NASA SPACE MATERIALS RESEARCH

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INTRODUCTION

Long-term stability within the space environment is a major materials concern for a variety of spacecraft components found on large space antenna systems. For low Earth orbit (LEO) applications, the key environmental variables are atomic oxygen, UV, high vacuum, and thermal cycling. For geosynchronous Earth orbit (GEO) applications, the key variables are high-energy electron and proton radiation, UV, high vacuum, and thermal cycling. For long-life (10 to 20 years) space missions (fig. 1), the durability of thin films, thermal control coatings, and structural composites in the space environment must be established for confident design of economical systems.

This paper will review some of the more recent work on the effect of the space environment on (1) thermal control coatings and thin polymer films, (2) radiation stability of 250°F and 350°F cured graphite/epoxy composites, and (3) the thermal mechanical stability of graphite/epoxy, graphite/aluminum, and graphite/glass composites. Degradation in mechanical properties due to combined radiation and thermal cycling will be highlighted. Damage mechanisms will be presented and suggested chemistry modifications to improve stability will be discussed. The dimensional instabilities in graphite/epoxy composites associated with microcracking during thermal cycling will be examined. Finally, thermal strain hysteresis found in metal-matrix composites will be discussed.

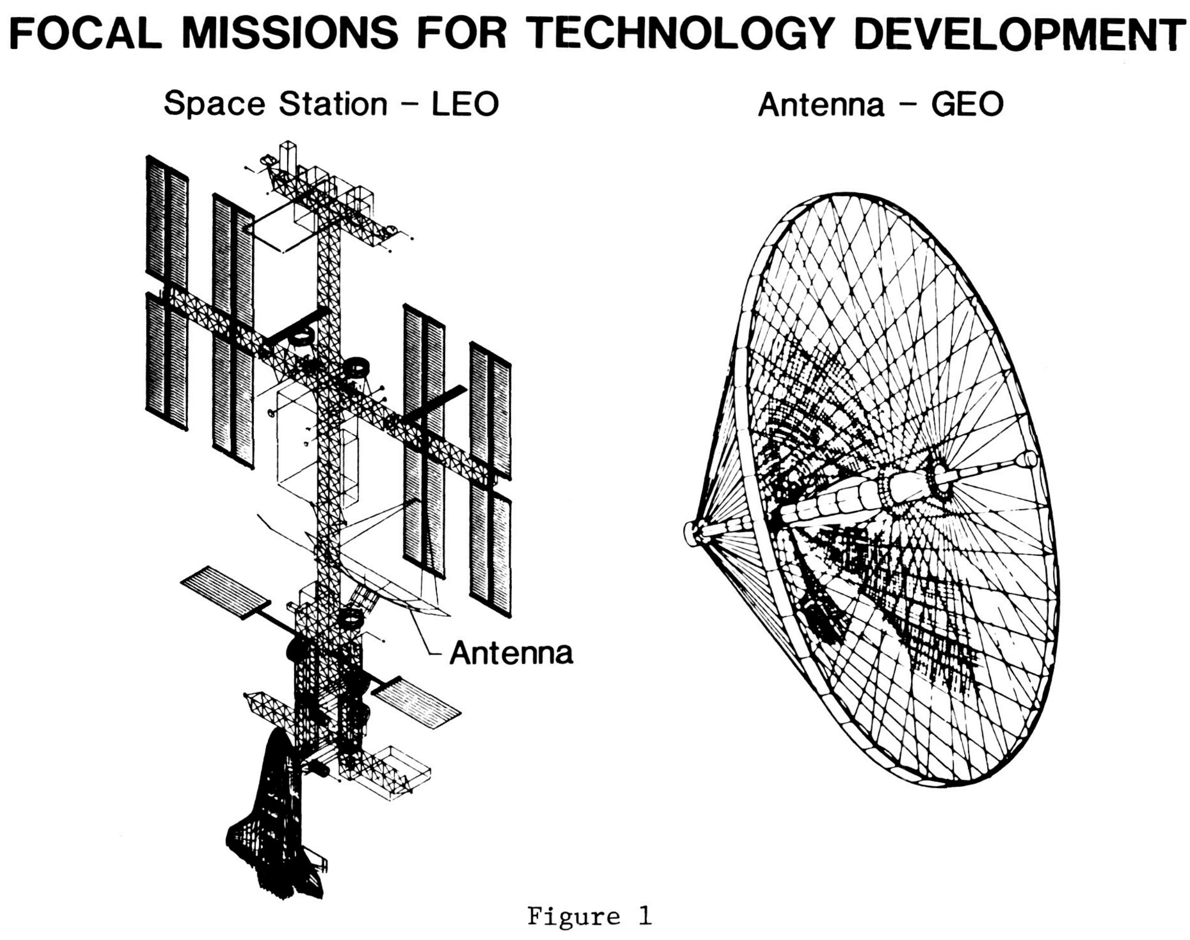

Figure 1

SILOXANE-MODIFIED POLYIMIDE FOR SPACE APPLICATIONS

Early Shuttle flights (STS-3 and STS-5) have demonstrated that the LEO environment could be a significant threat to certain classes of polymeric films and coatings. Polymer films exposed on these early short flights, for example, showed serious surface erosion and corresponding weight losses (ref. 1). Surface and chemical analyses following exposure have attributed the erosion to interaction of atomic oxygen with the material surface. Efforts are now under way to identify new stable materials and/or protection schemes for existing materials that would provide for the materials needs of long-life LEO missions.

A siloxane-modified polyimide copolymer being investigated at Langley (ref. 2) has been found to be less reactive to atomic oxygen than other commonly used spacecraft polymer films (fig. 2). The copolymer incorporates stable siloxane segments into the basic polyimide backbone to provide a unique material that retains general polyimide properties. Characterization of cast films of this material has shown that the siloxane segments of the copolymer migrate to the surface and provide a siloxane "molecular coating" on a basic polyimide substrate. The atomic oxygen stability of this material has been evaluated in ground-based facilities and a recent Shuttle flight (STS-8). In the Shuttle flight, polymer film strips were mounted on a temperature-controlled plate in the cargo bay and received a high flux of atomic oxygen. Following the exposure flight, weight loss analysis showed that the copolymer had about an order-of-magnitude lower reaction rate than the normally used polyimide Kapton (manufactured by E. I. du Pont de Nemours & Co., Inc.). Some erosion of the surface was observed, however, and research on this novel class of copolymers is continuing in an attempt to obtain additional improvements and an understanding of the mechanisms of atomic oxygen interaction with these materials.

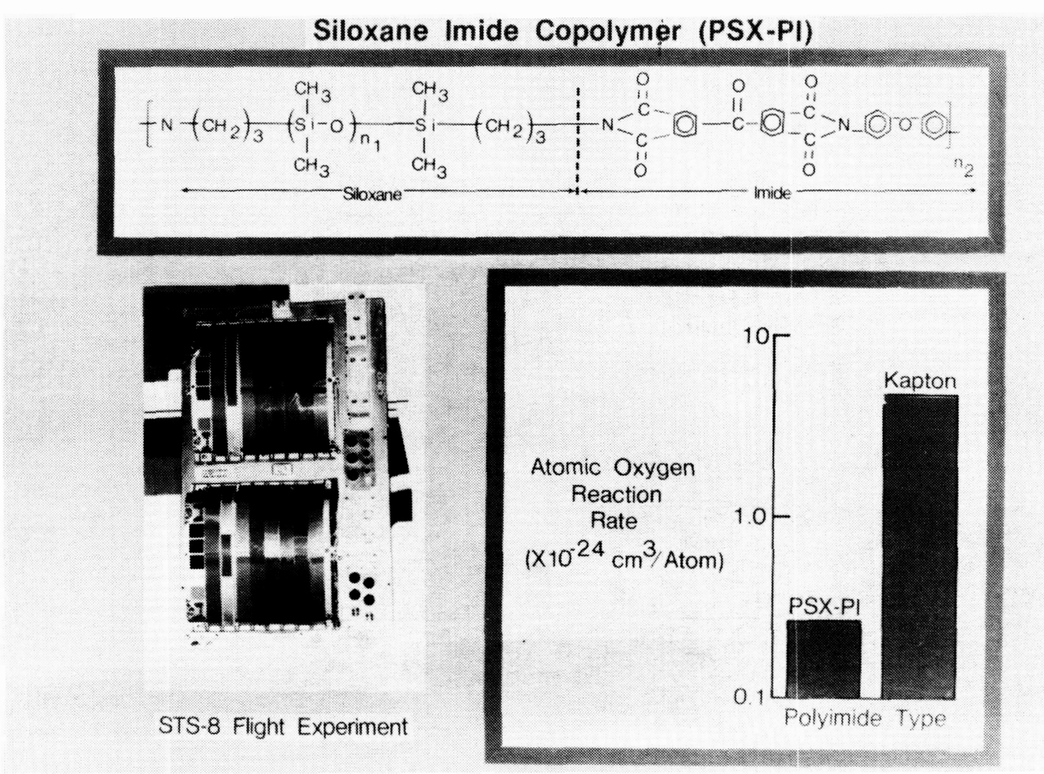

Figure 2

DR-FTIR SPECTRA OF T300/5208 COMPOSITE

The effect of atomic oxygen on the erosion of neat polymer films is well documented, as noted in the previous figure. However, other materials such as composites have important potential uses in low Earth orbit and, therefore, their response to atomic oxygen has also been considered. Figure 3 shows results from characterization (ref. 2) of one material, T300/5208 graphite/epoxy system (manufactured by Narmco Materials, a subsidiary of Celanese Corporation), following exposure to atomic oxygen. This sample was flown on the STS-8 flight experiment (225-km orbit) where it was exposed for 41 hours (oxygen impingement normal to surface) and received a total oxygen fluence of 3.5×10^{20} atoms/cm^2. The figure shows diffuse reflectance infrared spectra of the control sample (covered during the flight) and spectra of both front and rear surfaces of the exposed specimen. The rear surface of the exposed specimen was shielded and not directly exposed during the flight. The data show that major chemical degradation does not occur during atomic oxygen exposure; that is, the exposed sample has generally identical major absorption bands as the control specimen. Several of the major bands are identified in the figure. However, the spectra do show, as evidenced by the appearance of the $>C = O$ absorption band near 1700 cm^{-1}, that oxidation is the primary mechanism of atomic oxygen interaction with the composite matrix. This oxidation is accompanied by an erosion very similar to that observed in the neat polymer films. The IR spectrum of the back surface of the composite also indicated oxidation of the polymer. The composites were mounted on an aluminum plate but were not sealed. The mechanism of oxidation of the back surface is not known. These data therefore indicate that use of current advanced composite systems, such as the T300/5208 system for LEO applications, will require development of stable atomic oxygen resistant coatings.

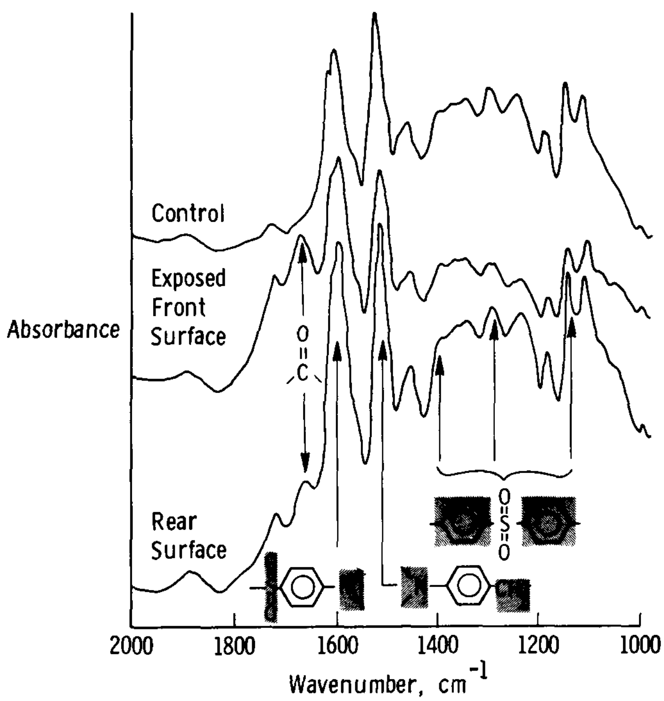

Figure 3

COMPOSITE COATING DEVELOPMENT

Graphite/epoxy composite tubes are being considered for use in space antenna structures because of their high specific strength and stiffness and good dimensional stability. Composites used in space need to be coated to protect the resin from UV degradation, atomic oxygen erosion (LEO applications), and low-energy electron and proton degradation for GEO applications. Coatings are also needed to minimize the temperature range over which composites will cycle in space. The upper left portion of figure 4 shows the range of temperatures that composite tubes will cycle over with (1) no coating, (2) white paint, and (3) aluminum coating. Also shown is the temperature range that a composite tube would cycle over if the coating had a solar absorptance (α) of 0.20 to 0.35 and emittance (ε) of 0.15 to 0.25.

Several experimental coatings have been developed (by W. S. Slemp, NASA Langley) which give optical properties in the desired range. Three of these are shown at the bottom of figure 4. These coatings were all produced by vapor depositing thin layers of metals and metal oxides to form multilayer coatings with the required combination of low α (0.20 to 0.35) and low ε (0.15 to 0.25). The thicknesses of the individual layers are a few thousand angstroms and were selected to achieve the optical properties shown. Aluminum, SiO_2, and Al_2O_3 have excellent resistance to atomic oxygen and should protect the composite substrate for LEO applications. These materials also have good stability to electron, proton, and UV radiation. Space qualification testing of these coatings is under way and, if they prove to have sufficient durability, alternate application techniques will be investigated to economically coat full-size structural subelements.

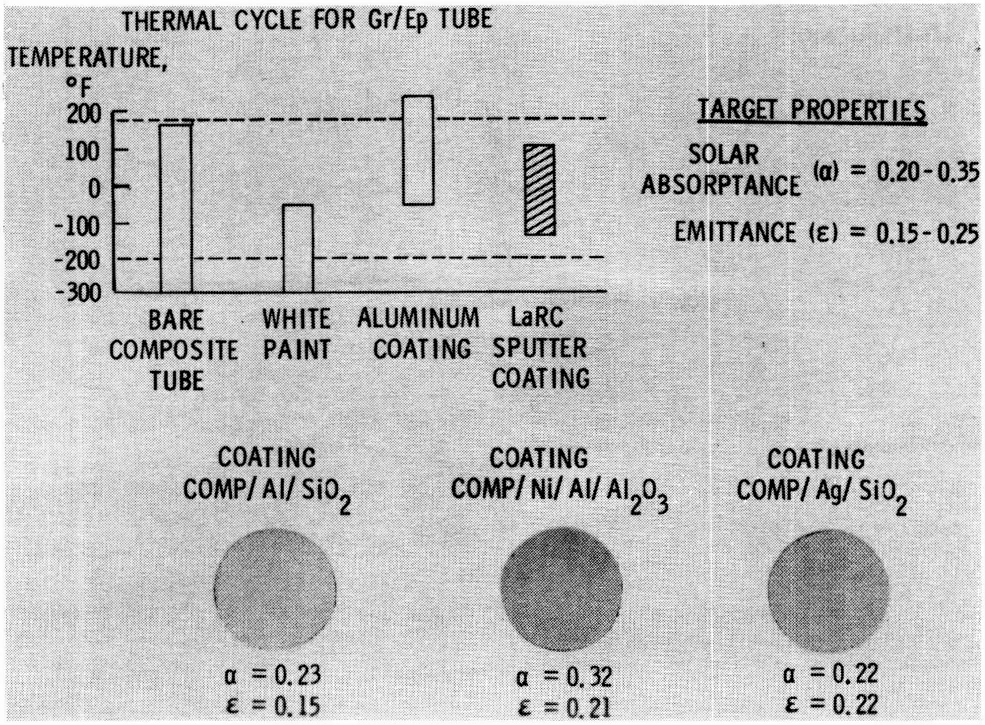

Figure 4

UV-VIS SPECTRA OF COLORLESS FILMS VERSUS KAPTON

A series of novel, optically transparent aromatic polyimides were synthesized to obtain films with maximum optical transparency for use in space as second surface mirror coatings (ref. 3). Color in the films was lowered by reducing the electronic interaction between chromophoric centers in the polymer chemical structure and by monomer purification procedures. The percent transmission of these films compared to Kapton in the UV-visible wavelength range is shown in figure 5. At the solar wavelength (500 nm), for space application on solar cells and thermal control systems, Kapton is approximately 40 percent transparent (depending on thickness) and the linear aromatic films are approximately 85 to 90 percent transparent. Also, some of these polyimides have potential for spray coating applications because they are soluble in low-temperature boiling solvents. Both UV and electron irradiation exposures will be conducted to evaluate the stability of these films to the space radiation environment.

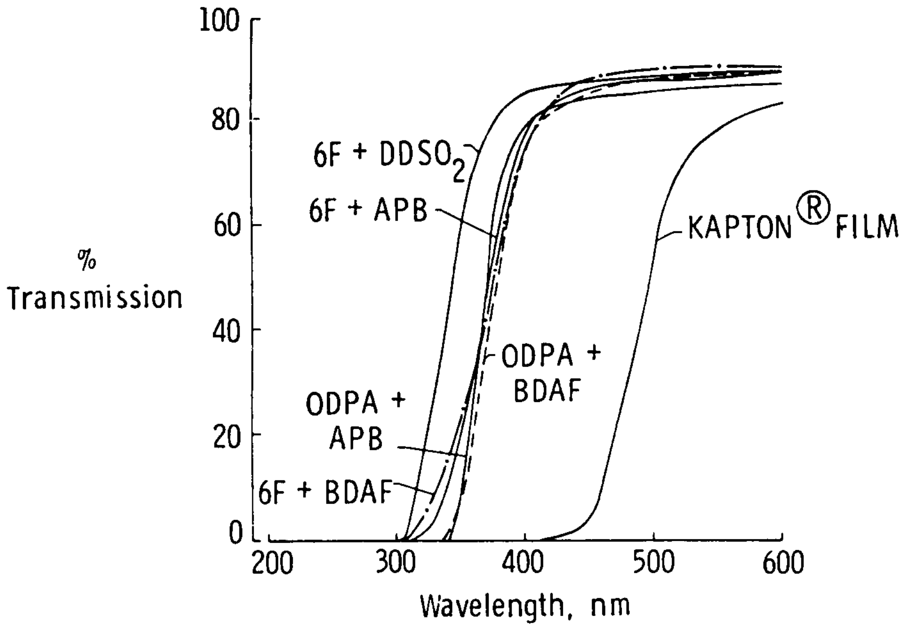

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Figure 5

RADIATION STABILITY OF POLYMERS AND COMPOSITES

Spacecraft placed at GEO will be subjected to high doses of electron and proton radiation. For long-term exposures (20 to 25 years), the total absorbed radiation dose in resin-matrix composites will exceed 10^9 rads and changes in matrix-dominated properties are likely.

NASA has been conducting research to determine the magnitude of property changes in composites when subjected to high doses of electron and proton radiation. This work has been conducted at Langley Research Center, at Jet Propulsion Laboratory, and at Marshall Space Flight Center. Emphasis has been placed on (1) determining the bounds on changes in mechanical properties, (2) measuring the matrix embrittlement of 250°F and 350°F cure epoxies resulting from radiation exposure, (3) studying the microcracking behavior during thermal cycling, and (4) establishing a fundamental understanding of damage mechanisms to guide future new materials development. Some of the recent results obtained in the areas shown in figure 6 are presented in figures 7 through 15.

- Mechanical property changes
- Matrix embrittlement
- Damage mechanisms

Figure 6

TRANSVERSE STRENGTH AND STIFFNESS FOR BASELINE AND IRRADIATED T300/934 GRAPHITE/EPOXY COMPOSITES

Several state-of-the-art advanced graphite/epoxy composite systems have been evaluated in the NASA programs to determine their response to the GEO environment. Figure 7 shows typical mechanical property data from a recent investigation (ref. 4). The transverse strength and modulus of T300/934 graphite/epoxy (manufactured by Fiberite Corporation) before and after radiation are shown for three different test temperatures. The irradiated samples received an electron dose of 1×10^{10} rads at a dose rate of 5×10^{7} rads per hour in the Langley radiation exposure facility. The total radiation dose simulates a worst case, 20- to 25-year GEO radiation exposure.

The data suggest that electron irradiation tends to embrittle this 350°F cured composite system. The stiffness of the irradiated specimens was greater than that of the baseline system in the room temperature and lower temperature tests. Transverse strength was also lower through this temperature range. Above room temperature, the stiffness of the irradiated material was slightly lower than that of the baseline material. Little or no change was found in the fiber-dominated tensile properties.

Other research on the chemistry of electron interaction with this epoxy resin has shown that some degradation products are produced and these products effectively plasticize the matrix in the elevated temperature test, thus giving lower stiffness. A corresponding lower glass transition temperature was also found for the irradiated matrix material.

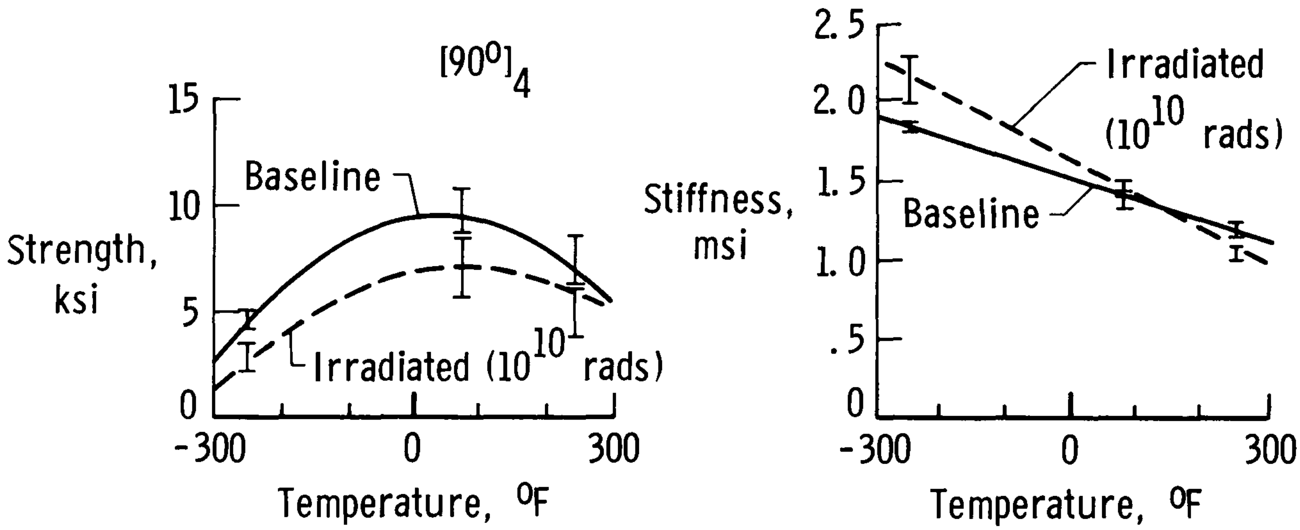

Figure 7

EFFECTS OF ELECTRON RADIATION ON CRITICAL STRAIN-ENERGY-RELEASE RATES OF T300/934 GRAPHITE/EPOXY

As noted previously, evaluation of the fiber-dominated tensile properties following electron irradiation (1 x 10^{10} rads) generally did not show differences between the irradiated and baseline material. However, because the matrix-dominated behavior $[90]_4$ indicated epoxy embrittlement, a series of tests were conducted (by J. Funk, NASA Langley) on the effect or radiation on the interlaminar fracture toughness of the T300/934 graphite/epoxy system. The results of these tests are given in ure 8. Two experimental test specimens were evaluated. Miniature specimens for both a double-cantilever-beam (DCB) test and an edge-delamination test (EDT) were used to determine the change in critical strain-energy-release rate (G_c) with radiation exposure. The DCB test used a unidirectional specimen 3 in. by 0.2 in. and the EDT used a 5 in. by 0.5 in. specimen. The irradiated specimens received 1 x 10^{10} rads in the Langley radiation exposure facility. In the DCB test the specimen is subjected to peel stresses (mode I) and in the EDT the specimen is subjected to a mixed-mode state of stresses, peel (mode I) and shear (mode II). In both experiments (DCB and EDT), the strain-energy-release rate for the irradiated specimens was equal to or slightly greater than that of the baseline material, indicating that radiation did not degrade the toughness of the exposed composites. However, radiation does cause matrix embrittlement which leads to a much higher density of microcracks in the composite after thermal cycling (see fig. 12). Therefore, additional fracture toughness testing has been initiated to evaluate samples after combined radiation and thermal cycling.

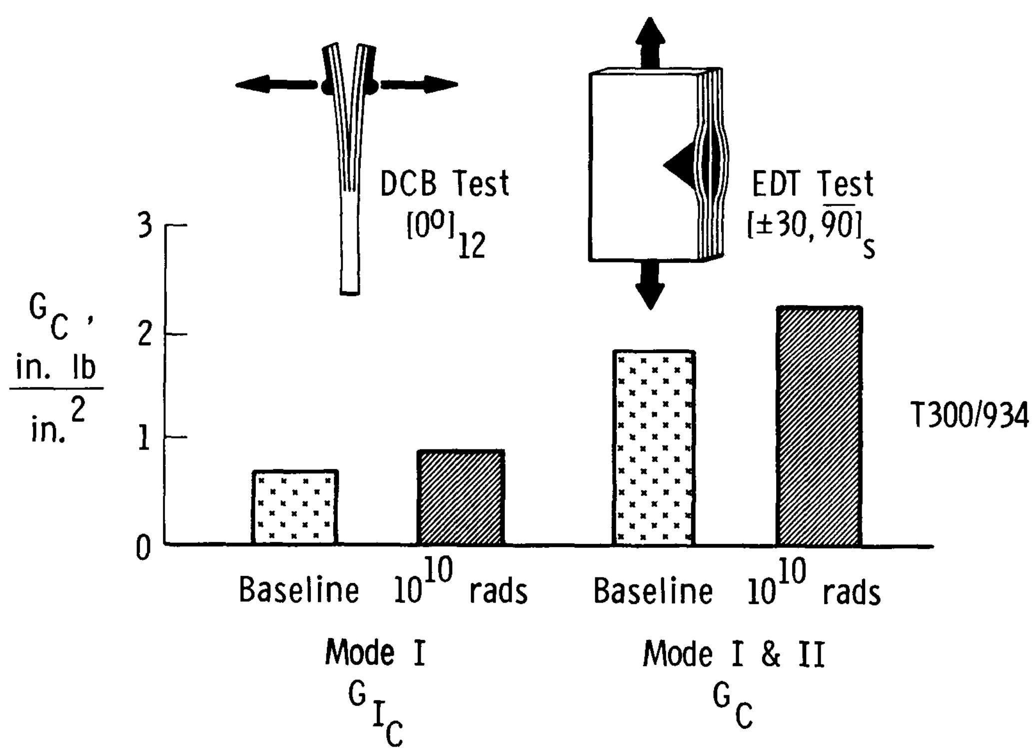

Figure 8

EFFECTS OF RADIATION ON THERMAL EXPANSION

A primary requirement of composite materials for precision space structural applications is that they be dimensionally stable and maintain this stability during their use life. For GEO applications, radiation and repeated thermal cycles are parts of the exposure environment that may affect the stability of composite materials. Figure 9 shows the effect of penetrating electron irradiation and a single thermal cycle on the expansion characteristics of the T300/5208 graphite/epoxy system. The thermal expansion of the baseline and irradiated (6×10^9 rads) laminates $[0_2/90_2]_s$ is shown through the temperature range from -250°F to 250°F. The thermal cycle involved heating from room temperature to 250°F and then cooling to -250°F followed by heating to room temperature. Although only one cycle is shown, the baseline laminate could be cycled repeatedly through this cycle without deviation from the expansion curve shown. The irradiated samples, however, upon reaching about 200°F on the first cycle, displayed a significant change in CTE that continued to the upper temperature of the test (250°F). During cooling to the lower test temperature, the sample took on a permanent set of about 100 microstrain, but displayed no change in CTE. Further cycling did not produce any further changes. These data indicate that the elevated temperature properties of the composite were probably modified by the electron irradiation (see fig. 10). A more complete analysis of the effects of radiation on dimensional stability of this 350°F system is provided in reference 5.

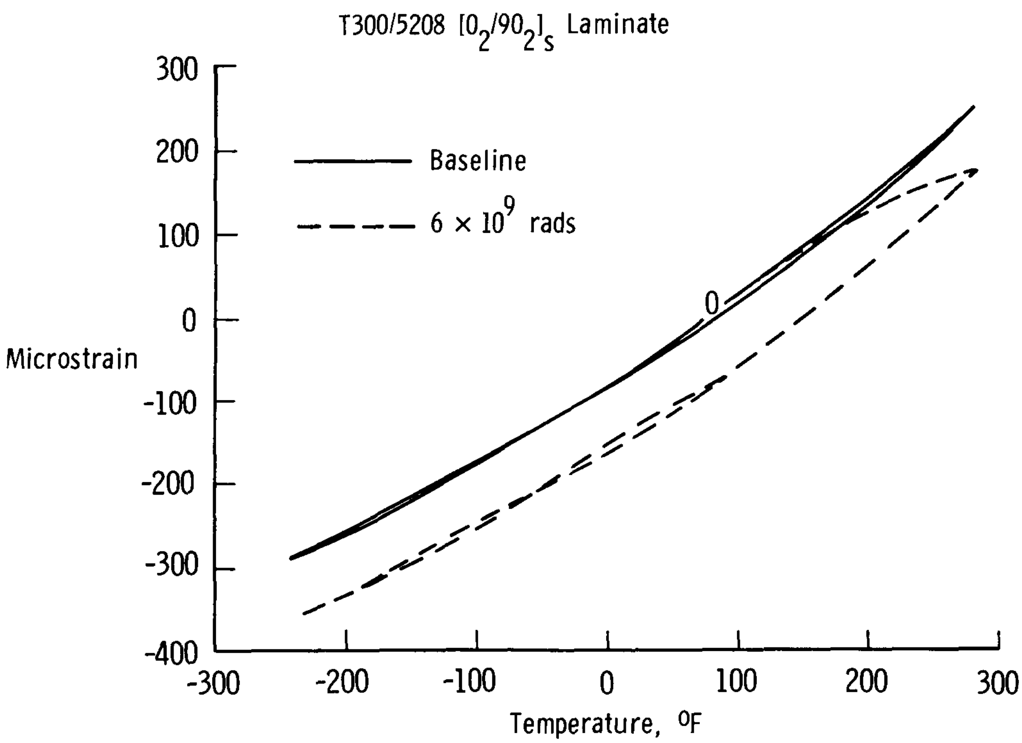

Figure 9

DYNAMIC MECHANICAL ANALYSIS OF T300/5208 GRAPHITE/EPOXY

Figure 10 shows the dynamic mechanical analysis of the T300/5208 baseline and irradiated laminates discussed in figure 9. The upper part of this figure shows the change in dynamic modulus with temperature and the lower part shows the loss modulus as a function of test temperature for both laminates. Electron irradiation lowers the glass transition temperature (T_g) of the epoxy about 50°F. Also, the "rubbery range" associated with the T_g is lowered in temperature and broadened so that it extends into the test range of the thermal expansion test. The dynamic modulus also shows the lower temperature of softening following irradiation. These data indicate that the change in slope of the expansion curve (fig. 9) for the irradiated material reflects a softening of the matrix and the expansion characteristics of the laminate take on the expansion behavior of the fibers. The lowered glass transition temperature and related rubbery range indicate that radiation has an effect upon the chemistry of the 5208 epoxy matrix. Because major changes in tensile properties and toughness have not been observed, the basic epoxy structure is assumed to remain essentially intact following radiation exposure.

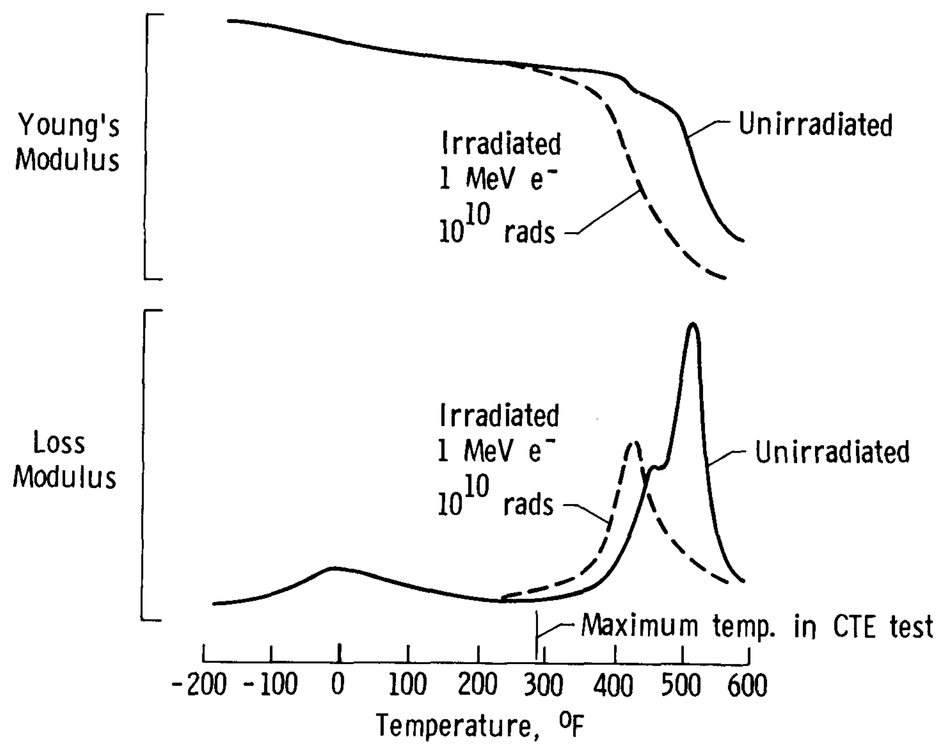

Figure 10

EFFECT OF RADIATION AND THERMAL CYCLING ON MICROCRACK FORMATION IN T300/934 LAMINATES [0/90/90/0]

Composite materials that are used in GEO applications will be exposed to a combined environment consisting of radiation, vacuum, and thermal cycles that will repeatedly raise and lower the material temperature during exposure. The effect of these temperature excursions during irradiation has not been determined, but a series of sequential (radiation followed by thermal cycles) exposures on the T300/934 graphite/epoxy system have been performed in an effort to obtain an initial assessment of the combined effects on this material. The results of this study are shown in figures 11 and 12. Figure 11 shows X-ray photographs of 4-ply [0/90/90/0] laminates before exposure, after 500 thermal cycles, after 500 cycles and irradiation (10^{10} rads), and after irradiation followed by 500 thermal cycles. In each case the thermal cycles consisted of cycling the specimen between -250°F and 250°F using a 20-minute cycle period. As shown in these photographs, the baseline and thermal-cycled-only specimens showed only a few microcracks in the [0] direction. The specimen that was thermal cycled and then irradiated also exhibited no increase in microcrack density and the cracks that were present extended in the [0] direction. However, the laminate that received radiation followed by 500 thermal cycles microcracked extensively. Cracks were noted in both the 90° and 0° plies for this latter exposure sequence. No 90° cracks were found near the end of the specimen where the clamp covered the specimen during the radiation exposure.

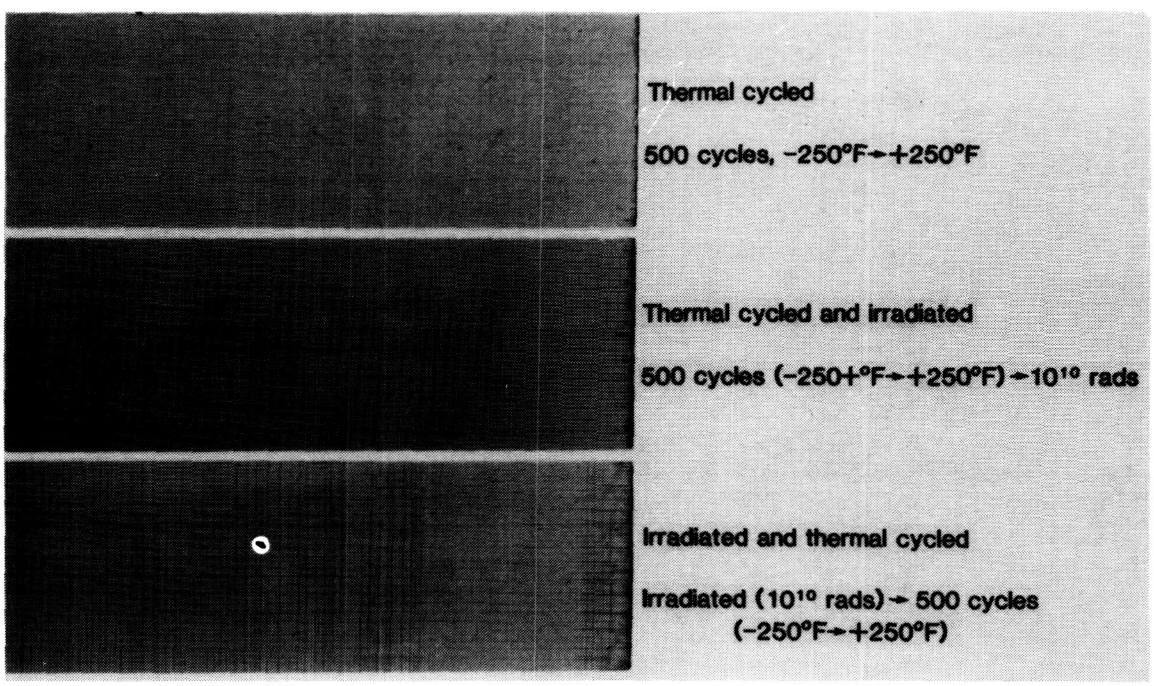

Figure 11

EFFECT OF RADIATION ON MICROCRACK FORMATION IN A 350°F CURED EPOXY COMPOSITE

Figure 12 presents, in bar graph form, the crack densities observed in the micrographs of the samples presented in figure 11. For the thermal-cycled-only specimen, the crack density was about 25 cracks/inch. For the specimen that was thermal cycled after receiving the radiation, the crack density was about 75 cracks/inch. The high density of microcracks suggests that the radiation exposure caused significant embrittlement of the matrix. The effect of crack densities of this magnitude on the thermal expansion characteristics of this epoxy system will be discussed in conjunction with the results presented in the section entitled "Effect of Thermal Cycling on Microcrack Density."

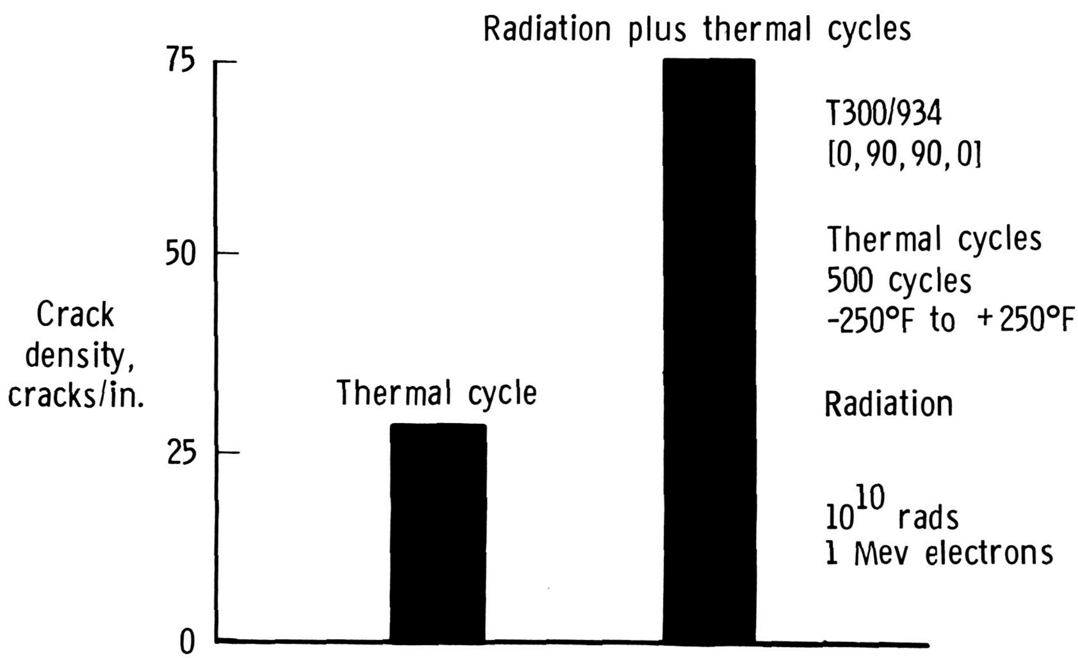

Figure 12

RADIATION EFFECTS ON THE TENSILE PROPERTIES OF T300/CE339 $[0]_4$
(1 MeV ELECTRONS AT 5 x 10^7 RADS/HR)

Radiation effects studies of advanced state-of-the-art aerospace 350°F cured graphite/epoxy composites show that this class of materials is generally embrittled by radiation doses equivalent to 20 to 25 years in GEO. This embrittlement results in extensive microcracking during thermal cycling (fig. 12). Because microcracking can significantly limit the usefulness of composites in precision space structures, toughened matrix material systems with lower initial crosslink density are being considered. Figure 13 shows results from an evaluation of the radiation effects on a system of this type (ref. 6). The material evaluated was the space-qualified T300/CE339 system, manufactured by Ferro Corporation, which has a 250°F cured elastomer-toughened epoxy matrix. The elastomer is assumed to be of the CTBN family frequently used to toughen epoxy resins. The figure shows the ultimate tensile strength of a unidirectional $[0]_4$ baseline and irradiated specimen tested at room temperature. The irradiated specimen received 1 x 10^{10} rads at 5 x 10^7 rads/hour using 1 MeV electrons. The ultimate tensile strength of the irradiated fiber-dominated specimen was about 50 percent of that observed for the baseline composite. The fractured specimen for each material, baseline and irradiated, was also significantly different. The baseline sample fractured, leaving relatively large broken fiber/matrix bundles. The irradiated sample left a single "fluffy" bundle of individual graphite fibers, indicating that the brittle matrix shattered at failure. Examination of individual fibers in the "fluffy" bundle of the irradiated specimen showed that little, if any, resin remained on the fiber surface after fracture. These data indicate that this 250°F elastomer-toughened system is more sensitive to radiation than the comparable 350°F cured epoxy systems and thus could not be considered for long-life use in a GEO environment.

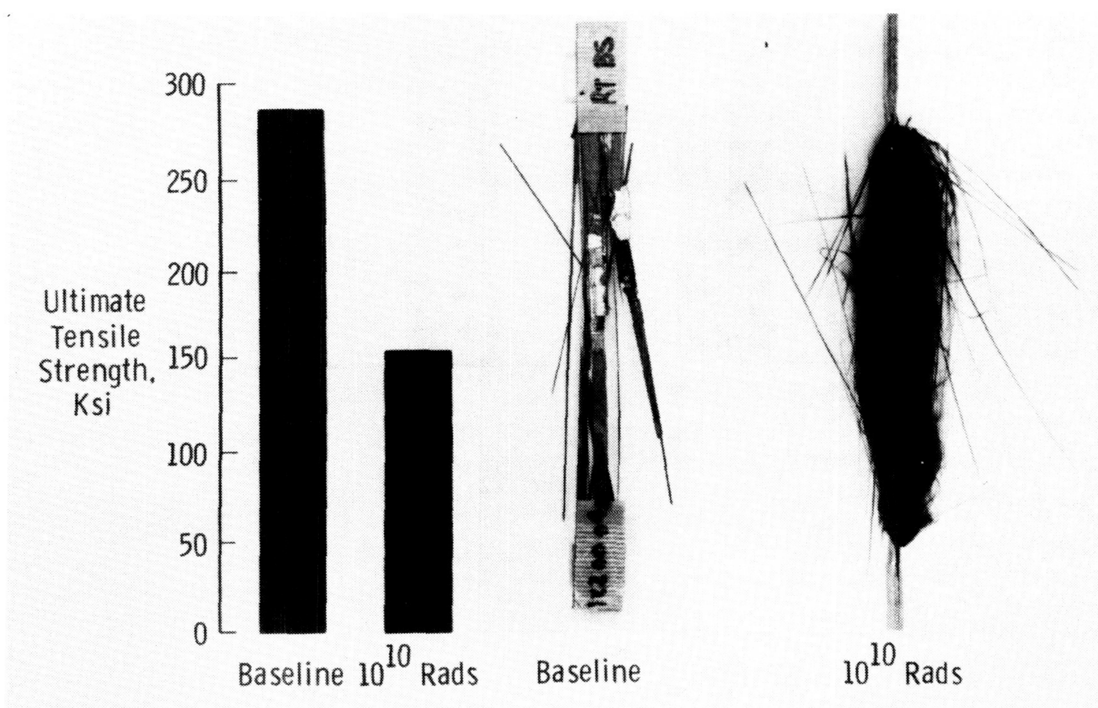

Figure 13

EFFECT OF RADIATION TOTAL DOSE ON MICROCRACK FORMATION IN THERMAL-CYCLED T300/CE339 COMPOSITES

Although it has been shown in the previous figure that the T300/CE339 elastomer-toughened graphite/epoxy composite system is sensitive to high doses of radiation (10^{10} rads), there are spacecraft materials applications that require high dimensional stability for relatively low-radiation-intensity orbits. A toughened epoxy system such as the CE339 matrix could be considered for these applications. Figure 14 shows the effect of total radiation dose and thermal cycling on microcrack formation in this epoxy system (ref. 6). The crack density is given for the baseline material and specimens that received electron doses between 1×10^7 and 1×10^{10} rads and then thermal cycled 1, 10, and 100 times between -200°F and 175°F. As shown, no microcracks were found in the baseline material after thermal cycling, indicating that this epoxy system is more resistant to microcracking than similar 350°F cured epoxy systems. However, after irradiation all samples microcracked, even those that received the lowest total dose of 1×10^7 rads. This shows that matrix crosslinking and embrittlement began at a low radiation total dose. At total doses greater than 1×10^8 rads, microcracking was extensive, and at 1×10^{10} rads the crack density was equivalent to that observed in irradiated and thermal-cycled 350°F cured epoxy systems. Chemical characterization of this CE339 system (ref. 6) indicates that the low-radiation-dose embrittlement is related to crosslinking of the CTBN type elastomers. This suggests that the radiation stability of the system might be improved by substituting a more radiation stable elastomer for the CTBN.

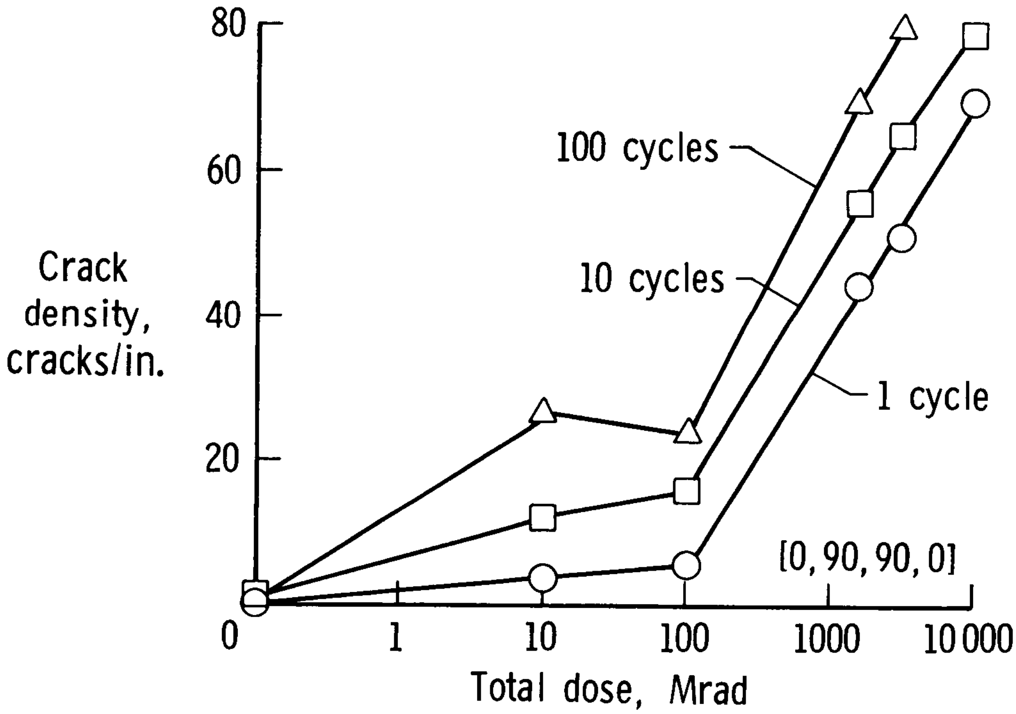

Figure 14

RADIATION DAMAGE MECHANISMS IN A THERMOPLASTIC MATERIAL

Ultem polyetherimide is a relatively new thermoplastic polyimide manufactured by the General Electric Company. This polymer is one of several recently developed advanced thermoplastic systems that has potential for space applications. To assess the durability of Ultem for possible long-term (20 to 25 years) use in the space environment (GEO), an experimental study of its radiation stability was conducted (ref. 7). Film samples (0.003 in. thick) were irradiated with 100 keV electrons in vacuum and given radiation doses of 1.6, 4.0, and 6.0 x 10^9 rads. Following the radiation exposure, changes in mechanical properties (tensile), radical concentration (EPR), and molecular structure (IR) were determined. Figure 15 provides a summary of this characterization.

Only small changes in tensile stress and modulus were observed following the radiation exposure. Elongation, however, was significantly reduced. Solubility of the baseline and irradiated films indicated that radiation-induced crosslinking was extensive.

Electron paramagnetic resonance (EPR) spectroscopy and infrared (IR) spectroscopy were performed to determine changes in the chemical structure of the polymer. The density of organic radicals (spins/gm) increased by over two orders of magnitude at 4 x 10^9 rads. Four distinct radical species were identified: phenoxyls, gem-dimethyls, ketones, and cyclohexadienyls. Analyses of the EPR and IR spectra indicated that radiation caused dehydrogenation of methyl groups and rupture of ether linkages. At the highest dose the imide ring was opened. At least three of the four radical species observed led to crosslinking between the linear chains.

Polyetherimide (Ultem)

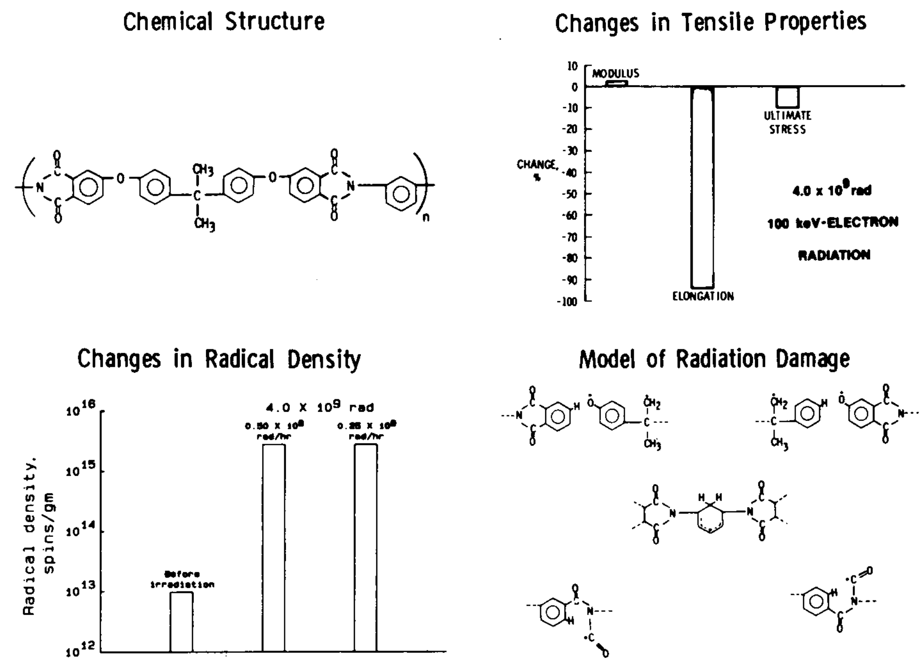

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Figure 15

THERMAL MECHANICAL STABILITY OF COMPOSITES

Graphite-reinforced composite materials are leading candidates for construction of precision space structures because of their high specific stiffness and low coefficient of thermal expansion (CTE). In addition to low CTE, good dimensional stability is required for precision antenna structures where maintaining a preset surface contour is critical to antenna performance. Small dimensional changes in the antenna structure can cause defocusing of the antenna or an increase in the surface roughness of the reflector surface. Dimensional changes in resin-matrix composites can be caused by thermal expansion, loss of moisture, microcracking of the resin, applied stress, and radiation damage to the resin matrix. For metal- and ceramic-matrix composites, thermal cycling and mechanical loading can cause dimensional changes.

A research program is under way at NASA Langley to characterize and model the dimensional stability of low expansion composites of interest for precision antenna structures. Emphasis has been placed on development of laser interferometry measurement techniques and characterization of thermal cycling damage to both resin- and metal-matrix composites. Some of the recent results obtained in the areas listed in figure 16 will be presented in the following figures.

- Laser interferometer technique
- Thermal cycling damage development
- Thermal strain hysteresis in Gr/metals and Gr/glass

Figure 16

SCHEMATIC DIAGRAM OF LASER INTERFEROMETER DILATOMETER SYSTEM

A Fizeau-type, laser interferometer dilatometer system has been developed to characterize the thermal expansion of low expansion composite materials (fig. 17). This system, described in reference 8, consists of (1) a Fizeau interferometer, (2) an environmental chamber, (3) an optical train, (4) a He-Ne laser, and (5) a camera. A photodiode array, a waveform analyzer, and a microcomputer have also been incorporated to automate the testing and data reduction process. The entire system is automated with the computer controlling the temperature in the environmental chamber, triggering the camera, recording data from the waveform analyzer, and then incrementing the temperature to cover a preselected temperature range. The thermal expansion of the specimen is measured relative to the thermal expansion of a known reference material. The current reference material is fused silica, calibrated by the National Bureau of Standards. The strain resolution with this system is less than 1 microstrain.

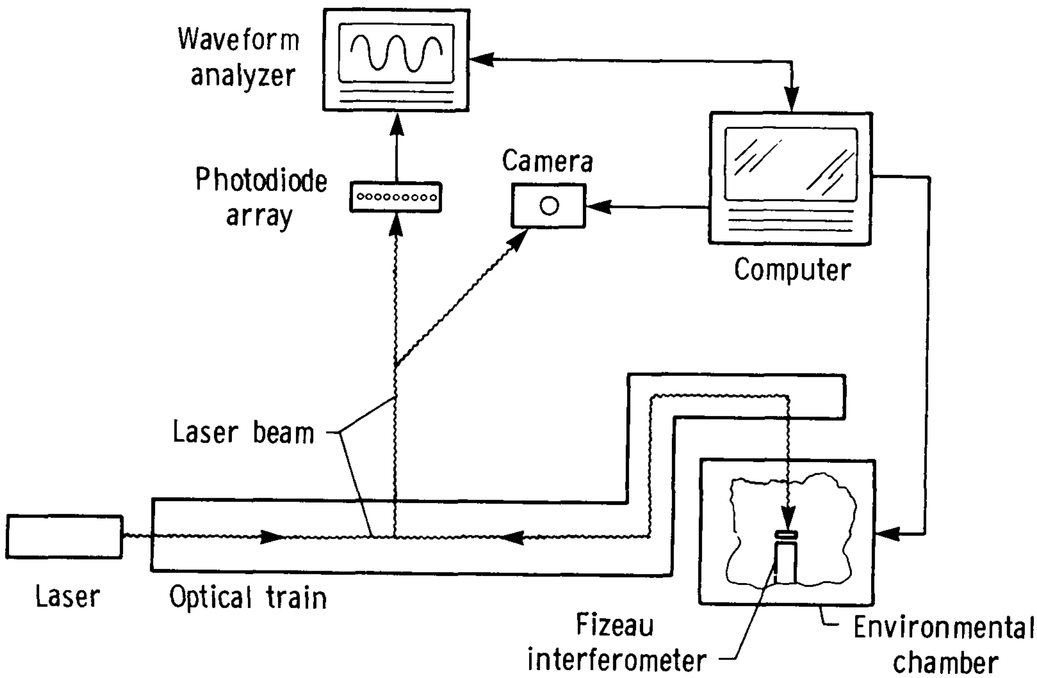

Figure 17

EXPANSION OF INVAR 36

The laser interferometer system described in figure 17 is used for precision measurement of small dimensional changes in composites. The change in length of a test specimen is directly proportional to the change in the light interference pattern, the fringe density. Two methods are used for recording and analyzing fringe data. In the first method, individual fringe patterns are recorded on 35-mm film. At the conclusion of a test, the 35-mm negatives are enlarged in a microfiche reader and the fringes are visually counted over a defined gauge length. The fringe density is the number of fringes divided by the gauge length. The second method allows fringe patterns to be analyzed in real time. The fringe pattern is imaged onto a light-sensitive linear photodiode array. The photodiode array is electrically scanned and the fringe pattern is digitized. The digitized signal, which has a sinusoidal form, is then transmitted to a waveform analyzer. The waveform analyzer performs a fast Fourier transform on the signal to determine the frequency content. The predominant frequency in the signal is the sine wave frequency, from which the fringe density can be directly computed.

A comparison of thermal expansion data computed from 35-mm film and from the fast Fourier transform analysis is shown in figure 18. The data shown are for one test of an Invar 36 specimen (ref. 8). The two sets of data are in good agreement, with a maximum difference of 4.7 microstrain. The majority of this difference is caused by a misalignment of the fringe pattern image on the photodiode array. A method to more accurately align the fringe pattern on the photodiode array is currently under development.

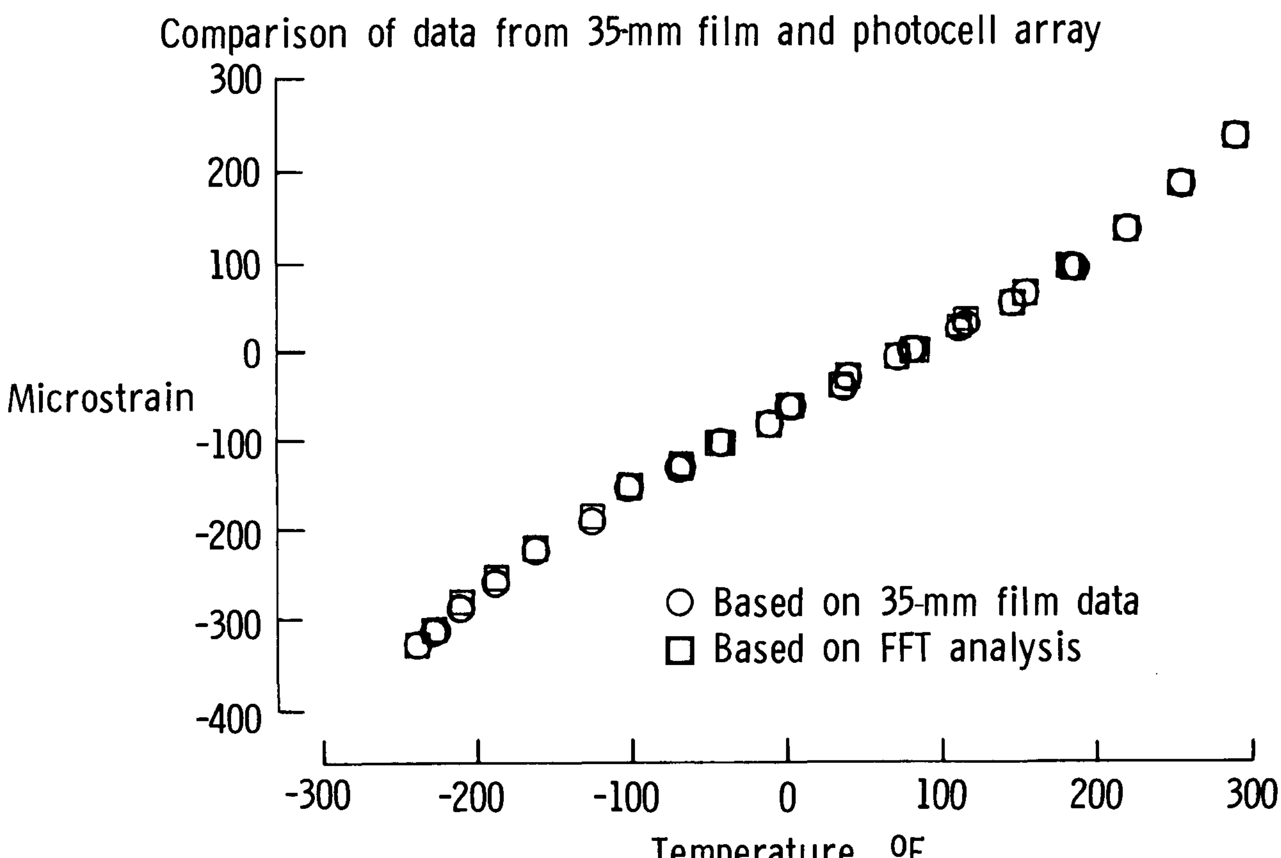

Figure 18

THERMAL EXPANSION OF GRAPHITE/POLYIMIDE COMPOSITE LAMINATES (C6000/PMR-15)

The laser dilatometer system previously described has a strain resolution of less than 1 microstrain and thus can be used to measure the expansion behavior of low expansion unidirectional composite laminates. However, higher expansion laminates such as quasi-isotropic layups where the total strain range may be several hundred microstrain can also be examined. The versatility of this dilatomer is illustrated by the data presented in figure 19. Expansion data are presented for an 8-ply unidirectional laminate and a quasi-isotropic laminate of graphite/polyimide composite, C6000/PMR-15, composed of Celion 6000 graphite fibers (Celanese Corporation) and PMR-15 polyimide resin. The total expansion of the unidirectional laminate was relatively small, about 80 microstrain, over the temperature range -250°F to 250°F. The total expansion of the quasi-isotropic laminate was about 475 microstrain over the same temperature range.

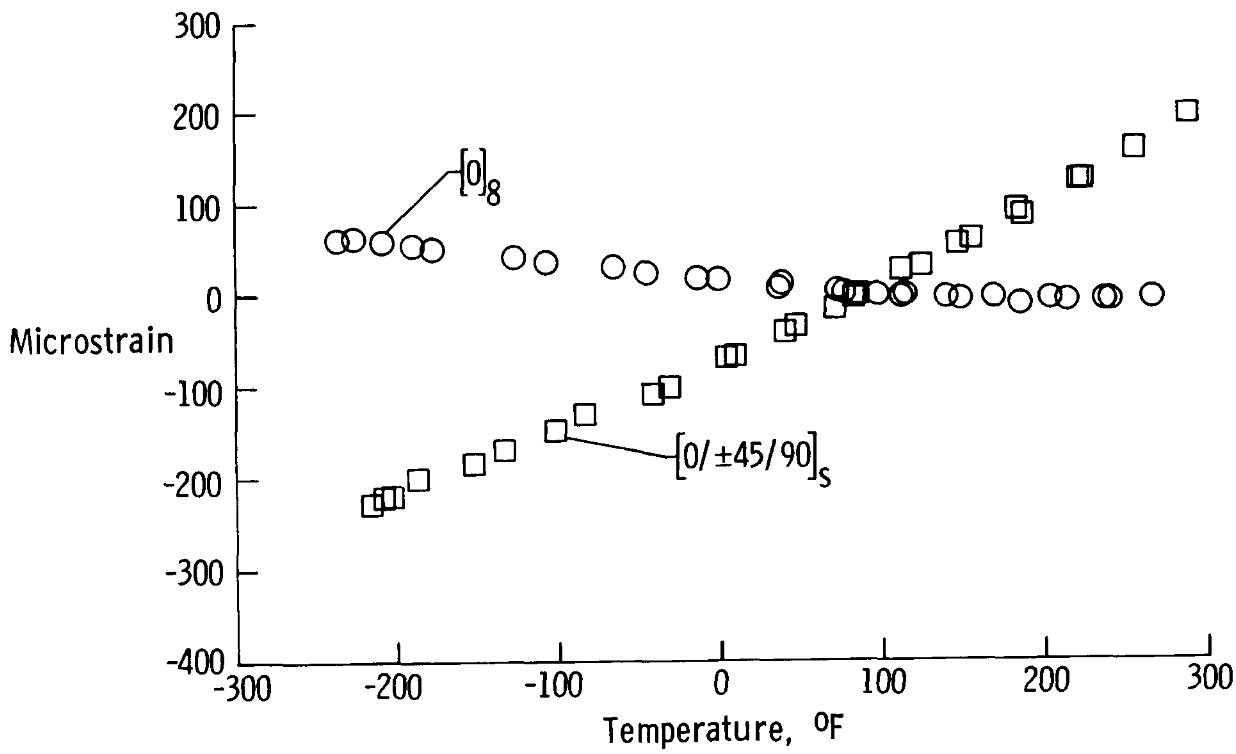

Figure 19

EFFECT OF THERMAL CYCLING ON MICROCRACK DENSITY

The room-temperature coefficient of thermal expansion (CTE) of T300 graphite fibers along the axis of the fibers is approximately -0.3×10^{-6} in/in/°F while 5208 epoxy resin has a CTE of approximately 30 to 32 x 10^{-6} in/in/°F at room temperature. This large difference in expansion behavior gives rise to high residual stresses being developed in graphite/epoxy composites when they are cooled from the processing temperature (350°F for T300/5208) or during any subsequent thermal cycling. The state of stress can be particularly high in cross-ply laminates, such as $[0_2/90_2]_s$, where a very low expansion [0] ply is bonded to a high expansion [90] ply. Stresses can reach sufficient magnitude to cause microcracking of the brittle epoxy resin.

Data presented in figure 20 show that for a $[0_2/90_2]_s$ laminate of T300/5208 cycled between -250°F and 250°F the number of cracks per inch increases with the number of thermal cycles. The crack density was 15 cracks/inch after 500 cycles and is expected to reach an equilibrium value between 20 and 22 cracks/inch after several hundred more cycles.

The equilibrium density estimated from a finite-element analysis (ref. 9) of microcracking in this composite system is shown by the dashed line on the figure (about 22 cracks/inch). This analysis appears to reasonably predict an upper bound on crack density due to thermal cycling.

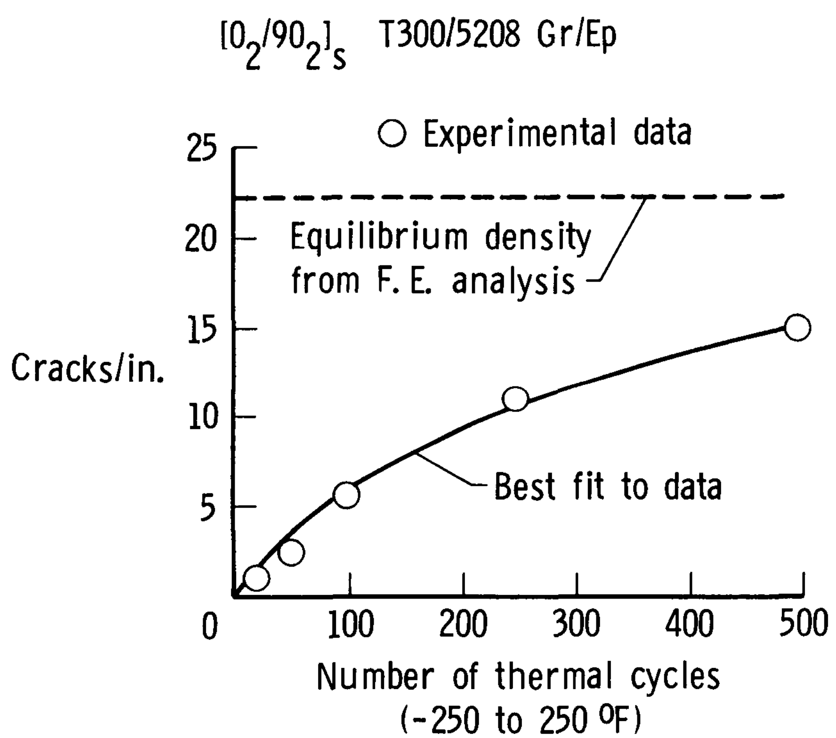

Figure 20

EFFECTS OF MICROCRACKS ON COEFFICIENT OF THERMAL EXPANSION OF $[0_2/90_2]_s$ T300/5208 GRAPHITE/EPOXY

Results from an analytical study of the effects of microcracks on the coefficient of thermal expansion (CTE) of $[0_2/90_2]_s$ T300/5208 graphite/epoxy (ref. 9) are shown in figure 21. The analytical results were obtained by using a finite-element analysis in conjunction with classical lamination theory (CLT) to model the effects of microcracks on the CTE of the laminate. The analysis shows, for a crack density of 75 cracks/inch in the 90° plies of a $[0_2/90_2]_s$ laminate, the CTE in the [0] fiber direction is reduced to 30 percent of its original value. For cracks in both the 0° and 90° plies at this same crack density, the CTE is reduced to 13 percent of its original value. Therefore, the addition of cracks in the 0° plies does affect CTE, but to a much lesser extent than cracks in the 90° plies.

Also shown in figure 21 are the values of CTE determined from CLT assuming a 100-percent reduction in the transverse stiffness (E_2) of the crack plies. The value of α_y, predicted by CLT for cracks in the 90° plies only, is nearly equal to α_y predicted for cracks in both the 0° and 90° plies. Therefore, the effect of microcracks on α_y is overpredicted by CLT even at large crack densities (75 cracks/inch).

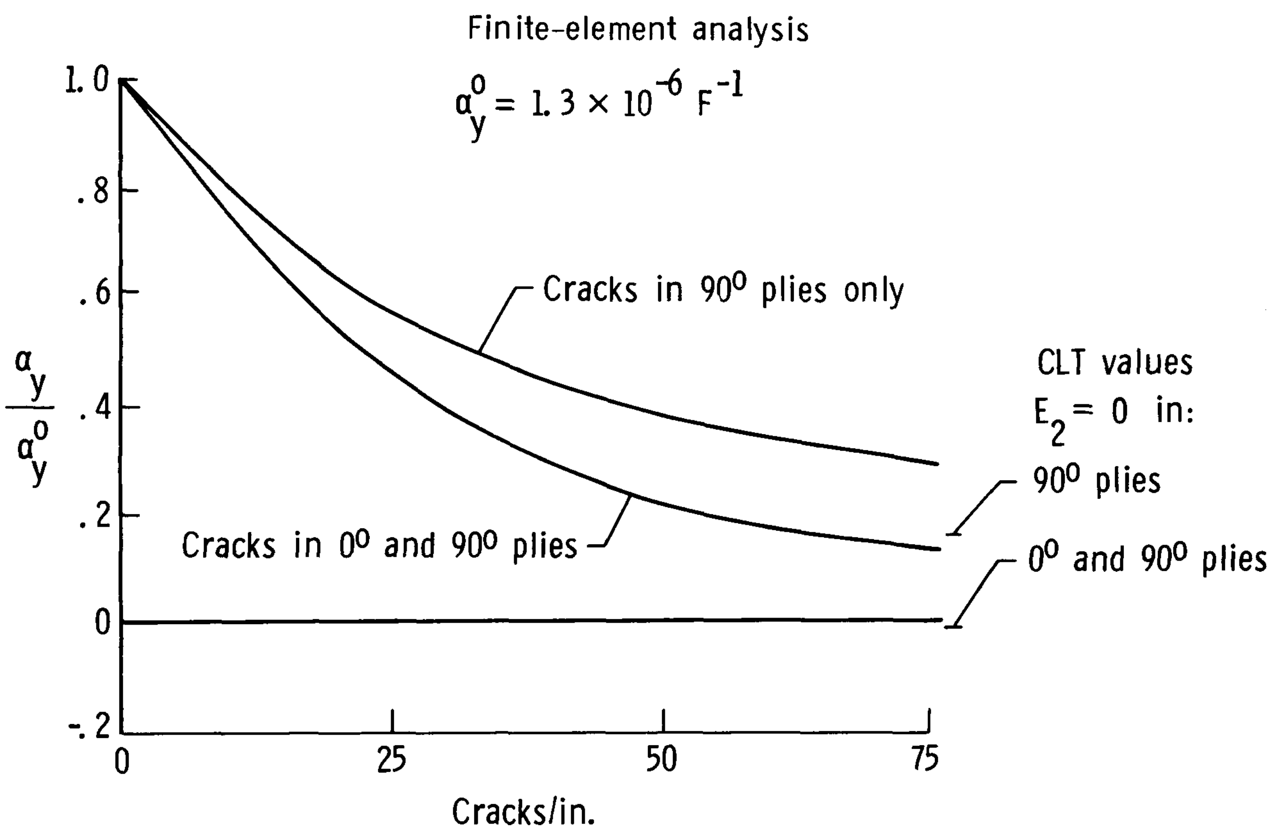

Figure 21

MICRODAMAGE IN GRAPHITE/EPOXY TUBES AFTER 500 THERMAL CYCLES BETWEEN -250°F AND 200°F

Graphite/epoxy tubes are being considered for construction of high stiffness truss structures for a number of different space systems. The durability of composite tubes subjected to the types of thermal cycles expected in space is a current research focus within NASA. A preliminary study of the effects of thermal cycling on the integrity has recently been completed (ref. 10). In this study, one-half-inch-diameter composite tubes with a $[90/0_6/90]$ wall were cycled between -250°F and 200°F up to 500 times. Different material systems were used in each of three tubes. Each tube had T300 fibers in the 90° plies. The same resin system was used throughout each tube. The 0° plies in each of the three tubes were T300/934, P75S/934, and P75S/CE339, respectively. Microdamage induced in the T300-P75S/934 tube by 500 thermal cycles is shown in figure 22. This is typical of the damage induced in each material.

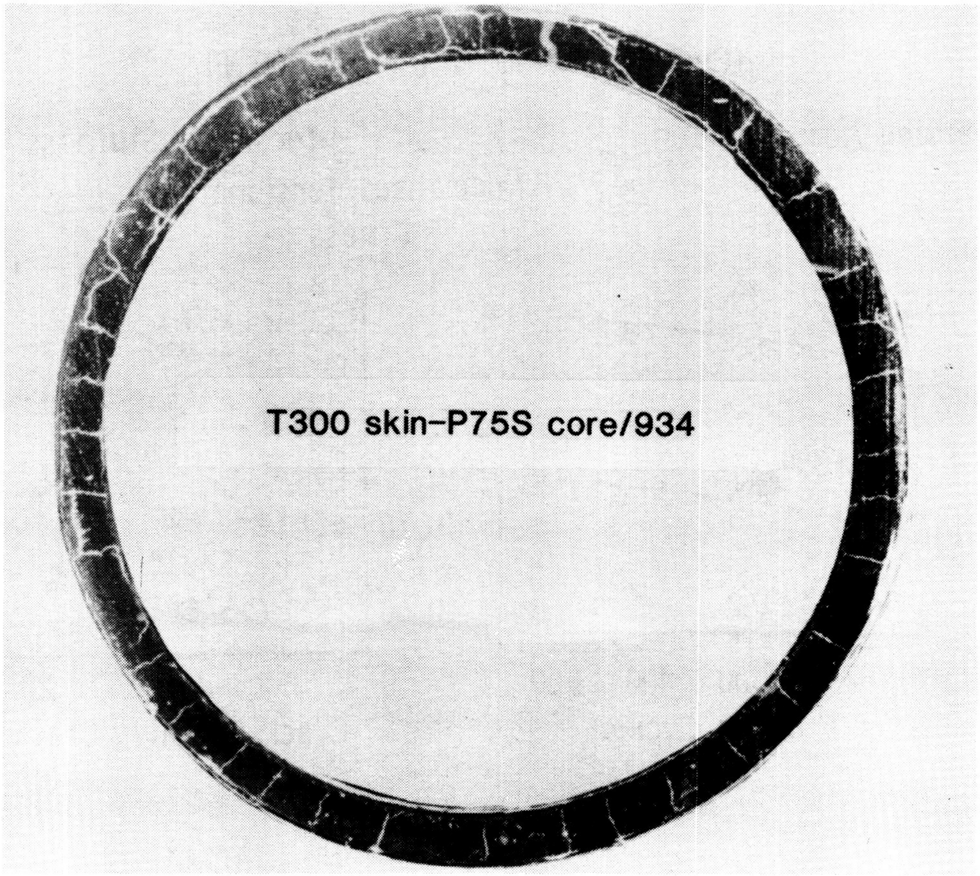

Figure 22

EFFECTS OF THERMAL CYCLING ON COMPOSITE TUBES

The changes in microcrack densities in each of three [90/0_6/90] graphite/epoxy 0.5-inch-diameter tubes due to thermal cycling are shown in figure 23. The crack density in each tube has not reached equilibrium even after 500 cycles. The data show a significant difference between the crack densities in the three materials. These differences are attributed to the differences in the expansion of fibers used and differences in the stiffness of the matrix resins used.

The effects of microcracks on the torsional stiffness of each tube (ref. 10) are also shown in figure 23. The data show a significant and rapid reduction in stiffness due to microcracks. The stiffness of all three materials appears to have the same sensitivity to microcracks. The data show about a 35-percent reduction in torsional stiffness at a crack density of about 25 cracks/inch or after 500 cycles between -250°F and 200°F.

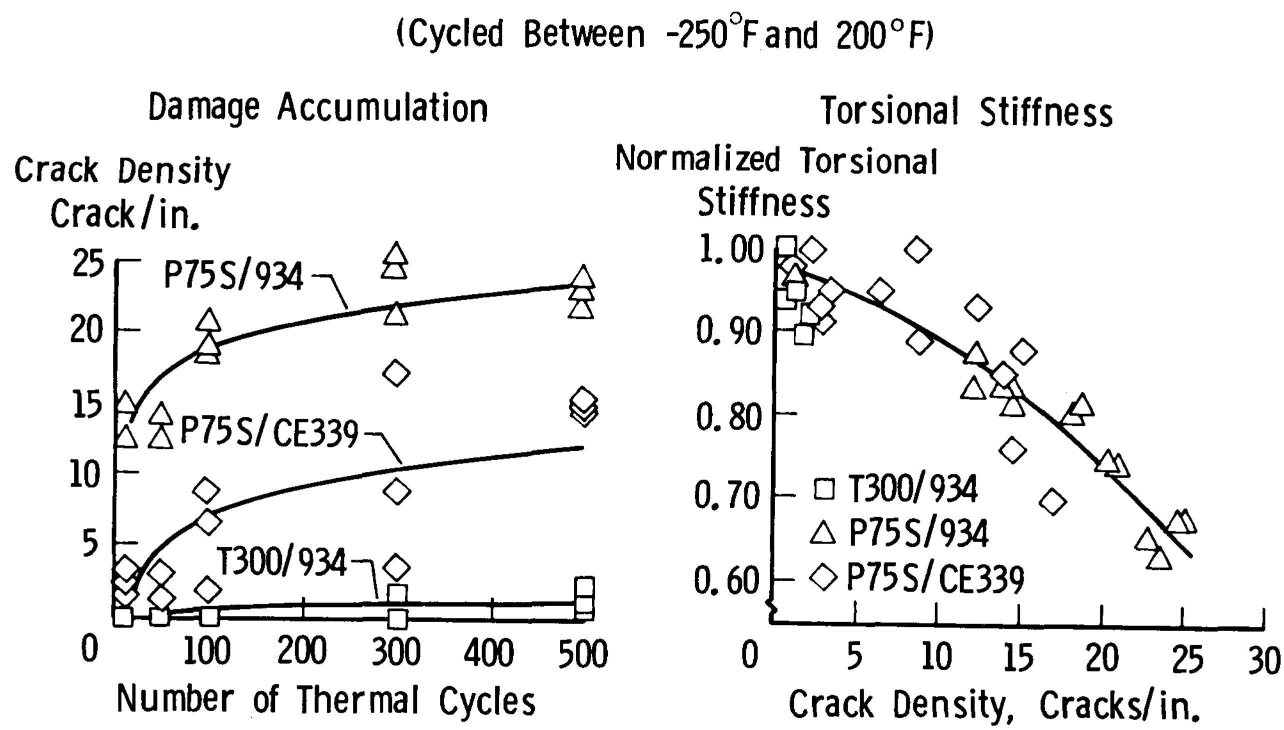

Figure 23

EFFECTS OF THERMAL CYCLING ON EXTENSIONAL AND FLEXURAL STIFFNESSES OF GRAPHITE/EPOXY TUBES

The extensional and flexural stiffnesses of each of three 0.5-inch-diameter [90/0_6/90] tubes before and after 500 thermal cycles between -250°F and 200°F (ref. 10) are shown in figure 24. The data show no significant effects of thermal cycling on the extensional and flexural stiffnesses of these tubes. Therefore, the radial microcracks induced by thermal cycling (fig. 22), which have a large effect on the torsional stiffness of these tubes (fig. 23), are not expected to be a major concern for truss structures designed with nonrigid joints where no appreciable torsional loadings are expected to be introduced in the tubes.

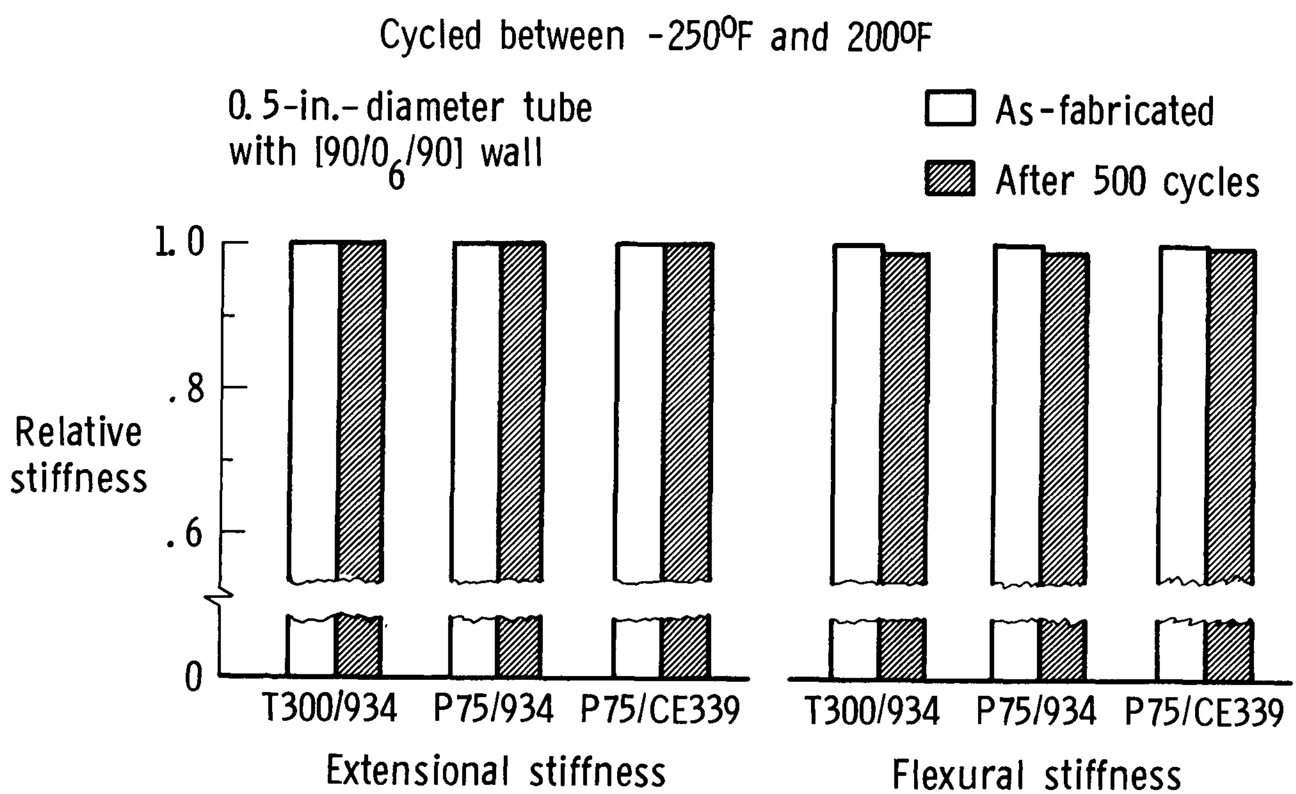

Figure 24

THERMAL EXPANSION OF P100/6061 BEFORE AND AFTER THERMAL PROCESSING TO MINIMIZE THERMAL STRAIN HYSTERESIS

Graphite-reinforced metal-matrix composites (MMC) represent a new class of high stiffness, low thermal expansion materials for structural applications on dimensionally critical spacecraft. These materials have higher electrical and thermal conductivity than resin-matrix composites, are more radiation resistant, and have no outgassing. Currently, the 6061 aluminum alloy is one of the primary metals being considered as the matrix for MMC. However, composites made with this alloy exhibit large hysteresis during thermal cycling and residual dimension changes induced by thermal cycling. This behavior is unacceptable for the reliable performance of dimensionally critical spacecraft.

The thermal expansion of a graphite/aluminum, P100/6061, as-fabricated, is shown in figure 25. The expansion is characterized by a large hysteresis loop and residual strain. Samples fabricated by both diffusion bonding (fabricated by DWA Inc.) and hot isothermal roll bonding (fabricated by MCI) exhibit essentially the same expansion behavior. A postfabrication process has been developed which significantly reduces the hysteresis loop and eliminates the residual strain. Thermal expansion after processing is also shown in figure 25. The postfabrication processes consist of standard heat treatments, followed by cryogenic prestraining, i.e., cryogenic soak. A different heat treatment was required for samples fabricated by each of the two methods. Different treatments were required because the fabrication techniques caused different microstructures and different amounts of magnesium depletion within the aluminum matrix (ref. 11). This change in chemistry alters the heat treatment required to strengthen the matrix alloy.

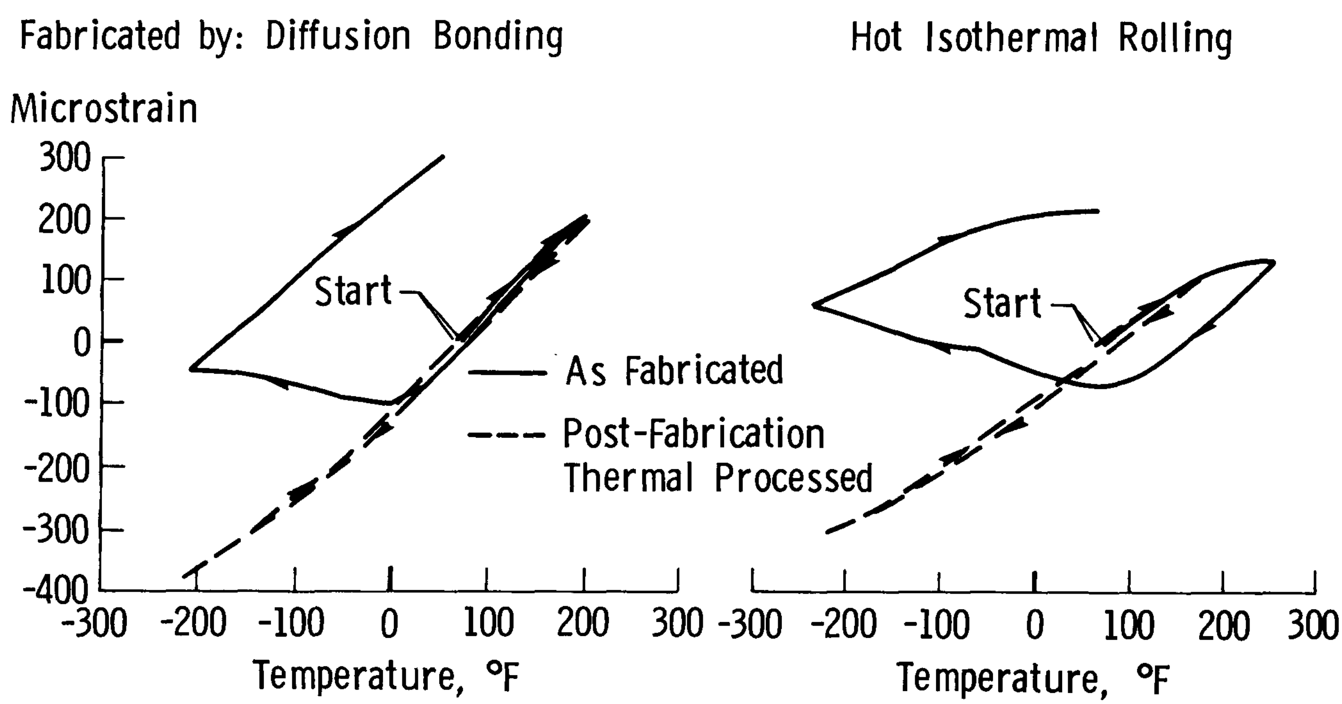

Figure 25

THERMAL EXPANSION OF $[0/\pm60]_S$ GRAPHITE/GLASS COMPOSITE

Graphite/glass is a relatively new material being considered for space structures. The thermal expansion of a quasi-isotropic graphite/glass (HMS/borosilicate) is shown in figure 26. The fiber volume fraction of this sample was 44 percent and the laminate orientation was $[0/\pm60]_S$. The expansion of this laminate is characterized by a hysteresis loop. However, there was no residual strain after one thermal cycle. The coefficient of thermal expansion for this laminate, based on the endpoints, is about 0.02 $\mu\varepsilon/°F$. Additional tests on other graphite/glass samples are under way to determine the cause of the observed hysteresis and the thermal mechanical stability of this class of composite.

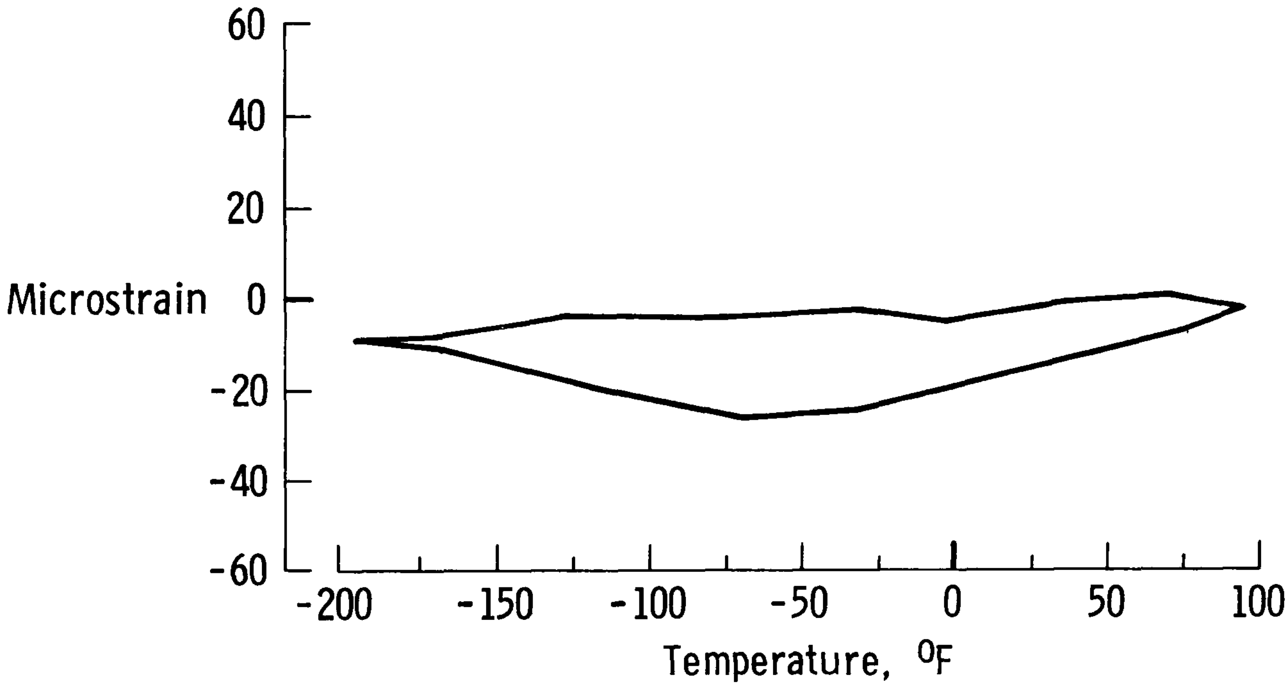

Figure 26

SUMMARY

Spacecraft materials research is a continuing research activity within NASA. The thrust of this research is to gain a fundamental understanding of the performance of advanced composites, coatings, and polymer films in the space environment. Emphasis has been placed on identification of damage mechanisms to guide new materials development. Some of the recent results of this activity are summarized in figure 27 below.

- Metallic coatings provide atomic oxygen stability and low solar absorptance and emittance
- Weight loss of siloxane-modified polyimides 1/10th that of Kapton in LEO exposures
- High doses of electron radiation cause embrittlement of Gr/Ep composites
- Radiation damage mechanisms identified in CE339 and Ultem resins
- Thermal cycling causes strain hysteresis in Gr/metal and Gr/glass composites
- Radial microcracks cause large (35%) reduction in torsional stiffness of composite tubes

Figure 27

REFERENCES

1. Leger, Lubert: Oxygen Atom Reactions with Shuttle Materials at Orbital Altitudes. NASA TM-58246, May 1982.

2. Slemp, Wayne S.; Santos-Mason, Beatrice; and Sykes, George F.: Effects of STS-8 Atomic Oxygen Exposure on Composites, Polymeric Films, and Coatings. Presented at the AIAA 23rd Aerospace Sciences Meeting, Reno, NV, January 14-17, 1985. (AIAA Paper No. 85-0421).

3. St. Clair, Anne K.; St. Clair, Terry L.; and Shevket, Keziban I.: Synthesis and Characterization of Essentially Colorless Polyimide Films. Proceedings of the 188th Meeting of the American Chemical Society, vol. 51, August 1984, p. 62.

4. Milkovich, Scott M.; Herakovich, Carl T.; and Sykes, George F.: Space Radiation Effects on Graphite-Epoxy Composite Materials. VPI-E-84-20, Virginia Polytechnic Institute and State University, June 1984.

5. Bowles, David E.; Tompkins, Stephen S.; and Sykes, George F.: Electron Radiation Effects on the Thermal Expansion of Graphite/Resin Composites. Presented at the AIAA 19th Thermophysics Conference, Snowmass, CO, June 25-28, 1984. (AIAA Paper No. 84-1704).

6. Sykes, George F.; and Slemp, Wayne S.: Space Radiation Effects on an Elastomer Toughened Epoxy/Graphite Composite. Presented at the 30th National SAMPE Symposium/Exhibition, Anaheim, CA, March 19-21, 1985.

7. Long, Sheila A. T.; and Long, Edward R., Jr.: Effects of Intermediate-Energy Electrons on Mechanical and Molecular Properties of a Polyetherimide. IEEE Transactions on Nuclear Science, December 1984.

8. Tompkins, S. S.; Bowles, D. E.; and Kennedy, W. R.: A Laser Interferometer Dilatometer for Thermal Expansion Measurements of Composites. Proceedings of V SESA International Congress on Experimental Mechanics, June 1984, pp. 367-377.

9. Bowles, D. E.: Effect of Microcracks on the Thermal Expansion of Composite Laminates. Journal of Composite Materials, vol. 18, March 1984, pp. 173-189.

10. Cohen, D.; Hyer, M. W.; and Tompkins, S. S.: The Effects of Thermal Cycling on Matrix Cracking and Stiffness Changes in Composite Tubes. Hi-Tech Review 1984, National SAMPE Technical Conference Series, vol. 16, 1984, pp. 577-588.

11. Dries, Gregory A.; and Tompkins, Stephen S.: Thermal Processing of Graphite Reinforced Aluminum to Minimize Thermal Strain Hysteresis. NASA TP-2402, 1985.