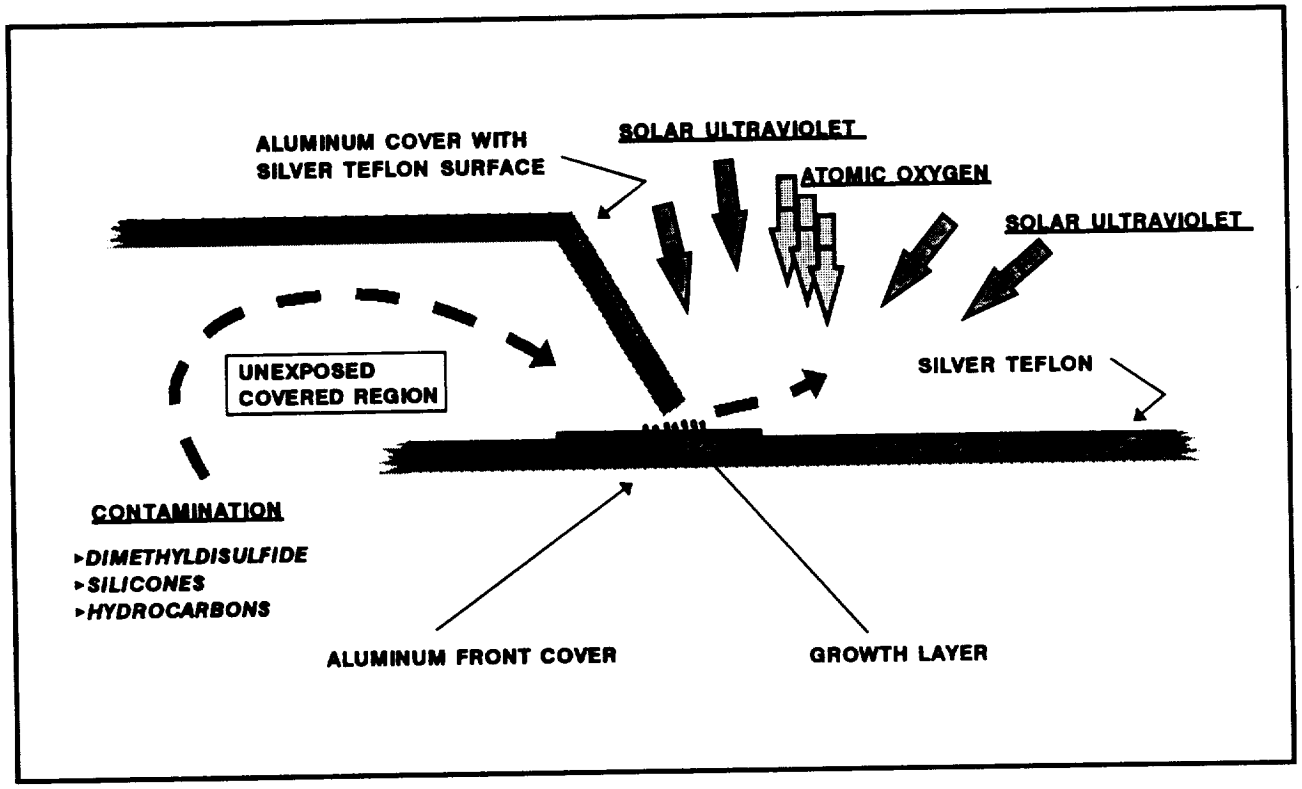


Figure 12. Schematic of Space Environmental Growth Conditions for the Whisker/Cone Growth.