Effects of Thermal Cycling on Mechanical Properties of Graphite Polyimide

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An experimental study to determine the effects of thermal cycling on residual mechanical properties of a graphite/polyimide composite has been conducted. Interlaminar shear, flexure, and compression strengths were measured at room temperature and 316°C on unidirectional and quasi-isotropic laminates subjected to 150 and 500 thermal cycles between −156 and 316°C. Thermal cycling produced transverse microcracks and delaminations in the laminates. The linear density of the microcracks (35 per cm) was about the same after 150 and 500 cycles. Although the crack density did not change from 150 to 500 cycles, crack growth did occur with intralamina cracks growing into adjacent lamina. The matrix controlled properties of the quasi-isotropic laminate at both room temperature and 316°C were reduced significantly by thermal cycling. However, the properties of the unidirectional laminate were reduced significantly only at 316°C.

Introduction

GRAPHITE-fiber/polyimide-matrix (Gr/PI) composite materials are an important class of structural materials for elevated temperature application on aerospace vehicles.[1] Before Gr/PI structures can be designed with confidence, the effects of the service environment on the composite material must be determined and understood. The temperature range of interest for aerospace application of these composites is from −156 to 316°C. Studies have been conducted over this temperature range to investigate the effects of static thermal exposure on mechanical properties.[2-6] However, only limited data are available characterizing the effects of thermal cycling on these materials. Hyer and Hagaman[7] investigated the deformation of Gr/PI when subjected to five quasistatic cycles between room temperature (RT) and 180°C, and RT and 316°C. Herakovich et al.[8] investigated microcracking developed in Gr/PI after single cycles between −195 and 330°C at various heating and cooling rates.

The purpose of the present study was to determine the effects of thermal cycling on the residual mechanical properties of Gr/PI composites at room temperature and 316°C. Both unidirectional and quasi-isotropic laminates of Gr/PI were subjected to 150 and 500 thermal cycles between −156 and 316°C. Residual interlaminar shear, flexure, and compression strengths after thermal cycling were measured and compared to as-fabricated strengths. The effects of thermal cycling on the morphology of the Gr/PI composite laminates were investigated also.

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Materials, Specimens, and Test Procedures

The test material used in this study was the C6000/PMR-15, graphite/polyimide, composite material. The Ceion‡ 6000 (C6000) graphite fibers had a polyimide sizing based on DuPont NR150B resin. The composite panels were consolidated at 325°C in a vacuum bag using previously developed procedures[9] and postcured for 6 h at 325°C. Four different laminate layups were investigated: $[0]_{20}$, $0/+45/90/-45)_2]_S$, $[0]_8$, and $[0/+45/90/-45]_S$. The 16- and 20-ply panels were fabricated in 61 cm^2 sheets. These panels, used for interlaminar shear specimens only, had a fiber volume content between 55 and 57%, and glass transition temperature T_g between 280 and 290°C after 6 h at 325°C. The 8-ply panels were fabricated in 64 × 94 cm sheets. These panels, used for flexural and compression specimens, had a fiber volume content between 60 and 67% and T_g greater than 320°C after 6 h at 325°C. The nominal composite ply thickness for all panels was 0.0152 cm. Schematic diagrams of the test specimens cut from these panels are shown in Fig. 1.

All specimens were stored at room temperature prior to thermal cycling. After thermal cycling and before mechanical tests, specimens were stored for a minimum of seven days in a desiccator at room temperature. The cross-sectional area of each specimen was measured prior to thermal cycling and these areas were used to calculate the failure strengths.

A schematic diagram of the dual-chamber exposure unit[10] used in this study is shown in Fig. 2a. Specimens, lightly held, but not loaded, on a mechanically driven sliding tray, were alternately heated to 316°C in a circulating air hot chamber and cooled to −156°C in a liquid nitrogen cooled, cold chamber. A typical temperature history for one cycle obtained with a thermocouple attached to the surface of a specimen is shown in Fig. 2b. Specimens were exposed to this environment for either 150 or 500 cycles. Reference 8 showed that only cooling rates associated with a liquid nitrogen quench would affect the microcrack density significantly. Since specimens did not come in contact with liquid nitrogen

‡Celion is a registered trademark of Celanese Corporation.

in this study, the same microcrack density should be induced by a thermal cycle with a period longer than 25 min, as was induced by the cycle used in this study.

The edge of a typical specimen of each type was scanned on an optical microscope at a magnification of 150 for transverse microcracks (TVM) and delamination before and after thermal cycling. The TVM and delamination on the edge were enhanced by wet (acetone) sanding with 600 grit silicon carbide paper. The specimen was then cleaned in an ultrasonic cleaner with acetone for about 10 min. The TVMs were counted in the 90° plies at three different locations along the length of the specimen. An average linear TVM density (number of cracks per unit length) was then calculated.

An x-ray opaque penetrant, tetrabromethane, was used to determine if the cracks extended the width of the sample. The penetrant was allowed to flow into the cracks along the edge of the sample. A radiograph of the sample was made after the penetrant was allowed to soak into the cracks for a couple of minutes.

Both interlaminar shear and flexure strengths were determined in three-point bending with a center point displacement rate of 0.127 cm/min until failure occurred. Span-to-thickness ratios of 4 and 40 were used for the shear and flexure tests, respectively. Tests at 316°C commenced after specimens were held at 316°C for approximately 15 min to allow thermal equilibrium to be reached. Compression strengths were determined with the test fixture and technique developed by Clark and Lisagor.[11]

Results and Discussion

Thermal Cycle Induced Microcracks

Two types of damage can be produced in resin matrix composites during thermal cycling. The predominant damage is microcracking of the matrix parallel to the fibers. These cracks develop when the internal stress exceeds the transverse strength of a lamina. In a unidirectional laminate, TVM result from the mismatch in thermomechanical properties between fiber and matrix. In a quasi-isotropic laminate, TVM result from stresses caused by the mismatch in properties between laminae of different orientations which are superimposed on stresses due to fiber-matrix property mismatch. The second type of damage which occur is delamination which appears at and extends parallel to the interface between laminae. They are formed when the thermally generated cyclc stresses exceed the interlaminar strength of the laminate.

Both TVM and delamination can be induced during the fabrication process. In the present study damage was not detected in the as-fabricated specimens. However both types of damage were detected after repeated thermal cycling.

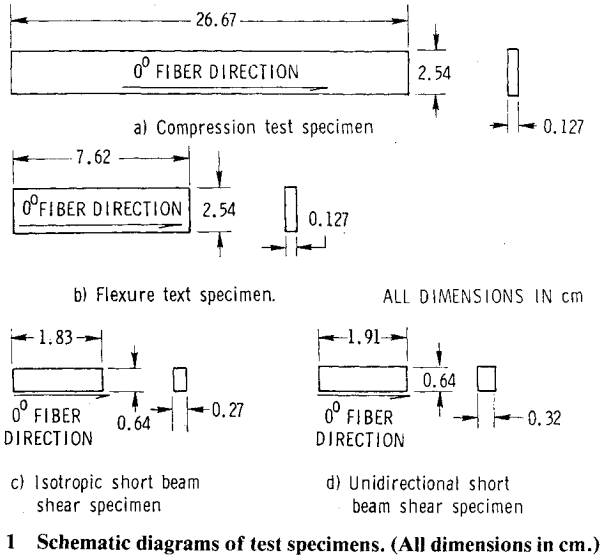

Fig. 1 Schematic diagrams of test specimens. (All dimensions in cm.)

Typical photomicrographs of unsanded specimens edges showing TVM and delamination that developed during thermal cycling are shown in Figs. 3 and 4. After 150 cycles, TVM are clearly seen spanning the thickness of the 90° plies, Fig. 3a. The cracks after 150 cycles, in general, did not spread into adjacent plies as they did after 500 cycles, Fig. 3b. The sizes of both the TVM and delimination were larger after 500 cycles than after 150 cycles.

Transverse microcracks were also induced in both the longitudinal and transverse unidirectional laminates, Fig. 4. The direction of the TVM appears random and the TVM appear to be concentrated in the center third of the thickness.

For the thermal case, all of the interior lamina have the same transverse loading and TVM would be expected in all lamina. However, the observed TVM pattern was greatly dependent upon which specimen edge was viewed. The TVM appeared best defined in plies in which the fiber direction was parallel to the line of sight. The crack patterns observed in the 90° plies were assumed to be typical of the damage induced throughout the laminate. To verify this, the outer three plies were partially removed as shown in Fig. 5. TVM are clearly seen running parallel to the fibers and across the entire width of each exposed ply. The extent of the TVM throughout the material is better seen with x ray . A radiograph of a sample, Fig. 6, also shows cracks in each ply running parallel to the fiber direction and the full width of the sample. The extent of the TVM in these specimens is consistent with that reported in Ref. 8.

The observed TVM densities were about 35 cracks per cm. Although the TVM densities were about the same after 150 or 500 cycles, the cracks after 150 cycles, in general, did not spread into adjacent plies as they did after 500 cycles. The TVM density of 35 per cm was much higher than the 8 per cm reported after only one cycle room temperature to cure temperature with a quench to liquid nitrogen.[8] This difference may be attributed to the dfference in the number of thermal cycles used in each study.

Residual Mechanical Properties

Flexure Strength

The residual flexure data for the $[0]_8$, $[0/+45/90/-45]_s$, and $[90]_8$ laminate at RT and 316°C are shown in Tables 1 and 2 and Fig. 7. Three specimens were used at all conditions except for the $[90]_8$ laminate reference specimens at both temperatures, Tables 1 and 2. The flexure strength of all three laminates, before cycling, were significantly lower at 316°C than at RT. Although matrix cracking was observed after

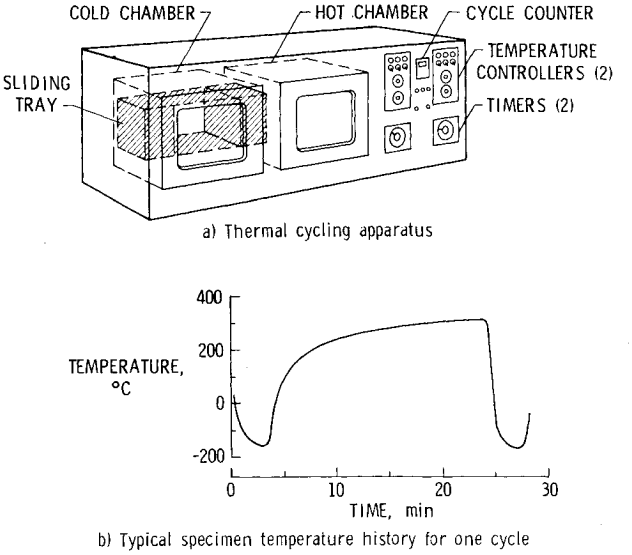

Fig. 2 Cyclic temperature exposure.

thermal cycling, the strengths of the $[0]_8$ and $[0/+45/90/-45]_s$ laminates were not significantly affected primarily because the flexure strength is fiber controlled. The initial increase in strength of the laminate at both test temperatures after 150 cycles may be attributed to further curing of the resin during thermal exposure.

The RT strengths of the $[90]_8$, which are almost totally matrix controlled, were degraded significantly by cycling. The strength was 15 and 41% lower than the references after 150 and 500 cycles, respectively. This sensitivity was not surprising since the $[90]_8$ laminate relies on the integrity of the matrix, which was degraded by cracks, for strength. However, the insensitivity of the strength of this laminate at 316°C to thermal cycling and therefore microcracks was unexpected (Table 2). Since the laminate relies on the matrix for strength, extensive damage and TVM in the resin were expected to reduce the flexure strength significantly at both RT and 316°C. The elevated temperature reduced the strength significantly (about 66%) as was expected. But since the elevated temperature strength was insensitive to thermal cycling, the data suggest that the exposure to 316°C weakened the resin and/or fiber-matrix interface to the extent that the thermally induced microdamage was secondary to the effect of temperature on the flexure strength.

Compression Strength

The residual compressive strength data for $[0]_8$ and $[0/+45/90/-45]_s$ laminates at RT and 316°C are shown in Tables 3 and 4 and Fig. 8. Three specimens were used at each test condition. The strength of both laminates, before cycling, was significantly lower at 316°C than at RT. At RT, the thermal cycling and resulting microdamage did not appear to affect the compressive strength of the $[0]_8$ laminate adversely. This was expected because cracking of the matrix, which would close under compression, would not significantly change the support for the fibers in the $[0]_8$ laminate. However, at 316°C and after 500 cycles, the strength of the $[0]_8$ laminate had dropped about 16%. This suggests a possible combined effect of temperature and microdamage.

The strength of the $[0/+45/90/-90]_s$ specimen, which is more matrix controlled than the $[0]_8$, should be more sensitive to induced damage than the $[0]_8$ specimens. At RT, the strength of the $[0/+45/90/-45]_s$ laminate after 150 cycles was not significantly different from the reference. However,

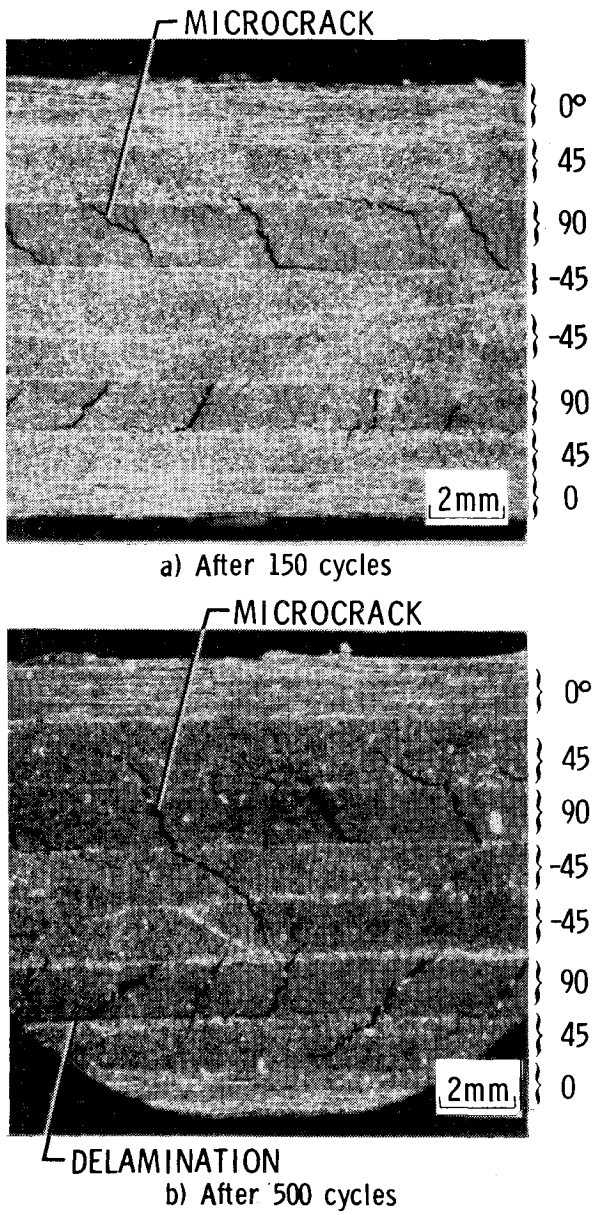

Fig. 3 Typical microcracks in C6000/PMR-15 laminate, $[0/+45/90/-45]_s$, after thermal cycling between −156 and 316°C.

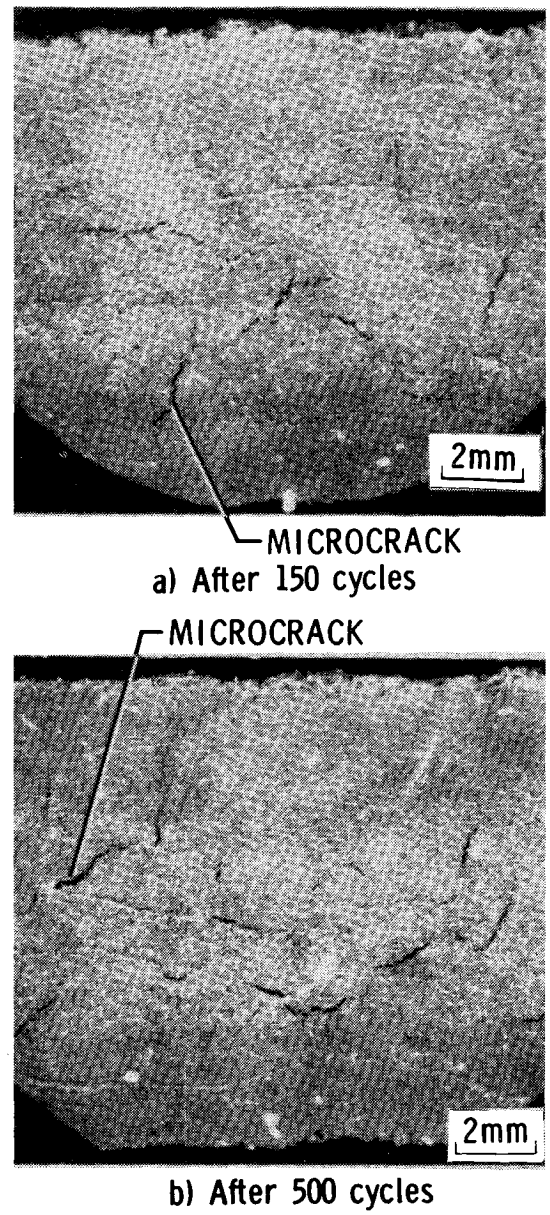

Fig. 4 Typical microcracks in C6000/PMR-15 laminate, $[90]_8$, after thermal cycling between −156 and 316°C.

after 500 cycles the strength was 22% lower that the reference. At 316°C the strengths of the $[0/+45/90/-45]_s$ laminate, after 150 and 500 cycles were about 6 and 25%, respectively, below the reference values. This indicates that the strength may be a function of both the size of the microdamage and TVM density.

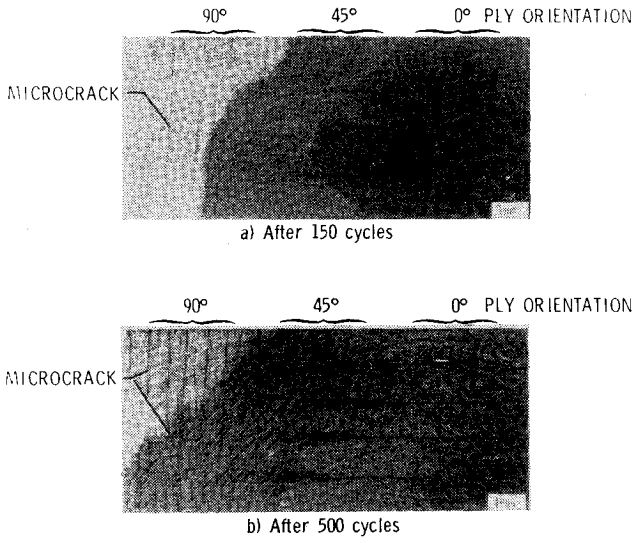

Fig. 5 Typical microcracks in sanded, tapered section of C6000/PMR-15 laminate, $[(0/+45/90/-45)_2]_s$, after thermal cycling between −156 and 316°C and interlaminar shear test at room temperature.

Table 1 Flexure strength of C6000/PMR-15 laminate at room temperature after cycling between −156 and 316°C

Specimen no.	No. of cycles	Ultimate stress, GPa	Average stress, GPa	Percent change from reference
		$[0]_8$ Laminate		
C7 71	0	1.83		
85	0	1.77	1.78	0
98	0	1.74		
57	150	1.89		
69	150	1.85	1.89	6
76	150	1.92		
60	500	2.09		
64	500	1.66	1.83	3
81	500	1.74		
		$[90]_8$ Laminate		
C7 42	0	0.069		
48	0	0.103	0.085	0
33	150	0.078		
37	150	0.073	0.075	−15
53	150	0.072		
46	500	0.051		
47	500	0.047	0.050	−41
52	500	0.052		
		$[0/+45/90/-45]_s$ Laminate		
C16 5	0	1.21		
13	0	1.18	1.20	0
20	0	1.21		
2	150	1.31		
11	150	1.13	1.23	2
22	150	1.24		
1	500	1.09		
7	500	1.05	1.07	−11
19	500	1.06		

Interlaminar Shear Strength

At 316°C, interlaminar shear strengths (ISS) of cycled specimens were found to be about 50% higher than the ISS of the as-frabricated specimens. Tests on uncycled, as-frabricated specimens showed that the glass transition temperature, T_g, of these laminates was significantly below the expected value, 320°C. Because the maximum temperature during thermal cycling (316°C) was above the T_g of these specimens additional curing of the specimens occurred during the maximum temperature part of each cycle.

To determine how much additional curing could occur, the ISS of uncycled specimens was measured after postcuring an

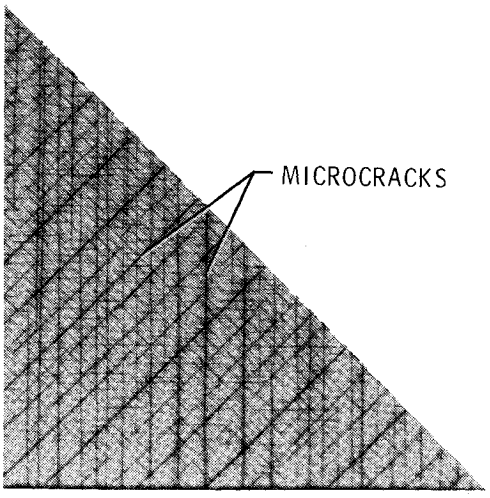

Fig. 6 Typical x-ray, with tetrabromethane penetrant, of C6000/PMR-15 laminate, $[0/+45/90/-45]_s$ after 500 thermal cycles between −156 and 316°C.

Table 2 Flexure strength of C6000/PMR-15 laminate at 316°C after cycling between −156 and 316°C

Specimen no.	No. of cycles	Ultimate stress, GPa	Average stress, GPa	Percent change from reference
		$[0]_8$ Laminate		
C7 65	0	1.107		
73	0	1.135	1.121	0
82	0	1.121		
83	150	1.284		
90	150	1.348	1.310	16.9
99	150	1.299		
86	500	1.370		
92	500	1.420	1.361	21.4
96	500	1.294		
		$[90]_8$ Laminate		
C7 39	0	0.031	0.029	0
107	0	0.027		
43	150	0.037	0.035	20
55	150	0.033		
32	500	0.037		
36	500	0.036	0.035	21.9
49	500	0.033		
		$[0/+45/90/-45]_s$ Laminate		
C16 15	0	0.705		
16	0	0.723	0.702	0
28	0	0.678		
21	150	0.743		
23	150	0.774	0.747	6.5
27	150	0.725		
10	500	0.695		
14	500	0.697	0.717	2.1
30	500	0.758		

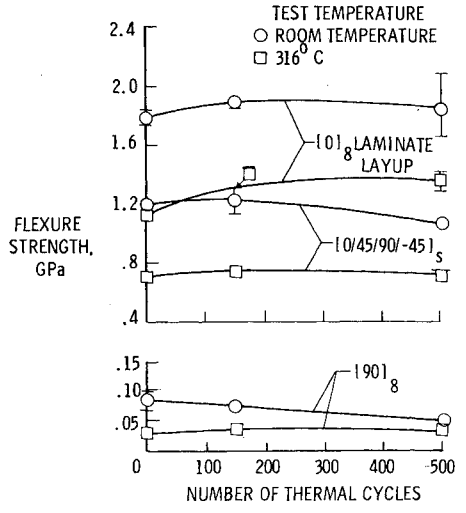

Fig. 7 Effects of thermal cycling on residual flexure strength of C6000/PMR 15 at room temperature and 316°C.

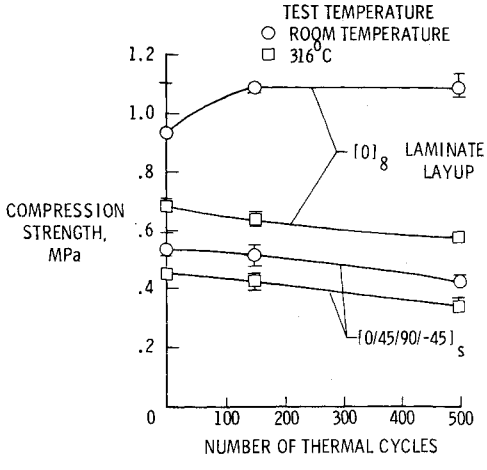

Fig. 8 Effects of thermal cycling on residual compression strength of C6000/PMR-15 at room temperature and 316°C.

Table 3 Compression strength of C6000/PMR-15 laminate at room temperature after cycling between −156 and 316°C

Specimen no.	No. of cycles	Ultimate stress, GPa	Average stress, GPa	Percent change from reference
		$[0]_8$ Laminate		
C5 36	0	0.703		
51	0	1.11	0.938	0
60	0	1.01		
14	150	1.09		
34	150	1.10	1.09	16
56	150	1.08		
44	500	1.14		
45	500	1.06	1.09	16
55	500	1.07		
		$[0/+45/90/-45]_s$ Laminate		
C15 13	0	0.538		
20	0	0.552	0.538	0
31	0	0.517		
14	150	0.524		
23	150	0.476	0.517	−4
53	150	0.545		
5	500	0.411		
24	500	0.412	0.421	−22
29	500	0.440		

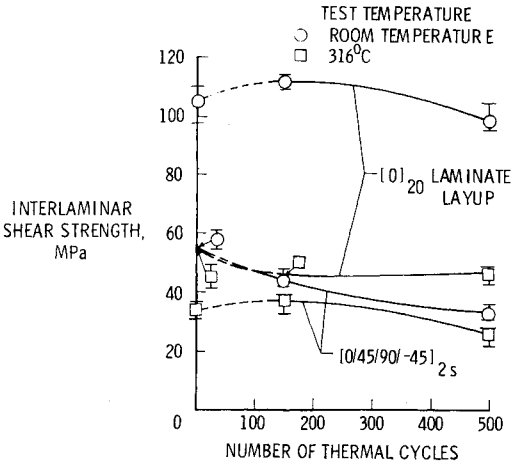

Fig. 9 Effects of thermal cycling on residual interlaminar shear strength of C6000/PMR-15 at room temperature and 316°C.

Table 4 Compression strength of C6000/PMR-15 laminate at 316°C after cycling between −156 and 316°C

Specimen no.	No. of cycles	Ultimate stress, MPa	Average stress, MPa	Percent change from reference
		$[0]_8$ Laminate		
C5 25	0	708.1		
42	0	594.1	688.2	0
46	0	762.6		
12	150	615.5		
22	150	665.8	634.3	−7.8
59	150	621.7		
2	500	580.5		
3	500	564.3	577.4	−16.1
21	500	587.5		
		$[0/+45/90/-45]_s$ Laminate		
C15 21	0	455.6		
26	0	440.2	451.5	0
59	0	458.6		
2	150	394.6		
10	150	453.3	425.3	−5.8
28	150	428.2		
1	500	325.1		
4	500	370.8	340.8	−24.5
7	500	326.5		

additional 48 and 96 h at 325°C. The RT ISS was essentially unaffected by these postcures. However, the strengths of the $[0]_{20}$ and $[(0/+45/90/-45)_2]_s$ specimens measured at 316°C after 48 h postcure were 82 and 24%, respectively, higher than the as-fabricated specimens. A small additional change was measured after 96 h.

Because the specimens were at 316°C for 48 h or more during 150 and 500 thermal cycles, these data indicate that curing nearly was completed during the cyclic exposure. Therefore, specimens postcured for 96 h were used as reference specimens for the 316°C residual tests and as-fabricated specimens were used as reference for the RT residual tests. None of the cycled specimens had been postcured in addition to the original 6 h.

The residual ISS data for cycled $[0]_{20}$ and $[(0/+45/90/-45)_2]_s$ specimens at RT and 316°C are shown in Tables 5 and 6 and Fig. 9. Five specimens were tested at each condition. The ISS of both laminates before cycling were significantly lower at 316°C than at RT. The data at 150 cycles are connected to the reference data by dashed lines to indicate that the specimens were continuing to cure during the first 150 cycles. The TVM density induced in these specimens by thermal

Table 5 Interlaminar shear strength of C6000/PMR-15 laminate at room temperature after cycling between −156 and 316°C

Specimen no.	No. of cycles	Ultimate stress, GPa	Average stress, GPa	Percent change from reference
		$[0]_{20}$ Laminate		
C8 63	0	98		
97	0	100		
151	0	109	105	0
172	0	108		
188	0	110		
96	150	109		
99	150	110		
146	150	111	112	7
167	150	114		
179	150	113		
116	500	105		
138	500	96		
166	500	97	99	−6
191	500	101		
199	500	97		
		$[(0/45/90/-45)_2]_s$ Laminate		
C4 28	0	59		
53	0	53		
70	0	54	56	0
71	0	54		
96	0	57		
54	150	46		
56	150	48		
57	150	43	44	−21
62	150	42		
65	150	43		
55	500	31		
69	500	36		
89	500	32	33	−41
90	500	34		
92	500	33		

Table 6 Interlaminar shear strength of C6000/PMR-15 laminate at 316°C after cycling between −156 and 316°C

Specimen no.	No. of cycles	Ultimate stress, GPa	Average stress, GPa	Percent change from reference
		$[0]_{20}$ Laminate		
C8 86	0	50.4		
89	0	51.6		
163	0	55.5	54.7	0
196	0	58.3		
197	0	57.8		
100	150	46.0		
133	150	46.5		
147	150	45.6	45.6	−17
170	150	45.5		
176	150	44.6		
56	500	47.5		
112	500	44.5		
117	500	43.3	46.3	−15
140	500	47.1		
177	500	49.1		
		$[(0/+45/90/-45)_2]_s$ Laminate		
C4 134	0	35.0		
159	0	32.5		
161	0	36.6	34.0	0
163	0	34.6		
175	0	31.4		
59	150	37.3		
64	150	36.2		
66	150	36.1	36.8	8
78	150	32.8		
97	150	36.8		
72	500	26.8		
85	500	27.8		
86	500	22.1	26.3	−22
87	500	26.7		
100	500	28.2		

cycling was essentially the same as measured in the flexure and compression specimen.

At RT, the ISS of the $[0]_{20}$ laminate was essentially unaffected by thermal cycling. At 316°C, the ISS for the laminate was reduced by about 17% after 150 cycles, with no additional significant change after 500 cycles. Davis[12] and Serafini[6] showed no significant drop in ISS of $[0]_{20}$ laminates after continuous exposure to 316°C in air for up to 1000 h. Therefore the lower ISS are attributed primarily to the effects of TVM (microcracking). Since the strength at 316°C was much more sensitive to thermal cycling than the strength at RT, the data suggest a possible combined effect of the elevated test temperature and the microdamage induced by cycling on ISS.

At RT, the ISS of the $[(0/+45/90/-45)_2]_s$ laminate was reduced significantly after 150 cycles and was further reduced after 500 cycles. This indicates that the strength was a function of the size of the microdamage, as well as the TVM density. The strength at 316°C was unchanged after 150 cycles; however, after 500 cycles the strength was 22% below the reference value. The ISS of the $[(0/+45/90/-45)_2]_s$ laminate was much more sensitive to the damage induced by thermal cycling than the strength of the $[0]_{20}$ laminate. Since the ISS of the $[(0/+45/90/-45)_2]_s$ laminate is more matrix controlled than the $[0]_{20}$ this sensitivity was expected.

Concluding Remarks

An experimental study was conducted to determine the effects of thermal cycling on residual mechanical properties of C6000/PMR-15, graphite/polyimide composite material. Both unidirectional and quasi-isotropic laminates were tested. Specimens were cycled 150 and 500 times between −156 and 316°C. Room temperature and 316°C residual interlaminar shear, flexure, and compressive strength were measured.

Thermal cycling induced transverse microcracks and delaminations in the laminates. The linear density of the microcracks (35 per cm) was about the same after 150 and 500 cycles. Although the crack density did not change, crack growth did occur with intralamina crack growing into adjacent lamina. The compression and interlaminar shear strengths of the unidirectional laminates were degraded by cycling only at 316°C. However, the compression and shear strengths of the quasi-isotropic laminate tested were affected significantly at both temperatures. Room temperature flexure strength of a unidirectional transverse laminate was reduced significantly by cycling. However, at elevated temperature, the flexure strengths of all laminates tested were not affected adversely by cycling.

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