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Effects of Cure Temperature, Electron Radiation, and Thermal Cycling On P75/930 Composites

by Joan G. Funk

Introduction

Graphite/epoxy composites are candidates for future space structures and are currently being used in moderately short duration, low earth orbit (LEO) space applications. Materials with high stiffness and good dimensional stability are required for these space structures. Because the typical graphite/epoxy composite is brittle and has high residual stresses it has often been found to microcrack during thermal cycling typical of the space service environment (ref. 1,2,3). It also has been shown (ref. 1) that composite materials exposed to radiation typical of 30 years in geosynchronous orbit (GEO) and then thermally cycled have a higher microcrack density than if only thermally cycled. To reduce matrix microcracking due to thermal cycling, materials which are less brittle, or have lower cure temperatures and hence lower residual stresses, are being considered. For GEO-applications a reduction in microcracking is desired without sacrificing the material's radiation stability.

The purpose of the present study was to determine the effects of cure temperature and radiation exposure on the shear properties and microcrack resistance of a graphite/epoxy composite caused by thermal cycling. Since high modulus composite materials, and therefore high modulus fibers, are of increasing interest in space structural applications, a 75-Msi pitch-based graphite fiber, P75, was used as the reinforcing fiber. The resin system, 930, is a 275°F cure resin and was used because it was developed by ICI Fiberite to minimize microcracking induced by thermal cycling between $\pm$ 250°F. Composite shear strength was used as an indication of the composite resin material's radiation stability. The results of the radiation effects on the shear strength and microcrack density are compared to results for two standard 350°F cure graphite/epoxy systems, T300/934 and P75/934. The 934 matrix is a 350°F cure resin also manufactured by ICI Fiberite and the T300 is a 33-Msi modulus PAN-based graphite fiber.

Materials and Specimens

The 275°F cure resin system, 930, according to ICI Fiberite Corp. product literature, was developed to produce minimal microcracking when repeatedly thermally cycled between ± 250°F. P75/930 prepreg tape, purchased from ICI Fiberite was laid-up into $(0)_4$ and (0,90,90,0) panels and fabricated at NASA LaRC. The panels were vacuum bagged, vacuum applied, and heated to 250°F ±10°F at 3-5°F/ min. After 30 minutes at 250°F, a pressure of 115 psia was applied. The panels were heated to their preselected final cure temperature, ranging from 250-350°F (in increments of 25°F) 30 minutes after the pressure was applied. The panels were held for 6 hours ± 15 minutes at the final cure temperature. The panels were then cooled under pressure to below 175°F at 3-5°F/min. Subsequently, the panels were postcured in a nitrogen environment at atmospheric pressure at their respective cure temperatures. Following postcure, the laminates were C-scanned to determine the laminate quality. No flaws or defects were detected and the panels appeared uniform. The panels were nominally 18 inches by 24 inches and were 0.20 inches thick with a fiber volume fraction ranging from 61-67 percent.

The unidirectional 4-ply panels were machined into 10°-off axis specimens for shear strength measurement tests and $[90]_4$ specimens for dynamic mechanical analysis (DMA). The 10°-off axis specimens were 0.5 inches wide and 6.0 inches long and the DMA specimens were 0.5 inches wide and 1.0 inch long. Following exposure to radiation, as described in the next section, fiberglass tabs and strain gages were bonded on to the 10°-off axis specimens. Cross-ply, 0.5-inch-wide and 0.5- to 6.0-inches long specimens were also machined from the [0,90,90,0] panels and used to obtain the microcrack density before and after thermal cycling.

Environmental Exposures

The composite specimens were irradiated in vacuum with 1 MeV electrons using a Radiation Dynamics Corporation Dynamatron accelerator. All specimens were irradiated at a dose rate of 5×10^7 rads/hr. At this dose rate, using a water-cooled specimen mounting plate the temperature of the composite specimens during irradiation remained below 100°F. The specimens were irradiated to a total dose simulating up to 30 years exposure (1×10^{10} rads) to the space environment.

A dual-chamber thermal cycling system was used to simulate the thermal cycling environment in GEO. In this system, specimens placed in a mechanically driven tray were alternately moved from a hot (150°F) chamber to a cold (-250°F) chamber. The upper temperature limit was chosen such that the temperature of the specimens did not enter the glass transition region of the material at any of the cure temperatures studied. The total time for one cycle was about 20 minutes. The specimens, which were used to determine the microcrack density, received up to 500 cycles.

Specimen Characterization

The 10°-off axis tensile specimens were tested in a hydraulic testing machine to determine the effects of cure temperature and radiation exposure on shear strength. During these tests the specimens were loaded at a rate of 0.02 in/min. parallel to the specimen axis. From ref. 4, the shear strength is defined as $S=0.171\sigma_{ult}$ where σ_{ult} is the ultimate strength of the 10° off-axis laminate.

Dynamic mechanical analysis for determining specimen glass transition temperature (T_g) was performed using a DuPont model 981 DMA. The DMA data were collected on 0.5-inch by 1.0-inch specimens between -200°F and 350°F at 9°F/min.

Thermally cycled specimens were examined for evidence of microcracking using an X-ray-opaque penetrant solution (zinc iodide/isopropyl alcohol). The penetrant was applied to the specimen along the edges and allowed to flow into the cracks for several minutes. Following this soak period, the specimens were wiped clean with a water-dampened cloth and radiographed. The number of cracks per inch along the length of the specimen were measured from the radiographs.

Results

Glass Transition Temperature

The effect of the cure temperature on the glass transition temperature is shown in figure 1. In general, T_g increases with increasing cure temperature from 230°F at a cure temperature of 250°F to 277°F at a cure temperature of 350°F.

The increase in T_g is indicative of an increase in the cross-link density of the 930 matrix material.

The effect of total electron radiation dose on the T_g of the materials cured at the different cure temperatures is also shown in figure 1. All of the materials, regardless of cure temperature, were effected by radiation exposure. The lowest radiation dose of 1 x 10^8 rads resulted in a T_g roughly equivalent to the T_g of the nonirradiated material processed at the same cure temperature with the exception of the 250°F cured material. The initial radiation dose of 10^8 rads significantly increased the T_g of the 250°F cured material indicating that the 10^8 rads dose induced cross-linking in the material. However, after exposure to 1 x 10^9 rads of electron radiation all the materials exhibited a significant decrease in T_g, indicating that chain scissioning is the dominate radiation-induced change. Prior to electron radiation exposure the T_g of the materials ranged from 227°F to 277°F, whereas after exposure to 1 x 10^{10} rads, the T_g ranged from 190°F to 210°F. The threshold dose at which chain scissioning by radiation begins is a total dose between 10^8 and 10^9 rads.

Shear Strength

The shear strength of the material was not significantly effected by cure temperature as shown in figure 2, but was significantly effected by exposure to 1 x10^{10} rads of electron radiation. All of the materials regardless of cure temperature suffered a substantial decrease in shear strength by about an order of magnitude, to about 0.7 ksi.

The effects of total dose of electron radiation on the shear strength of the 275°F cured material was determined. As shown in figure 3, the shear strength decreases markedly with increasing radiation dose. After exposure to 1 x 10^8 rads the material retains about 80 percent of its original strength. However, at a total dose of 1 x 10^{10} rads, the shear strength decreased to only 10 percent of its original value.

Microcrack density

The as-processed materials did not exhibit any initial microcracks at any of the cure temperatures. After exposure to 50 cycles from -250°F to 150°F, all of the materials, regardless of cure temperature, exhibited significant microcracking with microcrack densities ranging from 20-30 cracks/inch as shown in figure 4. As the materials underwent additional cycling, the microcrack density continued to increase. After 500 thermal cycles the microcrack densities were between 41 and 46 cracks/inch, and independent of cure temperature.

A summary of the microcrack density as a function of cure temperature, radiation dose, and number of thermal cycles for the P75/930 composite is shown in Table I. Examples of the radiographs which show the microcrack density for the 275°F cured material are shown in figure 5. The radiation exposure alone did not result in any microcracking regardless of cure temperature. Exposure to electron radiation followed by thermal cycling, regardless of the total radiation dose, resulted in higher microcrack densities than for the nonirradiated thermally cycled materials. The microcrack density resulting from radiation exposure followed by thermal cycling increased with increasing radiation dose and increasing number of thermal cycles until the microcrack density approached 50-55 cracks/inch after 500 thermal cycles.

Comparison with 934 composite systems

A comparison of the present results can be made with similar data available in the literature for a standard, well-characterized, epoxy resin system, 934. The T_g of T300/934, ref. 5, as processed was 410°F and after exposure to 1×10^{10} rads electron radiation it is reduced by 27 percent to 300°F. The present P75/930 material cured at 275°F has a T_g of 226°F as processed and 192°F after exposure to 1×10^{10} rads of electron radiation; a reduction of 15 percent. These T_g data indicate that in both systems, 930 and 934, a high radiation dose (1×10^{10} rads) induces chain-scissioning in the matrix material.

It is of interest to compare the effect of electron radiation on the shear strength of the composites made from the two resin systems, 930 and 934. No data are currently available in the open literature on radiation effects on the shear strength of P75/934. However, the effect of radiation exposure on the shear strength of T300/934 is documented in ref. 5. As shown in figure 6, the effect of radiation on the shear strength of the two graphite/epoxy materials, P75/930 and T300/934, is different. In the case of P75/930, the shear strength of the 275°F cured material after exposure to 1×10^{10} rads is degraded to 10 percent of the nonirradiated shear strength. However, in the case of T300/934, this same electron radiation exposure does not significantly degrade shear strength (ref.

5).

Microcrack density data from $(0_2,90_2)_s$ P75/934 specimens thermally cycled between ± 250°F have been published in ref. 2. The P75/930 data (table I) and the P75/934 data (ref. 2) have the same lower temperature extreme, -250°F in the thermal cycle. Although the upper temperature extreme of the thermal cycle is different, it should not have a major impact on the microcrack density since the extremes are well below the materials' T_g. The lay-ups for the two materials are also different: the P75/930 being $[0,90]_s$ and the P75/934 being $[0_2,90_2]_s$. However, microcracking is believed to be controlled by the plies which constrain the ply in question. In both cases, the 90° plies are surrounded by a 0° and a 90° ply. Therefore a comparison between the two lay-ups and thermal cycles, although not a direct one-to-one comparison, can be used to indicate the relative microcrack resistance of the two materials. As shown in figure 7, the P75/930, cured at 275°F, had 0 cracks/inch as fabricated and 45 cracks/inch after 500 thermal cycles. The P75/934 had 12 cracks/inch as fabricated and the microcrack density, which appeared to stabilize after 10 cycles, had 27 cracks/inch after 250 thermal cycles (ref. 2). Therefore, the P75/930 did not exhibit a higher microcrack resistance than the P75/934. No microcrack density data are available for the P75/934 after radiation exposure followed by thermal cycling, so this comparison is not possible.

Based on the present results, and a comparison of these results with data on 934 composite systems from the literature, P75/930 does not have an improved microcrack resistance over P75/934, and, in addition, the 930 resin system appears more sensitive to electron radiation-induced degradation than the 934 resin system.

Conclusions

Results of this study reveal that the P75/930 graphite/epoxy composite system is significantly degraded by total doses of electron radiation greater than 10^8 rads and by thermally cycling between -250°F and 150°F. Specifically the following conclusions can be drawn from this study:

1. The cure temperature, between 250°F and 350°F, had little effect on the shear strength and microcrack density.
2. The T_g decreased from 230-277°F (depending on the cure temperature) to 190-210°F after exposure to 1×10^{10} rads electron radiation.

3. The shear strength of the material cured at the manufacturer's recommended (275°F) cure temperature decreased after 1 x 10^{10} rads electron radiation to only 10 percent of the nonirradiated material's shear strength.
4. Repeated thermal cycling between -250°F and 150°F resulted in extensive microcracking.
5. Exposure to electron radiation prior to thermal cycling increased the microcrack density induced by thermal cycling.
6. P75/930 did not exhibit improved microcrack resistance over a well-characterized, standard 350°F cure system, P75/934.
7. The 930 resin system appears to be more sensitive to electron radiation-induced degradation than the 934 resin system.

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Table I. Microcrack density of P75/930 $(0,90)_s$ laminates before and after exposure to electron radiation followed by thermal cycling.

Number of cycles, -250 °F to 150 °F	Cure Temperature, °F	Microcrack density, cracks/inch			
		Unirradiated	1×10^8 rads	1×10^9 rads	1×10^{10} rads
0	250	0	0	0	---
	275	0	0	0	---
	300	0	0	0	---
	325	0	0	0	---
	350	0	0	0	---
50	250	30	---	34	---
	275	24	40	46	---
	300	32	36	42	---
	325	20	32	26	---
	350	---	38	44	---
100	250	36	36	42	---
	275	30	38	44	---
	300	38	34	40	---
	325	36	36	38	---
	350	40	38	52	---
250	250	38	44	58	---
	275	38	42	58	---
	300	38	46	46	---
	325	40	40	42	---
	350	38	58	56	---
500	250	44	41	54	54
	275	45	48	55	50
	300	41	45	41	54
	325	46	48	46	54
	350	45	51	49	52

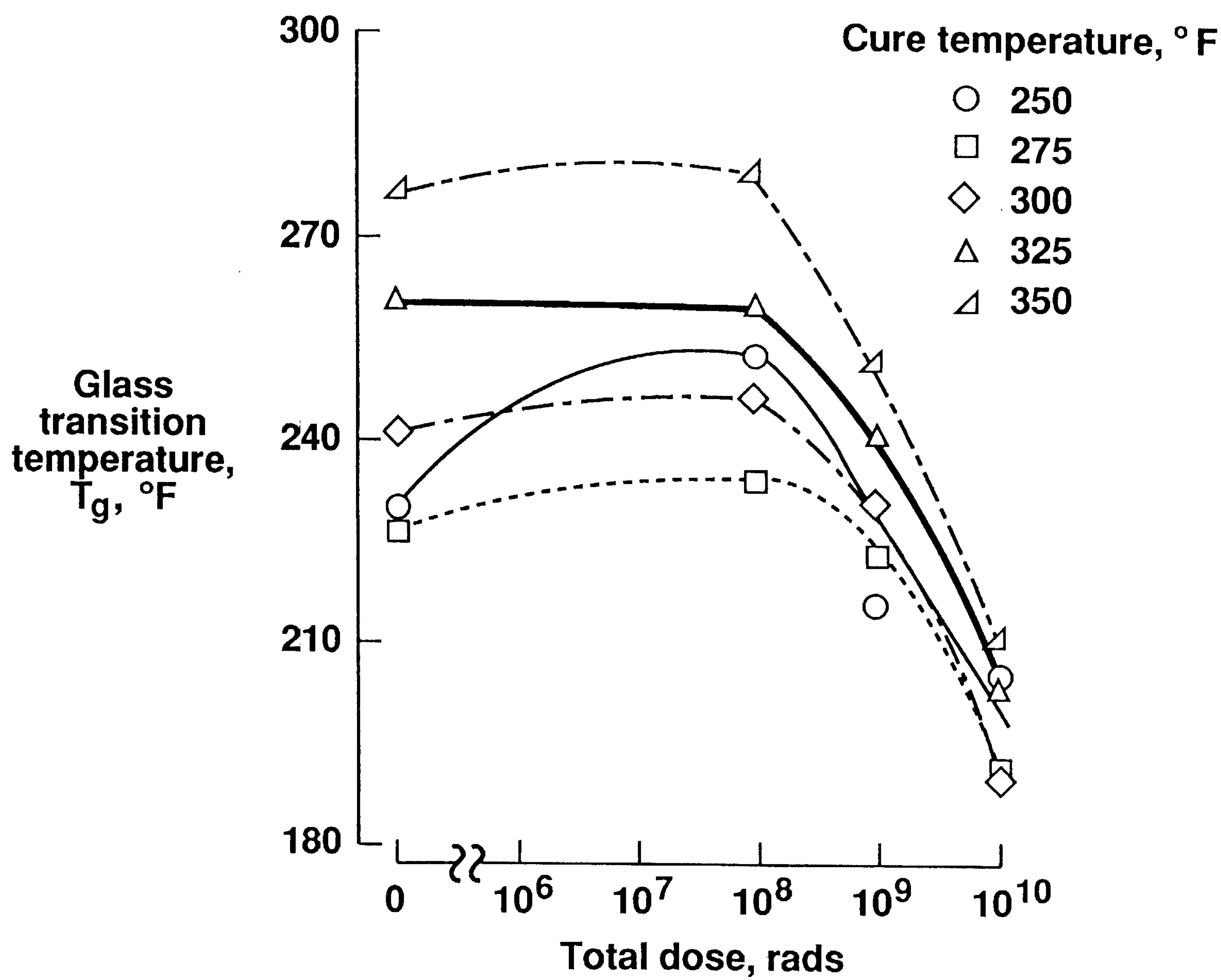

Figure 1. Effect of cure temperature and electron radiation exposure on glass transition temperature of P75/930.

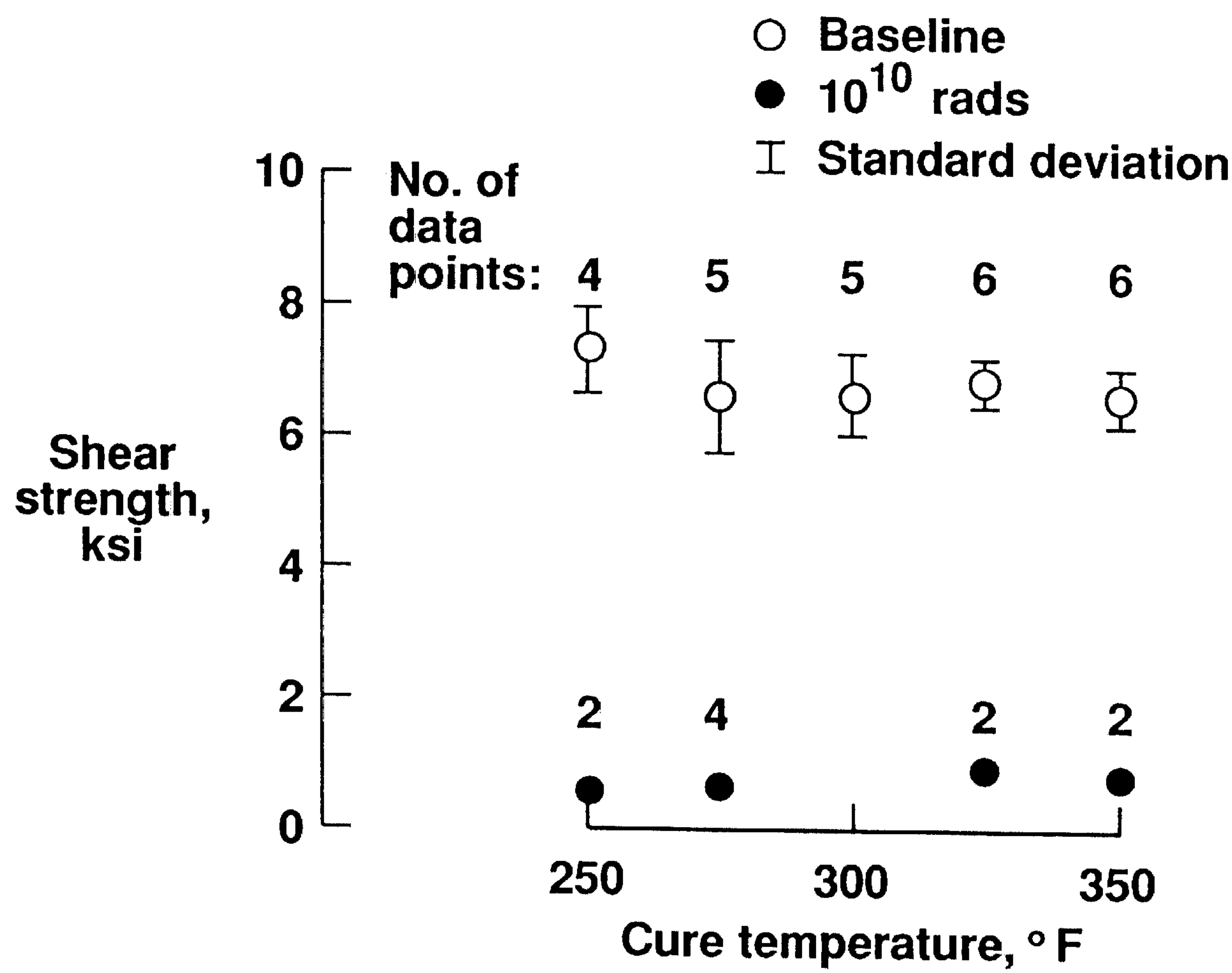

Figure 2. Effects of cure temperature and electron radiation exposure on the shear strength of P75/930.

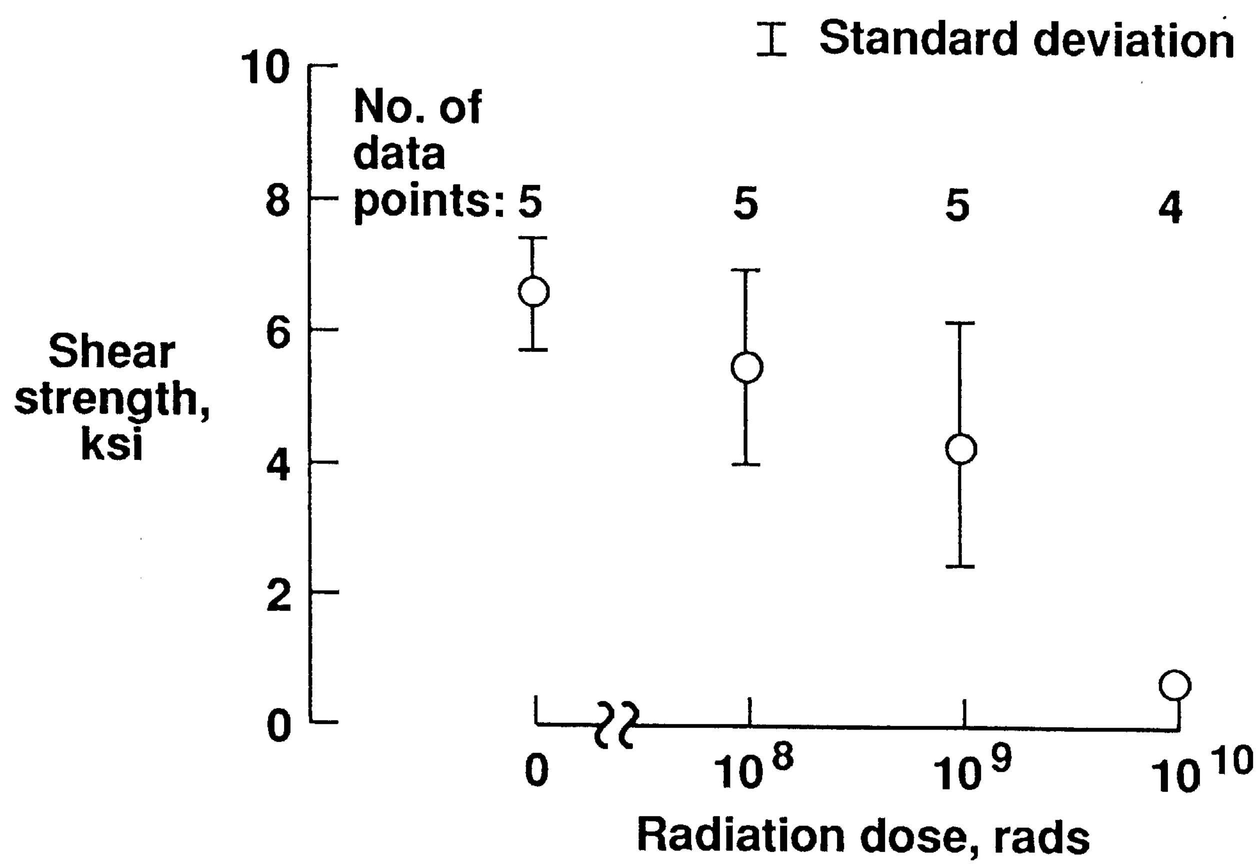

Figure 3. Effect of electron radiation exposure on the shear strength of P75/930 cured at 275°F.

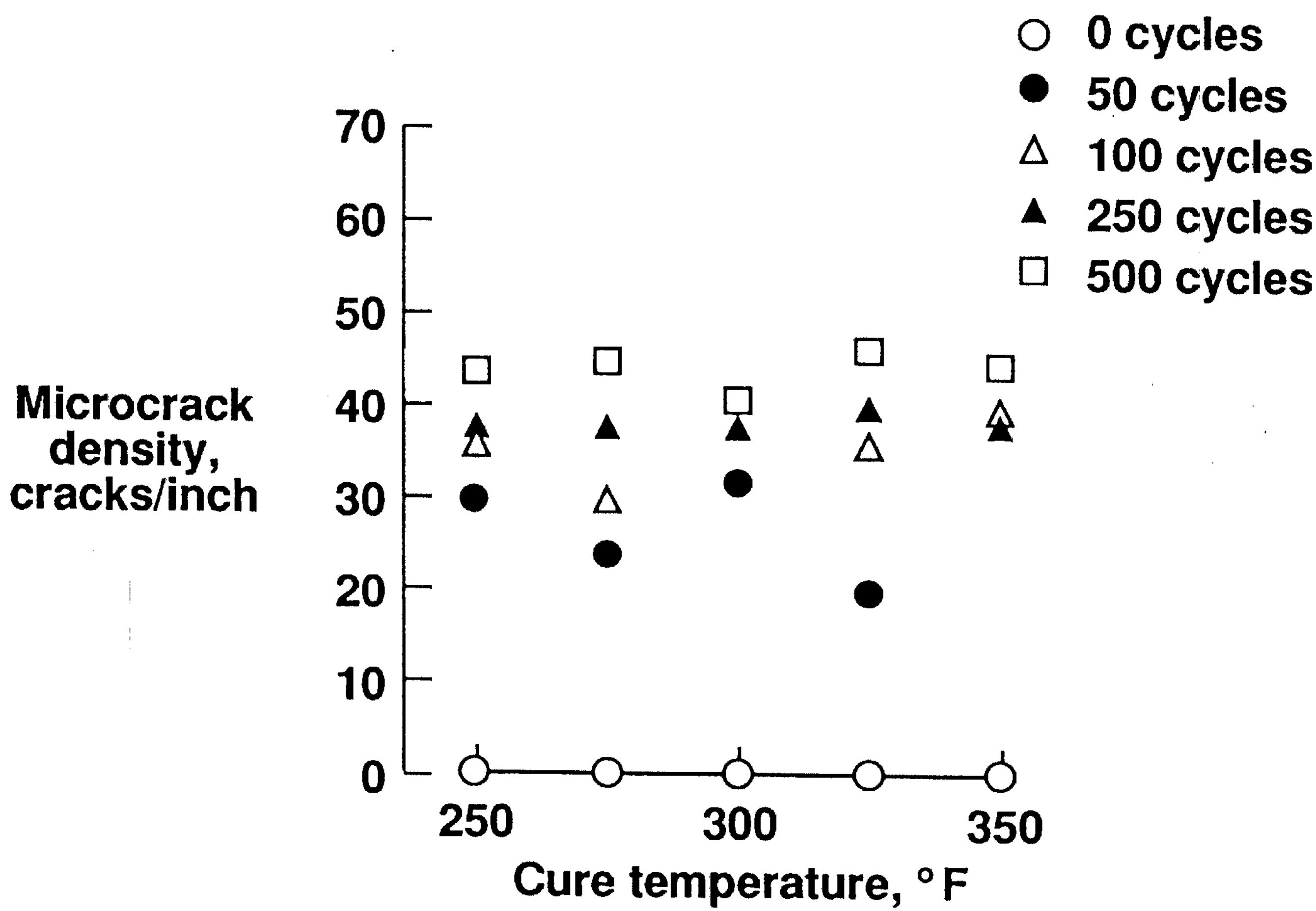

Figure 4. Microcrack density of [0, 90,90,0] P75/930 laminates as a function of the number of -250°F to 150°F thermal cycles.

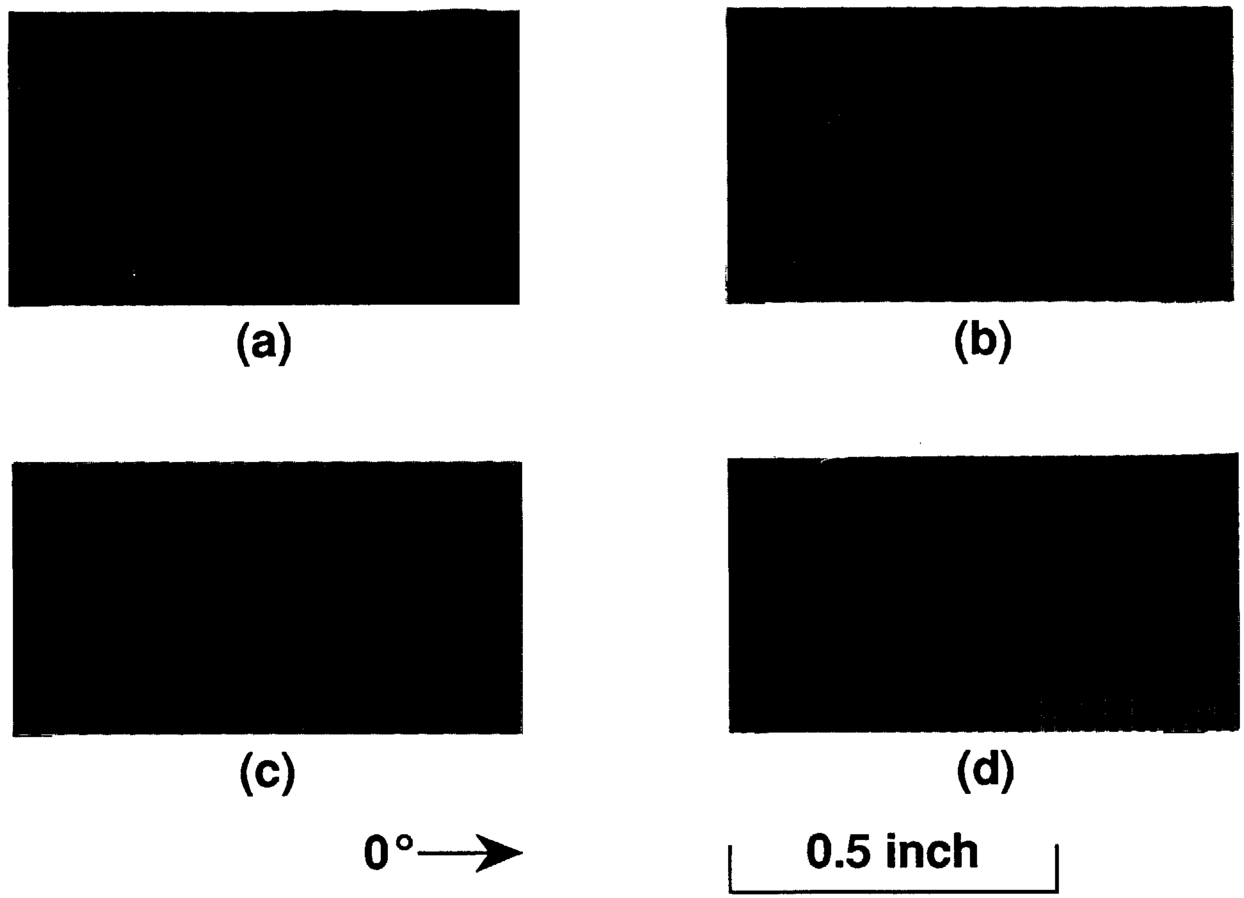

Figure 5 . Radiographs of the 275° F cured material (a) as-processed, (b) after 500 thermal cycles between -250°F and 150°F, (c) after exposure to 1 x 10^9 rads electron radiation, and (d) after exposure to 1 x 10^{10} rads electron radiation followed by 500 thermal cycles between -250°F and 150°F.

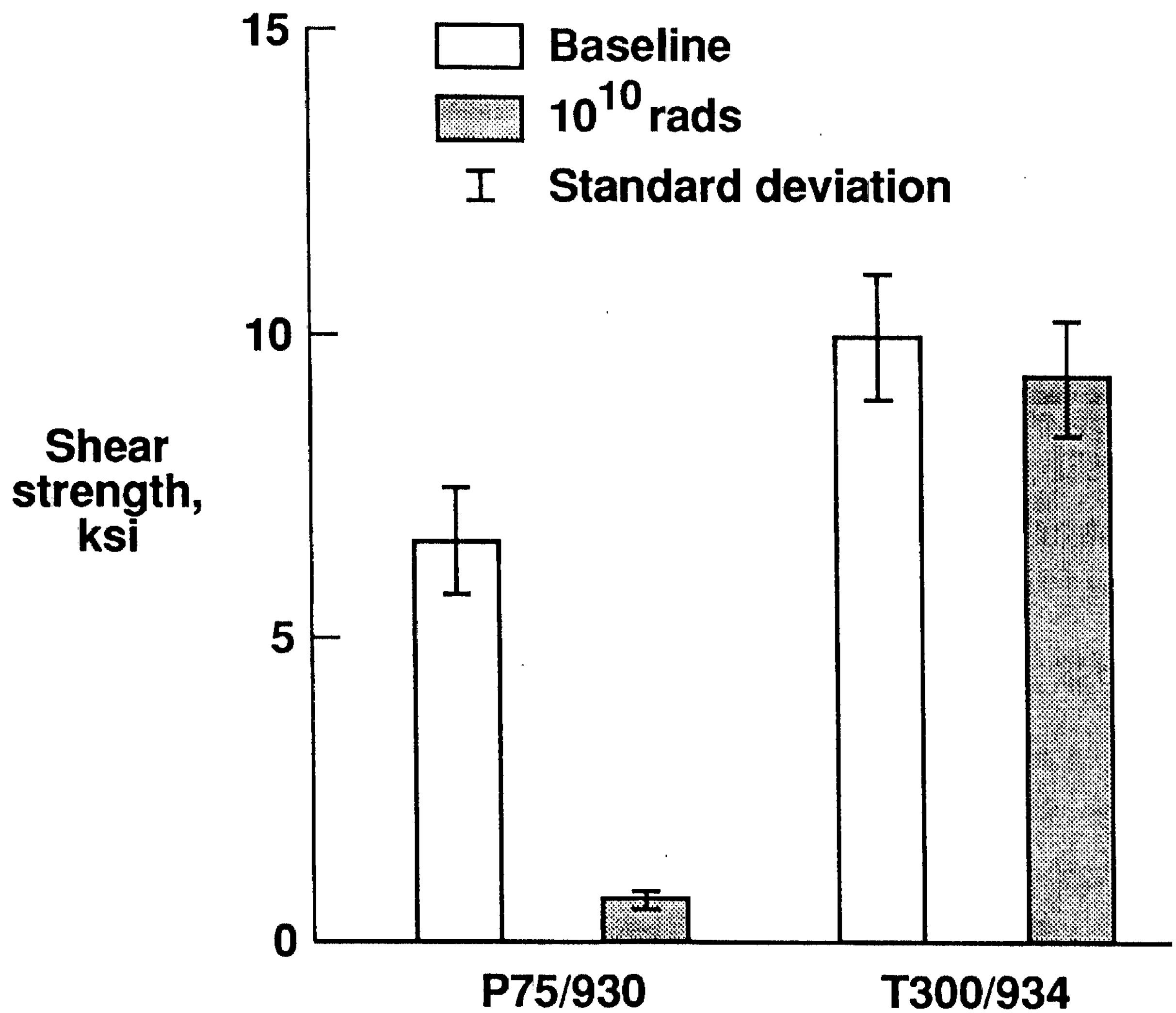

Figure 6. Change in shear strength of P75/930 and T300/934 due to electron irradiation.

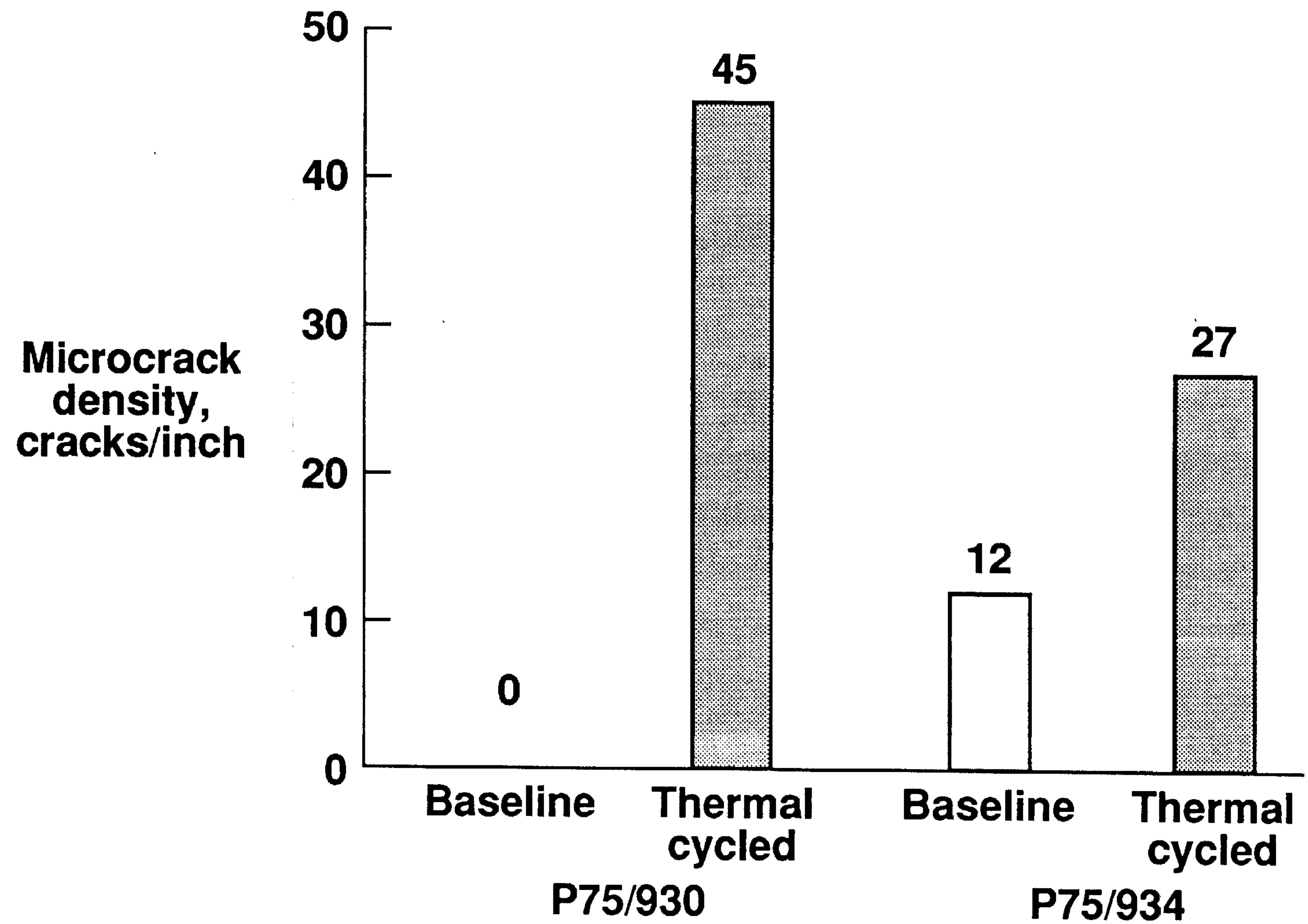

Figure 7. Comparison of microcrack density of thermally cycled P75/930 and P75/934 composite laminates. The P75/930 $[0, 90]_s$ laminate was thermally cycled between -250°F and 150°F 500 times. The P75/934 $[0_2, 90_2]_s$ laminate was thermally cycled between $\pm$ 250°F 250 times.

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16. Abstract

Graphite/epoxy composites are candidates for future space structures due to high stiffness and dimensional stability requirements of these structures. Typical graphite/epoxy composites are brittle and have high residual stresses which often result in microcracking during the thermal cycling typical of the space environment. Composite materials used in geosynchronous orbit applications will also be exposed to high levels of radiation. The purpose of the present study was to determine the effects of cure temperature and radiation exposure on the shear strength and thermal cycling-induced microcrack density of a high modulus, 275°F cure epoxy, P75/930. The results from the P75/930 are compared to previously reported data on P75/934 and T300/934 where 934 is a standard 350°F cure epoxy. The results of this study reveal that P75/930 is significantly degraded by total doses of electron radiation greater than 10^8 rads and by thermally cycling between -250°F and 150°F. The P75/930 did not have improved microcrack resistance over the P75/934, and the 930 resin system appears to be more sensitive to electron radiation-induced degradation than the 934 resin system.

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