

LDEF FIBER-COMPOSITE MATERIALS  
CHARACTERIZATION

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ABSTRACT

In this study we observed and compared degradation of a number of fiber/polymer composites located on the leading and trailing surfaces of LDEF where the atomic oxygen (AO) fluences ranged from  $10^{22}$  to  $10^4$  atoms/cm<sup>2</sup>, respectively. While matrices of the composites on the leading edge generally exhibited considerable degradation and erosion-induced fragmentation, this "ashing" process was confined to the near surface regions because these degraded structures acted as a "protective blanket" for deeper-lying regions. This finding leads to the conclusion that simple surface coatings can significantly retard AO and other combinations of degrading phenomena in low-Earth orbit. Micrometeoroid and debris particle impacts were not a prominent feature on the fiber-composites studied and apparently do not contribute in a significant way to their degradation or alteration in low-Earth orbit.

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## INTRODUCTION

Composites have played an important role in a host of space and aerospace materials systems and are currently one of the most promising materials areas not only in the context of advanced aerospace systems but also a wide range of commercial applications as well. Early re-entry ballistic missile components, especially nose cones and heat shields, relied upon composites and related fiber materials systems, and when the Long Duration Exposure Facility (LDEF) was conceived as a space materials test facility, numerous composite materials experiments were designed to examine the effects of low-Earth orbit on these contemporary as well as more advanced composites of that period (early 1980's). These included a range of medium-to-light weight polymer (epoxy) matrix/fiber composites, especially unidirectional, bidirectional composites and laminated graphite and glass fiber composites.

As shown in Fig. 1, LDEF was a 12-sided re-usable, hollow satellite about the size of a bus (4.6m x 9.2m). It weighed roughly  $10^5$  kg and contained some 10,000 specimens for test and analysis when deployed on orbit April 7, 1984 by the Shuttle Orbiter Challenger. When retrieved by the Shuttle Orbiter Columbia on January 12, 1990, LDEF's circular, non-geosynchronous, low-Earth orbit of 257 nautical miles (476 km) had decayed to roughly 180 nautical miles (333 km).

Composite samples to be described in this study were located either in row 9, bay D (D09) on the leading edge of the stabilized satellite, or in row 3, bay D (D03) on the trailing edge of the satellite. These distinctions, as illustrated in Fig. 2, were especially dramatic in the context of atomic oxygen (AO) fluence which was observed to vary from about  $10^{22}$  atoms/cm<sup>2</sup> on the leading edge to about  $10^4$  atoms/cm<sup>2</sup> on the trailing edge.

In addition to the AO flux difference, the leading edge samples experienced a temperature difference of nearly 100°F over 34,000 orbital cycles. Exposure during these cycles included intense UV, X-ray, electron, proton, gamma ray, and cosmic radiations. Micrometeoroid impacts (nearly 1 billion over the 130 m<sup>2</sup> of LDEF surface) and other contaminating particles (more than a trillion over the surface) along with outgassing of a silicone-hydrocarbon film also influenced the surface structure and integrity of many test materials [1, 2].

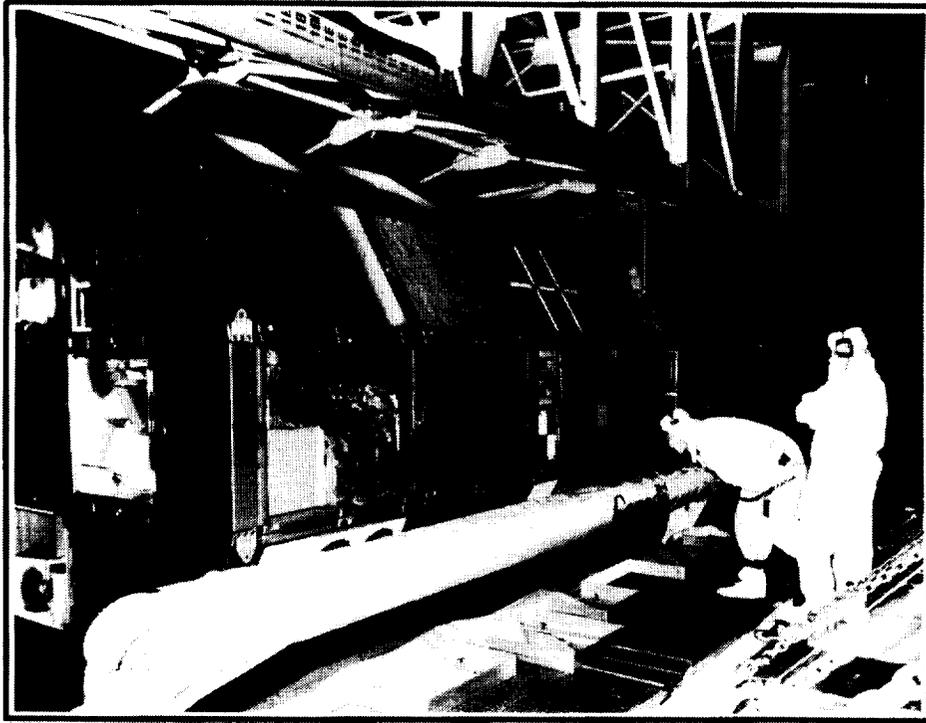


Figure 1. Long Duration Exposure Facility (LDEF) during post-recovery examination at Kennedy Space Center.

#### EXPERIMENTAL DETAILS

In this study we have examined representative fiber composites from both the leading and trailing edges of LDEF and compared them with control samples which were not flown on LDEF using optical metallography and scanning electron microscopy (SEM) techniques. Samples which were examined in this study included the following: graphite polyimide, graphite polysulphone, tape-wrapped carbon phenolic (a multi-directional carbon fiber weave in a phenolic binder) pyrocarb 431, and quartz phenolic.

#### RESULTS AND DISCUSSION

**Graphite Polyimide** - Figure 3 shows for comparison purposes, both leading and trailing edge examples of the graphite polyimide composite in the same relative orientations between corresponding bays (refer to

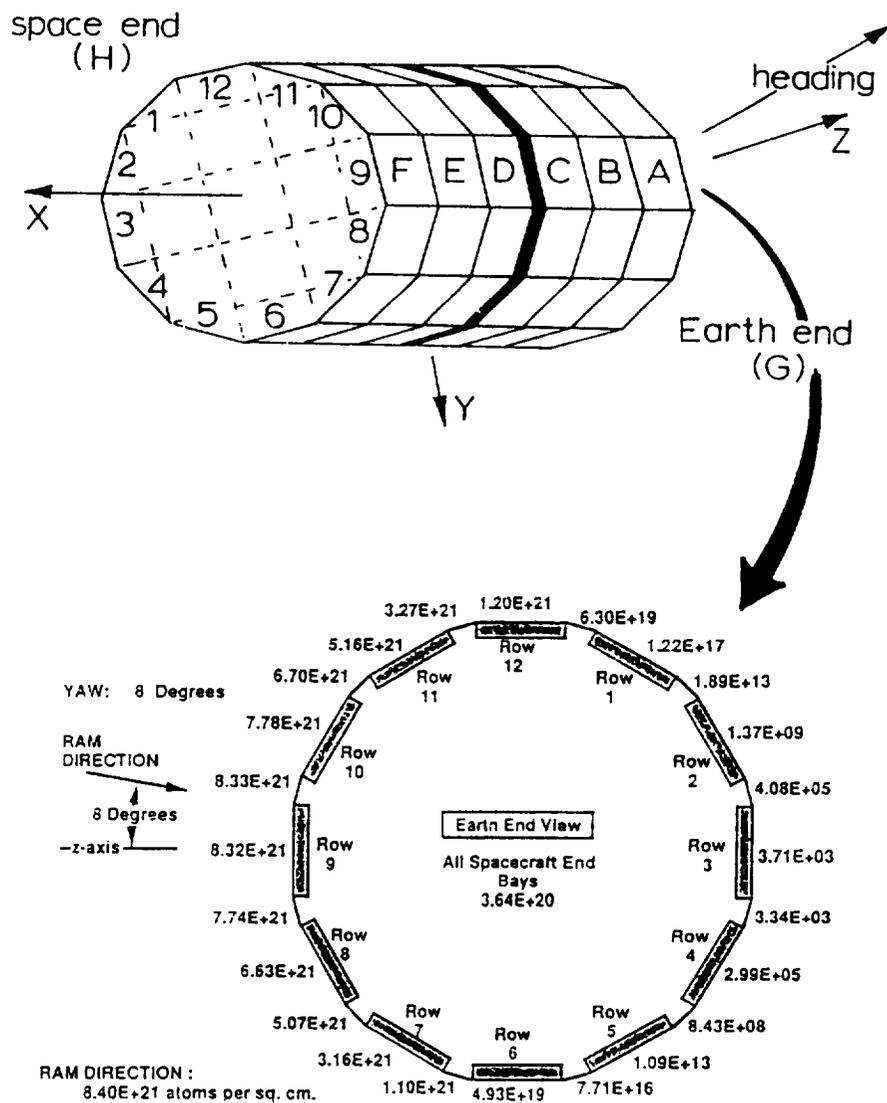


Figure 2. Schematic view of LDEF oriented as in Fig. 1 showing orientation and tray notations (top) and corresponding atomic oxygen fluences (bottom) (Courtesy of NASA).

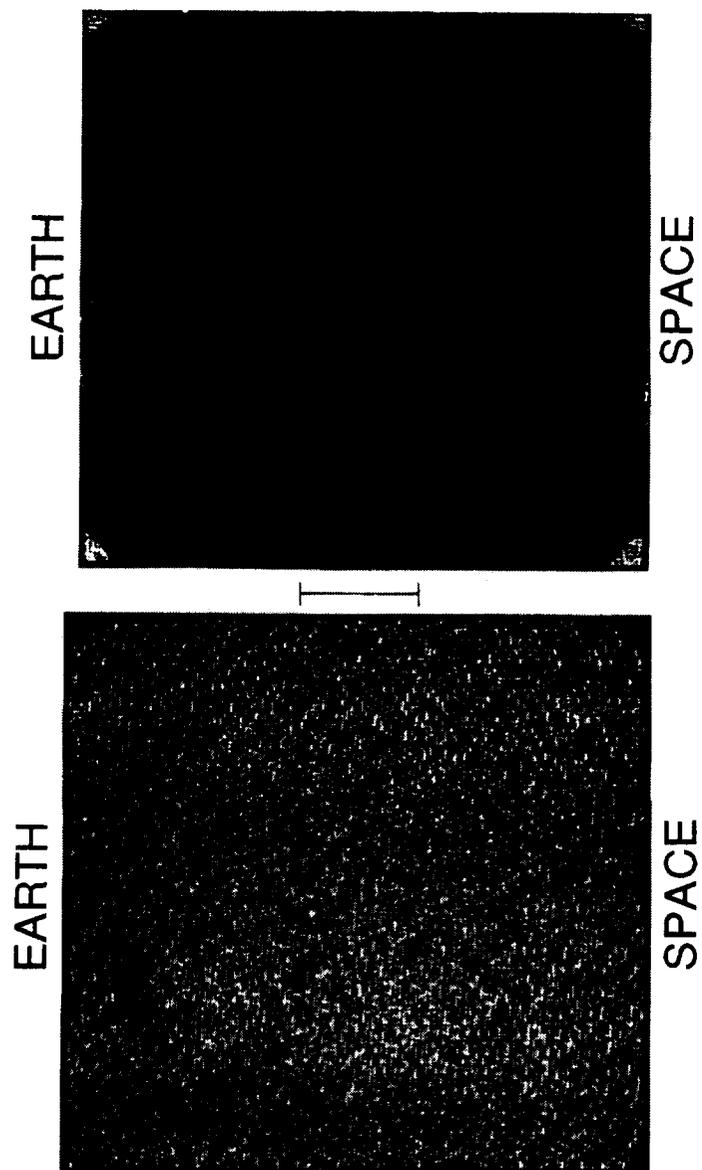


Figure 3. Leading (D09) (top) and trailing (D03) (bottom) edge samples of graphite-polyimide composite from LDEF. Note corners of leading edge sample protected by clamping washers appear similar to the trailing edge sample (marker corresponds to 1 cm).

Fig. 2(a)). While the weave (fiber) pattern degradation is not apparent in this comparative figure, the magnified views provided in Fig. 4 show that the leading edge degradation is much more severe than that experienced in the trailing edge.

**Graphite Polysulphone** - Figure 5 shows a similar pattern to that of Fig. 4 for leading-edge degradation in graphite polysulphone composite. In addition Fig. 5 shows a comparative view of a control sample (Fig. 5(c)) which suggests that while the trailing edge degradation was not very noticeable compared to that observed on the leading edge, there were some subtle changes which may be related to volatilization or related phenomena. Morphologies essentially identical to those shown in Fig. 3 were also observed on the leading and trailing edge graphite polysulphone samples and are therefore not reproduced here.

**Tape-wrapped Carbon Phenolic** - Figure 6 shows typical examples of light microscopy observations of the tape-wrapped carbon phenolic composite taken from the leading and trailing edge locations in bay D of LDEF. These views show the degradation and morphology on the leading edge to be essentially identical to that observed for the graphite polyimide shown in Figs. 3 and 4 and the graphite polysulphone shown in Fig. 5, as well. Although the surface features of Figs. 4 to 6 were observed at low magnifications, they are similar in appearance, leading us to conclude that the degradation mechanisms are essentially the same for each of these fiber composite systems.

**Pyrocarb 431** - Figure 7 shows a low magnification view of the leading edge Pyrocarb 431 composite. It is interesting to note that a chalk number provided some protection against underlying degradation.

Figure 7(b) shows that areas under the chalk mark were maintained relatively undegraded, which attests to the ability to provide simple protective measures for polymers exposed in space in low-Earth orbit (LEO).

**Three Dimensional Quartz Phenolic** - In contrast to the other composites examined in this study, the quartz phenolic exhibited much less degradation as indicated in the comparative views reproduced in Figs. 8 and 9. Figure 9(a) also shows the interface between two groups of quartz fibers perpendicular to each other and shows little degradation in this (interface) region. Apparently considerably less

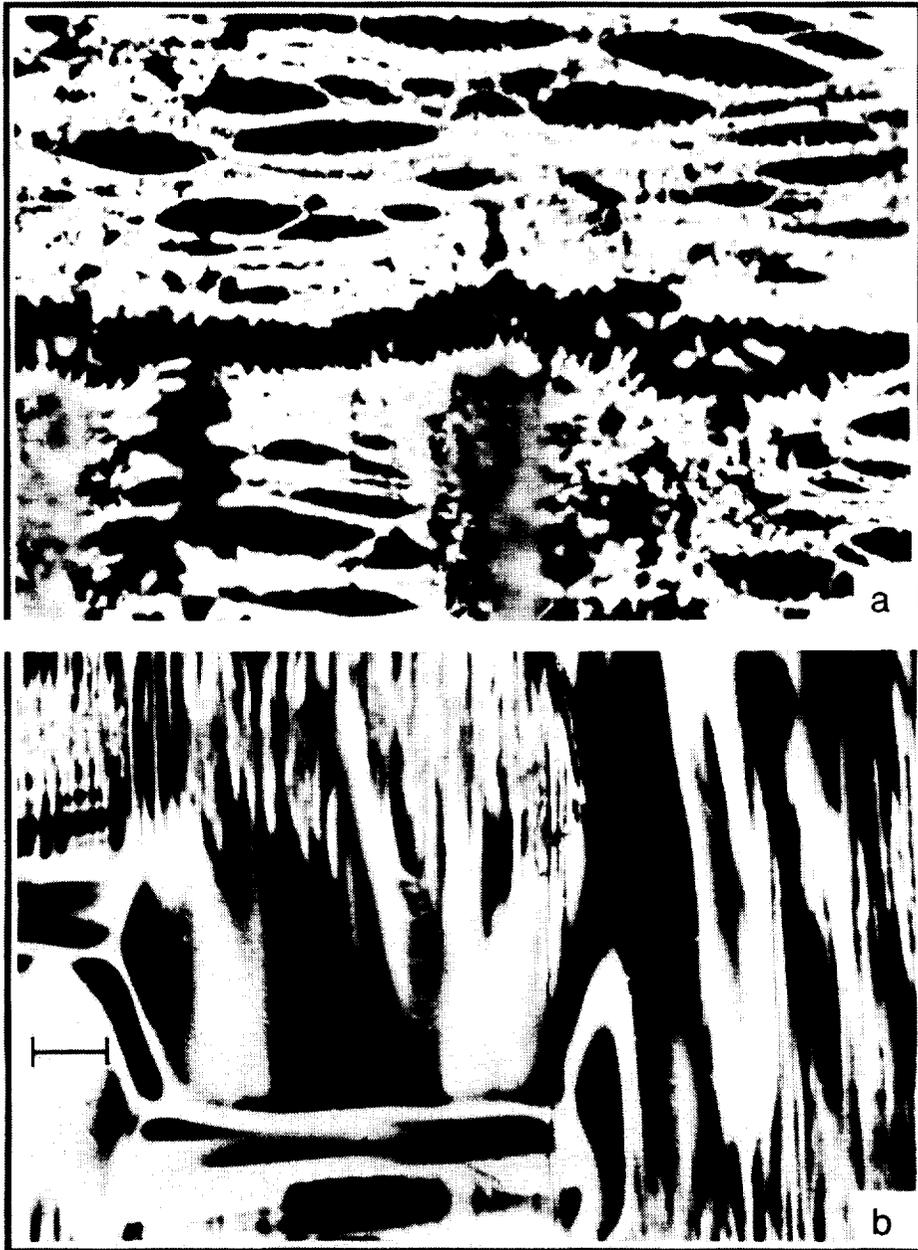


Figure 4. Magnified views (in the light microscope) of leading (a) and trailing (b) edge samples of graphite-polyimide on LDEF showing leading edge surface degradation. (Marker is 0.1 mm.)

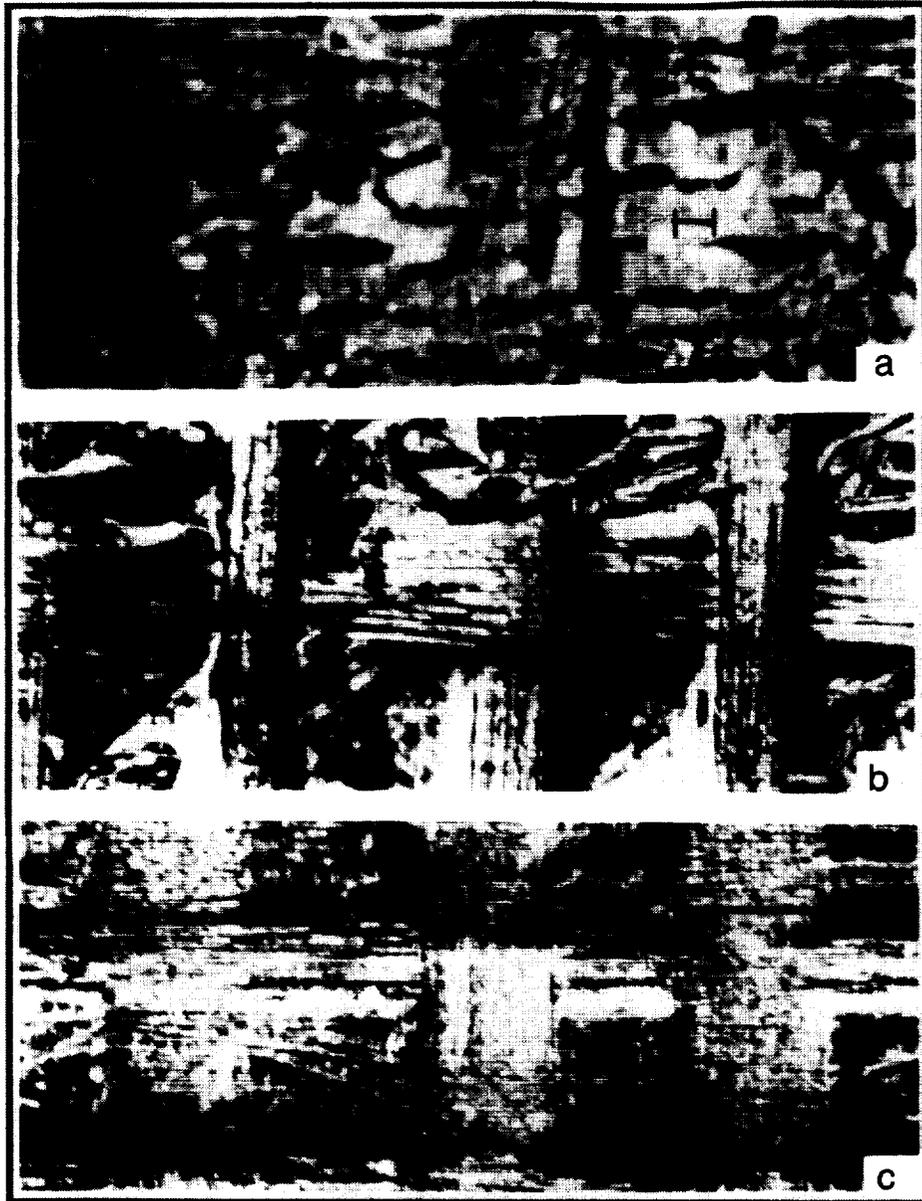


Figure 5. Comparison of light microscope views of graphite polysulphone composite. (a) Leading edge LDEF sample of graphite polysulphone. (b) Trailing edge LDEF sample. (c) Control sample not flown on LDEF. (Magnification marker corresponds to 0.1 mm).

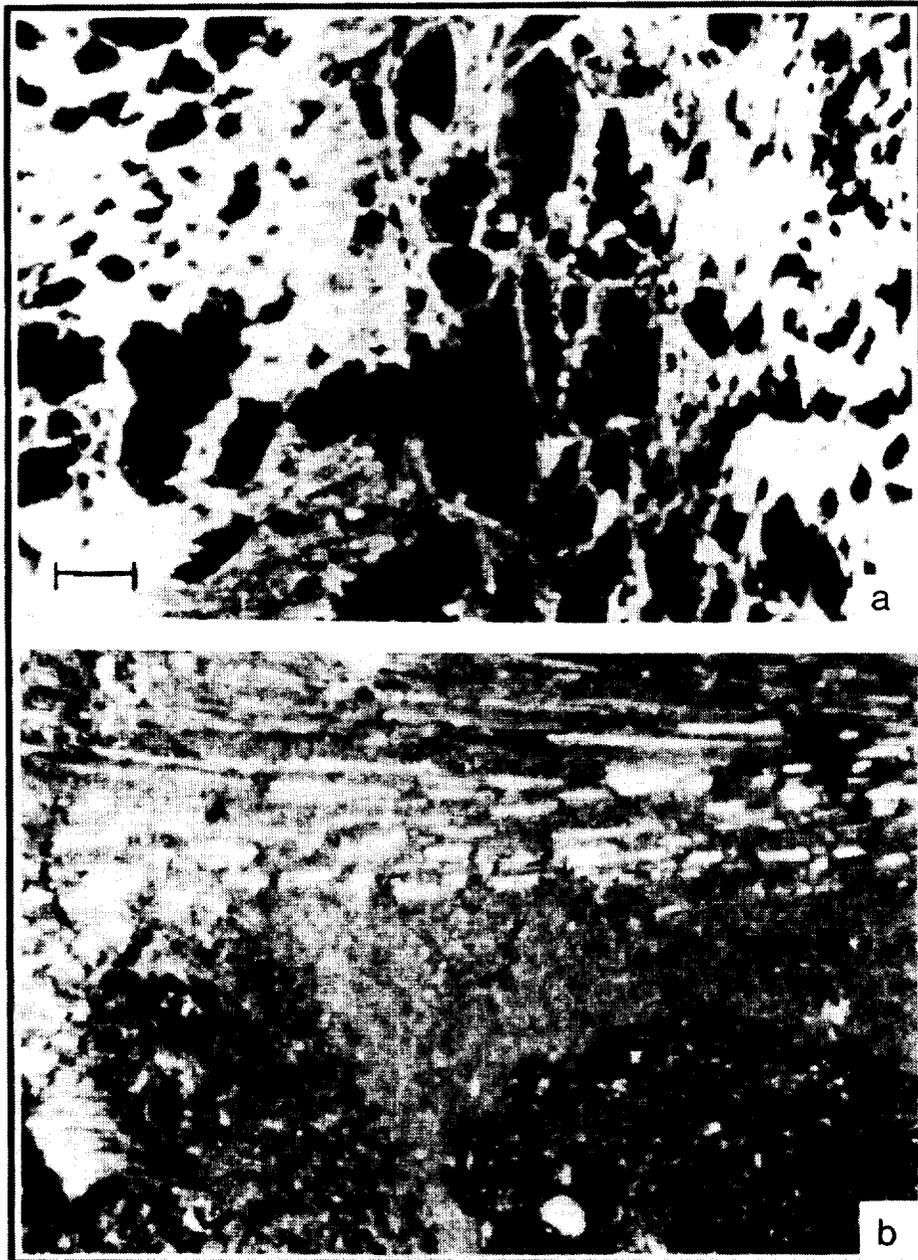


Figure 6. Leading (top) and trailing (bottom) edge views of tape-wrapped carbon phenolic on LDEF. (Marker is 0.1 mm).

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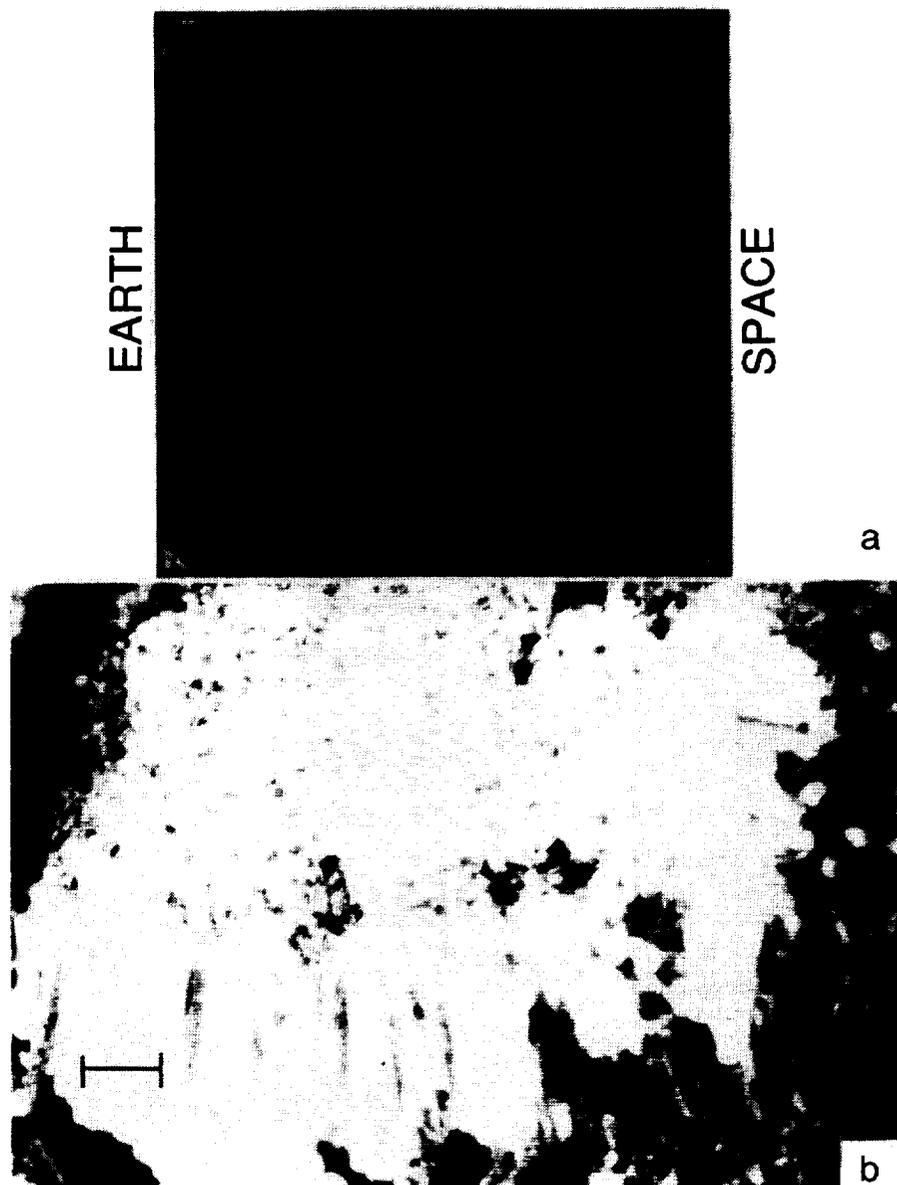


Figure 7. Comparative views of Pyrocarb 431 composite. (a) Low magnification view of leading edge sample (a). (b) Leading edge area under the chalk number shown in (a) is illustrated in (b). The chalk tends to protect the underlying regime from degradation. (Marker is in (b) 0.1 mm).

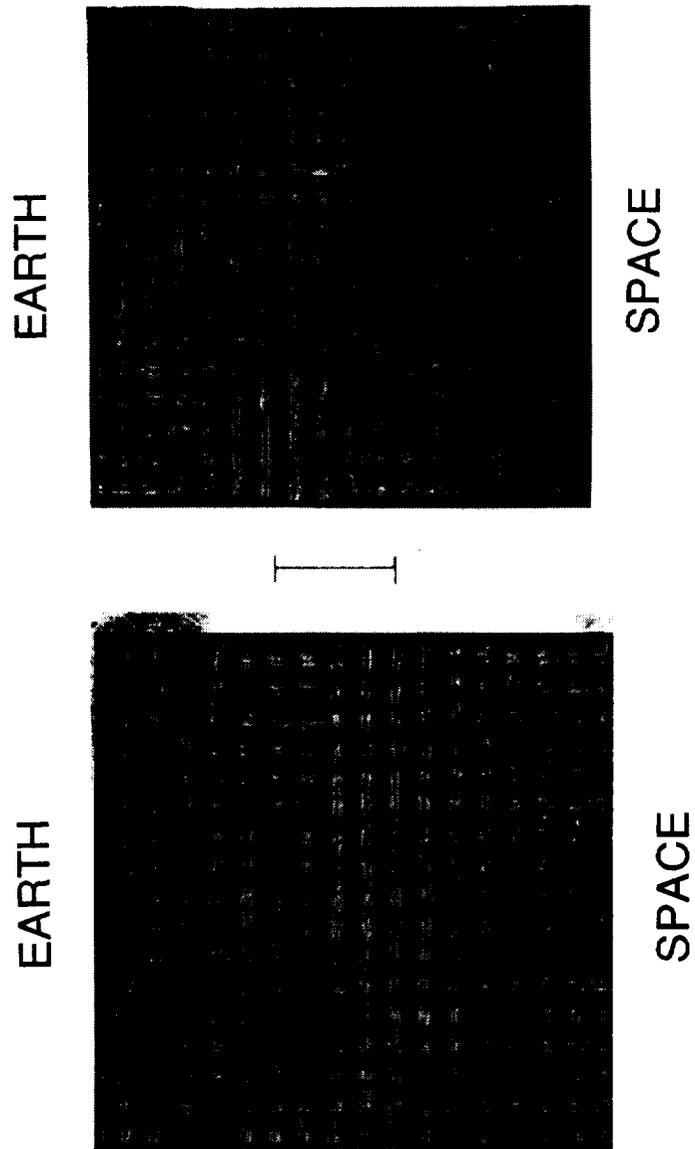


Fig. 8. Leading (top) and trailing (bottom) edge views of the 3-dimensional quartz phenolic composite on LDEF. (Marker is 1 cm).

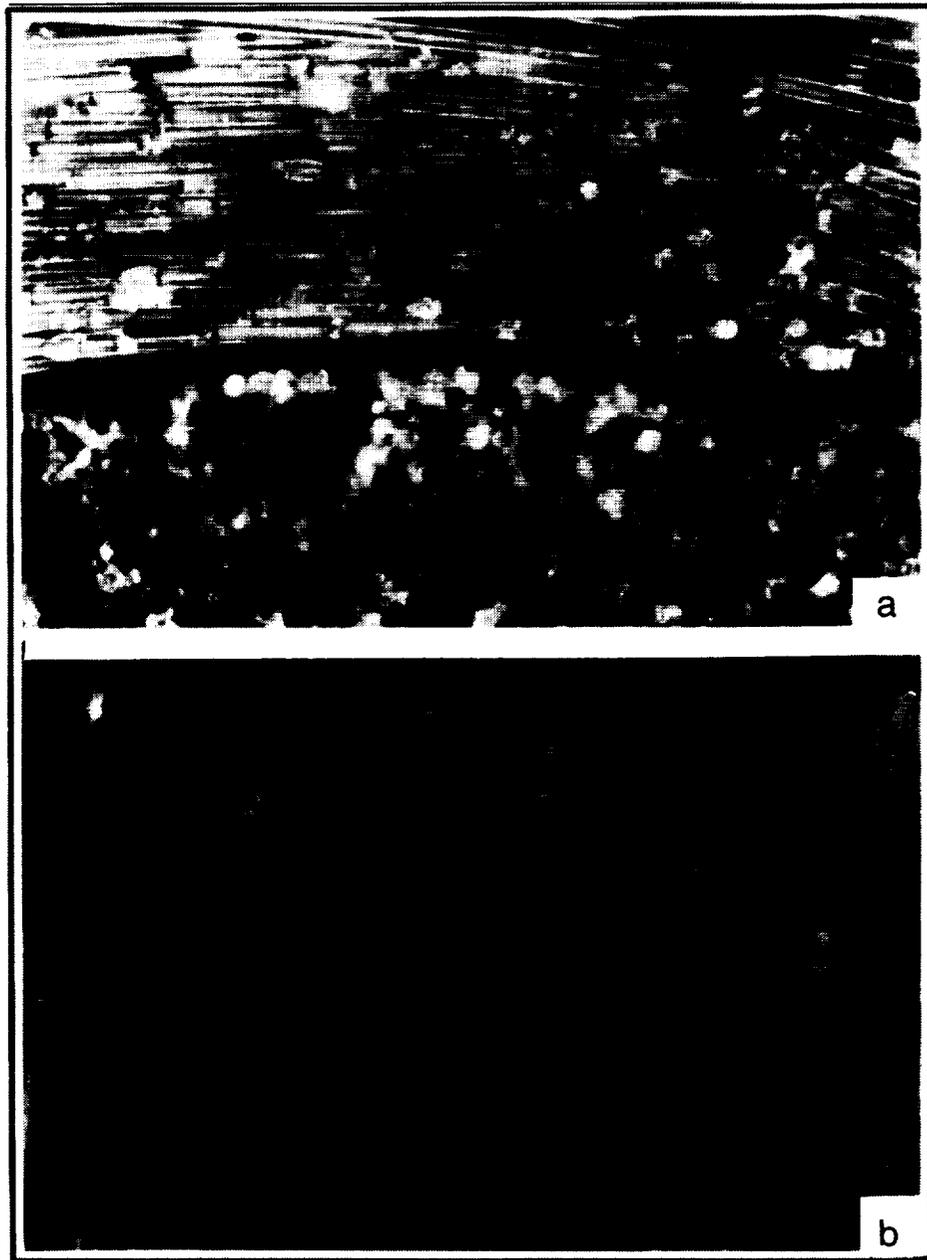


Figure 9. Magnified leading (a) and trailing (b) edge views of the quartz phenolic composite shown in Fig. 8. (Magnification markers equal 0.1 mm).

damage occurs in quartz phenolic composites in LEO than for the other composites examined (compare Figs. 4, 5, 6, and 9).

While the optical (light) microscope views shown in Fig. 9 do not exhibit any noticeable degradation of this composite, more detailed observations in the SEM reveal several degradation features especially in the phenolic (polymer) binder which are similar to other SEM observations for the other polymers. These features are illustrated in a series of SEM views reproduced in Fig. 10.

**Surface Erosion Phenomena** - The degradation of the polymer matrices in high AO fluences in LEO as illustrated for the leading edge LDEF composites has been discussed previously to be a consequence of polymer bond breaking and subsequent molecular fragmentation leading to erosion of material [3-6]. This phenomenon is particularly severe for certain polymer chain structures such as polyethylene, kapton [5], polyimide, and polysulphone studied here. This energetic AO erosion process (8 km/s orbital velocity produces 5 eV collision energy) is catalyzed and accelerated by UV radiation and altered in some cases by orbital thermal fluctuations and temperature localization which alters the eroded surfaces, creating a plethora of erosion-degradation structures (Fig. 10).

As noted earlier the AO-induced surface erosion, especially for carbon in polyimide and polysulfone binder matrices (Fig. 11), creates a surface region of molecular fragments and larger ash-like fragments. Like chalk marks on the surface (see Fig. 7), these fragments provide a protective regime that retards the erosion process and limits the degradation to a few microns of surface region at worst in polymer composites observed in this investigation.

**Observations of Micrometeoroid Impact Phenomena** - Because of the size of the fibers and the weave spacings it is often difficult to observe micrometeoroid or debris particle impact damage in fiber/polymer composites. This is because the cratering will cause melting or vaporization which can trap the particles (which are usually 1/5 the crater diameter) below the surface where it (the crater) would be unobservable. Carbon fiber composites, because of their melt/vapor features and fiber weave, can serve as an efficient absorber of impacting particle residue as well. These features are illustrated in Figs. 11 and 12. Figure 11 shows a large (0.1 mm) impact crater in a graphite polyimide which was probably a paint chip because of its Ti-Ca-Si composition. Figure 12 shows two examples of micrometeoroid impacts in the aluminum frame surrounding the leading edge quartz phenolic

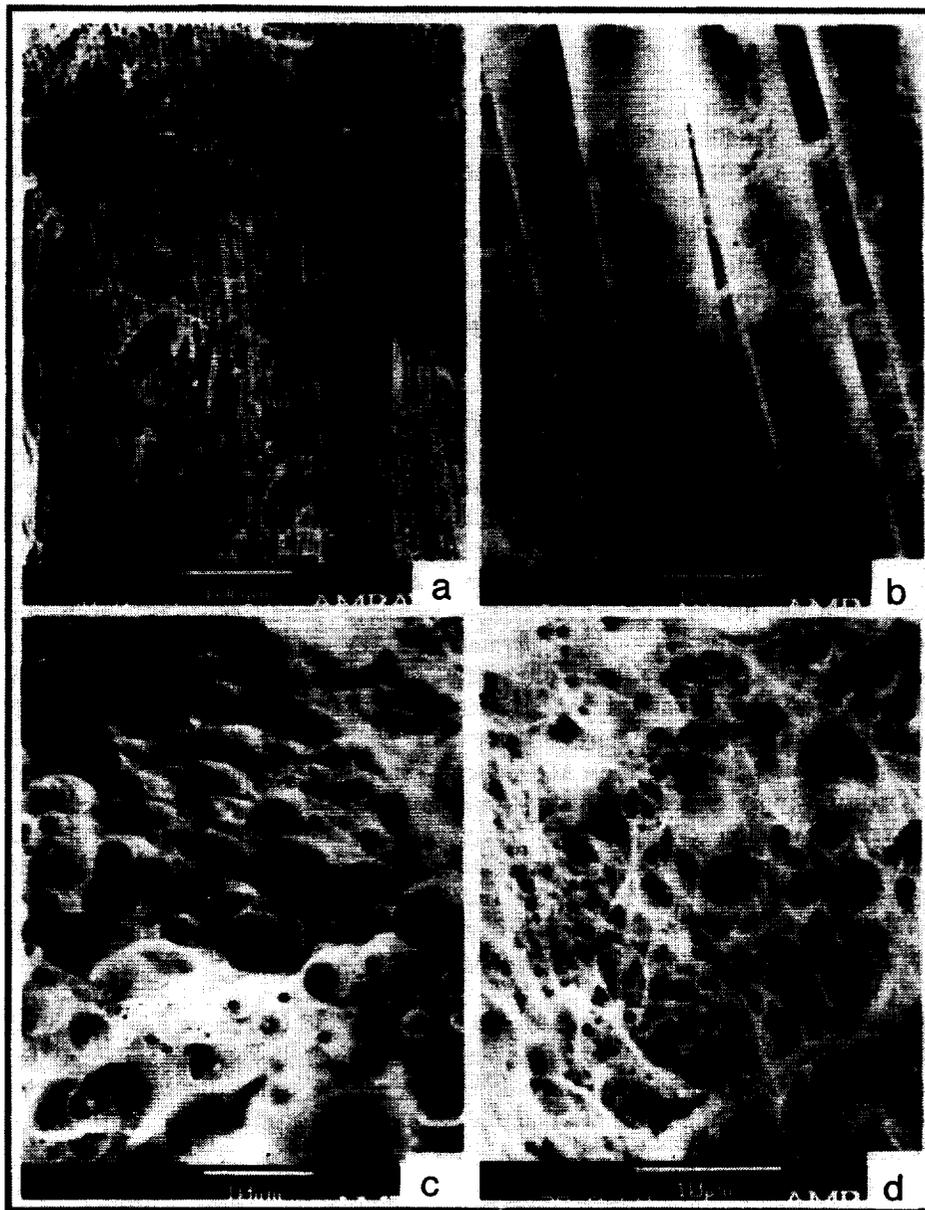


Figure 10. SEM views of phenolic degradation in quartz phenolic composites on the leading edge of LDEF. (a) Low magnification view showing filmy surface residue. (b) Magnified views of (a). (c) Protruding surface features. (d) Surface erosion pits presenting an inverse view of features in (c).

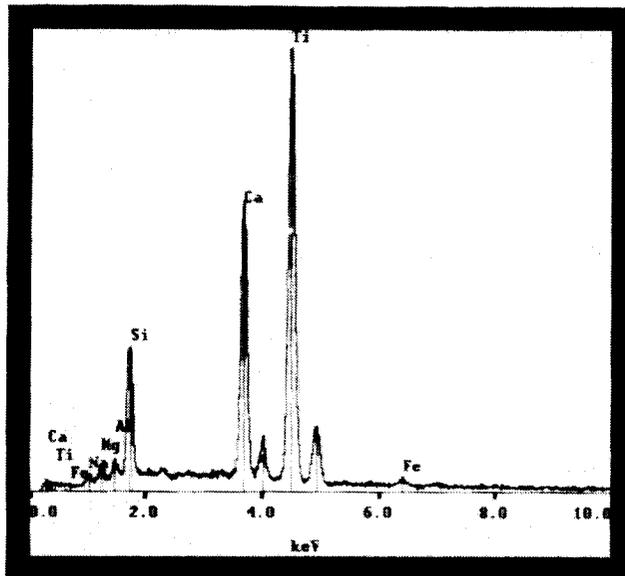


Figure 11. Impact crater in graphite polyimide composite (a) and corresponding energy-dispersive X-ray spectrum showing particle residue composition.

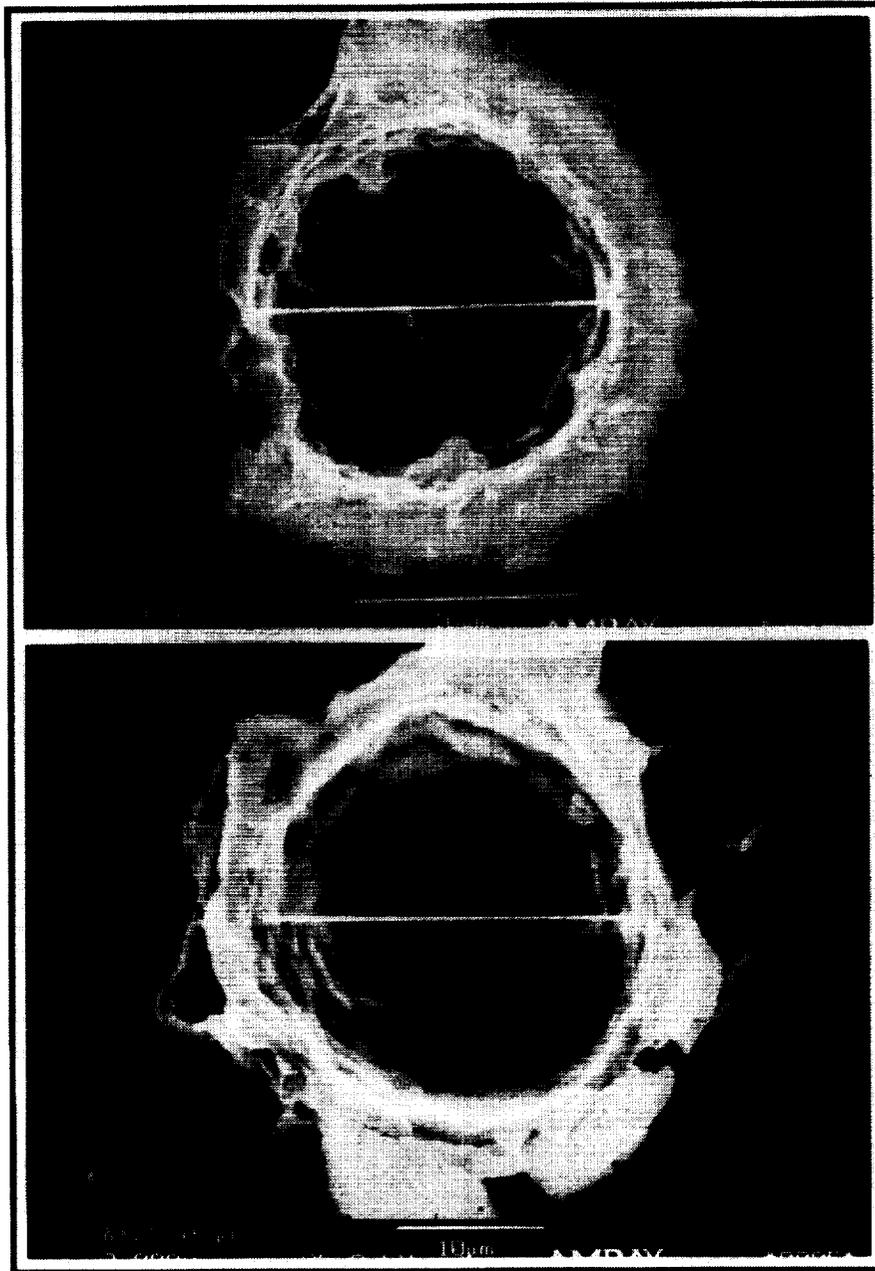


Figure 12. Examples of micrometeorite impact craters in aluminum frame surrounding the leading edge quartz phenolic sample in Fig. 8.

sample shown in Fig. 8. These are the more normal-appearing impact craters observed in metal surfaces [1, 7]. No similar observations were made in the quartz phenolic itself. This is also different from the microparticle impact damage in other composites such as the teflon-fiber glass woven beta cloth which exhibits considerable glass particle shedding and impact-induced glass fiber fragmentation [8].

## SUMMARY AND CONCLUSIONS

Many matrices in fibers in polymeric composites are quite susceptible to the erosive effects of atomic oxygen (AO) in low-Earth orbit. These features were particularly notable in this study for polyimide and polysulphone matrices supporting graphite fibers, as well as phenolics. However, the ash-like erosion products which accumulate on the surfaces of these fiber composites act like a barrier to retard or prevent underlying erosion. Some systems, like quartz phenolic, exhibit less erosion as has been shown previously [4, 6]. The observations suggest that relatively simple coating schemes might be employed to significantly reduce AO erosion even for susceptible polymer composites.

Damage to polymer composites as a consequence of debris particles and micrometeoroids also seems to be less, in most instances, than in metallic surfaces and structural alloys, for example. This feature combined with relatively simple coating applications to reduce AO erosion could have important consequences for a number of polymer composite applications on spacecraft and space structures in low-Earth orbit. However, the synergistic effects of UV, electron irradiation, protons, etc. can certainly complicate specific situations.

## ACKNOWLEDGMENTS

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