

Effects of 50,000 h of Thermal Aging on Graphite/Epoxy and Graphite/Polyimide Composites

J. R. Kerr* and J. F. Haskins†

General Dynamics Corporation, San Diego, California

Thermal effects on tensile strengths of advanced composite systems have been determined for exposure times of 100 to 50,000 h (5.7 years). Exposures were conducted at both ambient and reduced pressures at two temperatures for each composite. At the completion of the various aging periods, specimens were removed from the specially constructed aging furnaces, visually examined, and tensile tested at elevated temperature. After tensile testing, many of the thermal aging specimens were examined using a scanning electron microscope. Results of these studies are presented, and the changes in properties and the degradation mechanisms during high-temperature aging are discussed and illustrated using metallographic techniques.

Introduction

ADVANCED composites will play a key role in the technology emerging for the design and fabrication of future supersonic vehicles. Research and development during recent years have led to advances in fabrication techniques and characterization of short-time properties, and have provided limited supersonic flight experience for these materials. However, information on the effects of long-time exposure to service environments representative of supersonic cruise aircraft on composite materials has not generally been available. An extensive program to generate such information has been in progress at General Dynamics Convair Division since 1973.¹ Changes in mechanical properties that occur over very long periods of time are being measured for ambient and thermal aging conditions, and for random cyclic loading temperature variations. These latter tests, the flight simulation exposures, are intended to provide data on the effect of 10,000-, 25,000-, and 50,000-h simulated supersonic-flight service on residual properties of the composites. The purpose of the thermal aging study (conducted at constant temperature without load) was to assist in understanding the results of the more complex flight-simulation program. While the flight simulation tests are still in progress, the thermal aging specimens have completed the required 50,000 h of exposure, and residual strength data are now available.

This paper presents the results of these aging tests for graphite/epoxy (G/E) and graphite/polyimide (G/PI) composites, each exposed to two elevated temperatures for time periods of 100 to 50,000 h (5.7 years). Exposures were conducted at ambient pressure and also at a reduced pressure of 0.014 MN/m² (2 psi) to simulate high-altitude flight conditions. A second oxygen pressure was selected because, in general, these resin matrix composites are oxidation prone. Previous work had shown a direct correlation of thermal aging and oxygen pressure on residual strength of resin matrix composites.² In actual use, a supersonic cruise vehicle would be at a very high altitude during much of the time at which the structure has reached its maximum temperature. Thus, if oxygen pressure is taken into account, the composite materials can likely be used at higher temperatures and for longer time periods.

Experimental

The composite systems employed in this program were A-S/3501 graphite/epoxy and HT-S/710 graphite/polyimide. Six-ply unidirectional and crossplied laminates of each system were fabricated by Convair Division from vendor-supplied prepreg material using conventional autoclave processing methods. Quality assurance testing was conducted on both the as-received prepreg and the fabricated laminates. These acceptance tests included fiber, resin, and volatile contents, resin flow, and process gel for the prepreg material and ultrasonic C-scan, fiber content, specific gravity, and metallographic examinations for the six-ply panels. Table 1 lists information for the two composites.

Baseline tensile and thermal aging specimens, 0.013 × 0.23 m (0.5 × 9 in.) were cut from the panels using a diamond impregnated saw. To prevent failures in the gripping fixtures during tensile testing, tapered doublers were bonded to the ends of the specimens. For the thermal aging specimens, the doublers were attached after exposure. Doublers, epoxy-glass for G/E, and polyimide-quartz for G/PI were bonded with HT-424, a modified epoxy-phenolic film adhesive with an aluminum filler on a glass carrier.

The thermal aging exposures were conducted in specially constructed aging furnaces similar to the sketch in Fig. 1. The heater plates consist of insulated wire sandwiched between two thin aluminum plates. The reduced pressure specimens are enclosed in containers consisting of an aluminum picture frame with vacuum fittings and thin aluminum covers. The top cover is sealed with a weld after the specimens are in place. The top is cut off and rewelded periodically during the 50,000 h to remove specimens for testing. The reduced pressure is obtained by a vacuum pump simultaneously pumping on one side of each container. Bleed air in valved into the other side to maintain the desired pressure, thus assuring a constant fresh supply of air to each container. All furnace temperatures were equilibrium-controlled, i.e., a constant amount of power was supplied to the heaters. The various aging temperatures were generally maintained to ±3K (±5F) with infrequent excursions to a maximum of ±6K (±10F).

The aging temperatures were: G/E, 394 and 450K (250 and 350°F); and G/PI, 505 and 561K (450 and 550°F). The reduced pressure exposures were all performed in 0.014 MN/m² (2 psi) air.

The procedure followed for the exposures was to cut the specimen blanks to final size, bake at 394 K (250°F) for 24 h to remove absorbed moisture, and load into the aging furnaces. The specimens were supported at their ends by narrow

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*Metallurgical Engineering Specialist.

†Engineering Chief, Product Support, Convair Division.

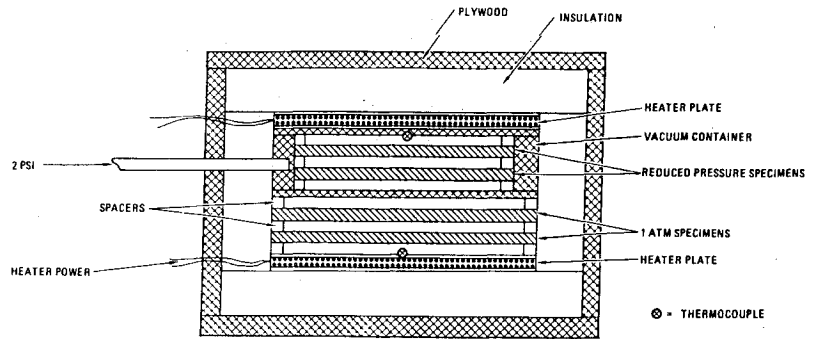


Fig. 1 Thermal aging furnace configuration.

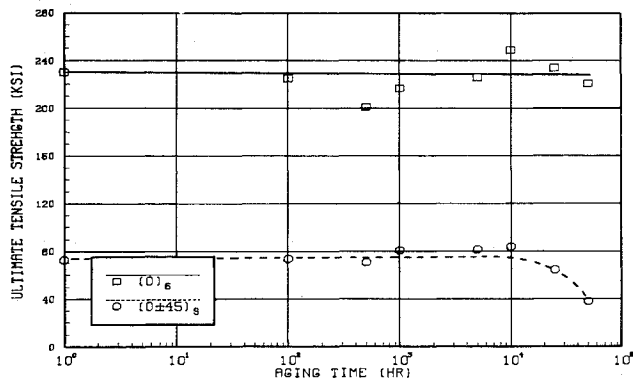


Fig. 2 Tensile strength of G/E at 450 K (350°F) after thermal aging at 394 K (250°F) in 1-atm air.

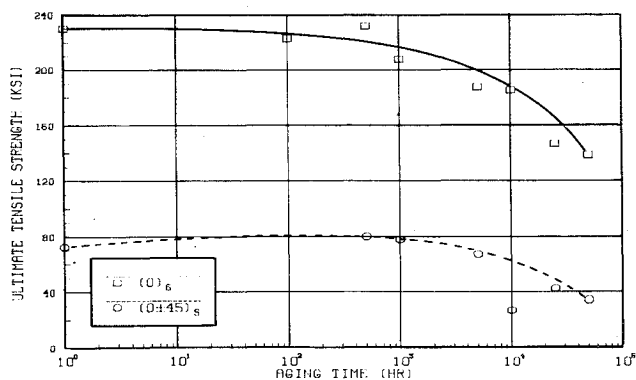


Fig. 3 Tensile strength of G/E at 450 K (350°F) after thermal aging at 450 K (350°F) in 1-atm air.

Table 1 Material systems

	G/E	G/PI
Type	A-S/3501-5	HT-S/710
Vendor	Hercules	Hercules
Orientation	$[0^\circ \pm 45^\circ]_s$	$[0^\circ \pm 45^\circ]_s$
Fiber content	67%	70%
Specific gravity	1.56	1.48

strips of stainless steel so that almost the entire surface was exposed to the air atmosphere within the furnaces. At the required time intervals, the ovens were shut down, opened, and the specimens were removed and stored in a desiccator until doubler bonding and tensile testing. Residual strength testing was performed at 450 K (350°F) for G/E, and 505 and 561 K (450 and 550°F) for G/PI. The data points shown in the residual strength-vs-time plots are averages obtained from three tensile specimens per test condition.

After tensile testing, many of the thermal aging specimens were sectioned and mounted for study using a scanning electron microscope (SEM). These studies were intended to detect changes that occurred in the composites during exposure in order to assist in identifying degradation mechanisms.

A more detailed account of the fabrication, quality assurance, thermal aging, and tensile testing procedures can be found in Ref. 1. This report includes all the material property determinations, and aging and simulation exposures up through 10,000 h.

Results and Discussion

Graphite/Epoxy

Residual tensile strength data for the G/E system have been plotted as a function of aging time in Figs. 2 and 3 for ambient pressure, and in Fig. 4 for reduced pressure. Curves for both undirectional and $[0^\circ \pm 45^\circ]_s$ crossplied specimens are included.

394 K, 1-atm (250°F, 14.7-psi) Exposure

After 25,000 h of aging, the appearance of the G/E specimens had not changed appreciably. Compared with unexposed material, the only observable differences were slight color changes. No surface damage was found. After 50,000 h, the specimen condition still appeared to be excellent. Some shallow cracking at the edges of the crossply specimens was visible, but did not appear to be of much consequence. During the cure of the doubler adhesive at 450 K (350°F), however, delamination of the 50,000-h crossplied G/E specimens occurred. The damage was not sufficient to preclude tensile testing, however.

The residual tensile strengths after aging at 394 K (250°F) and 1 atm are shown in Fig. 2. For the first 10,000 h of aging, no degradation of the 450 K (350°F) tensile properties was observed. After 25,000 h, however, an observable decrease in tensile strength of the crossply material had occurred. The effect was not large, but appeared to be a real one. Significant decreases in strength were observed for the 50,000-h specimens.

While no changes in appearance, other than slight color differences, were noticed following aging for 25,000 h, there were significant changes after tensile testing. The failed tensile coupons were markedly different than those from material that had been aged for time periods up through 10,000 h. The epoxy matrix at the surface of the 25,000-h specimens had broken up during test and crumbled off, revealing the graphite fibers in the outer plies. At failure, the graphite tows opened up giving a very fuzzy appearance to the test section of the specimens. The specimens aged at 450 K (350°F) in 1-atm air showed this effect to some degree after 5,000 h. Specimens aged 10,000 h at 450 K (350°F) and 1-atm were almost identical to the ones described above, which were aged 25,000 h at 394 K (250°F) and 1-atm. The appearance of the failed specimens is supporting evidence that the slight falloff in tensile properties after 25,000 h marks the beginning of significant degradation to the epoxy matrix specimens when aged at 394 K (250°F) in 1-atm air. After 50,000 h of ex-

posure, the failed G/E tensile specimens had lost not only the resin at the surface, but much of the matrix throughout the thickness. Portions of failed test specimens before and after aging at 394 K (250°F) are shown in Fig. 5. The change in appearance of the failed specimens between 10,000 h and 25,000 h of exposure is quite distinct.

450 K, 1-atm (350°F, 14.7-psi) Exposure

At 450 K (350°F) the G/E began to show slight color changes after 10,000 h, and a few small edge cracks after 25,000 h. Otherwise, the appearance was visually unchanged. After 50,000 h, the surface of the specimens had a burned appearance, with some loose graphite fibers at the edges. There was some slight edge cracking and indications that delamination might be starting. The specimens gave off a dead sound when dropped, and offered little resistance to bending.

During doubler bonding prior to post-exposure tensile testing, delamination in the central section between the doublers occurred in most of the crossplied specimens aged for 10,000 h or longer. Again, the damage was not sufficient to preclude tensile testing.

The residual tensile strengths after aging at 450 K (350°F) and 1-atm are shown in Fig. 3. A degradation in tensile properties combined with a significant change in appearance of the specimens after tensile testing occurred after 5000 h of exposure. The tensile results are somewhat misleading as to the actual quality of the material. The values do not indicate how badly the G/E had been degraded, particularly after 25,000 and 50,000 h of exposure. The resin matrix had retained almost no strength as a result of the aging exposure. A test that measured matrix strength—e.g., shear, compressive or transverse tensile—would have given extremely poor results. The residual tensile values, on the other hand, are 50% or more of the unexposed values even after 50,000 h of aging. Figure 6 shows the effect of aging time on the appearance of failed tensile specimens. Embrittlement of the epoxy matrix begins between 1000 and 5000 h. Large amounts of resin have crumbled and fallen away from the 5000- and 10,000-h specimens, leaving the bare graphite tows looking like bundles of yarn. For the 25,000- and 50,000-h specimens, the effect is increased and almost no resin matrix remains after tensile testing.

450 K, 0.014 MN/m² (350°F, 2-psi) Exposure

The first visible effects of the reduced pressure exposures were noted after 25,000 h, with slight color changes and a few small edge cracks. The 50,000-h specimens were not much different from the 25,000-h group, other than a somewhat burned-looking surface and slightly less ring when dropped.

Tensile results are plotted in Fig. 4. The first slight decrease in tensile strength occurred after 25,000 h, and was accompanied by a change in appearance of the failed test

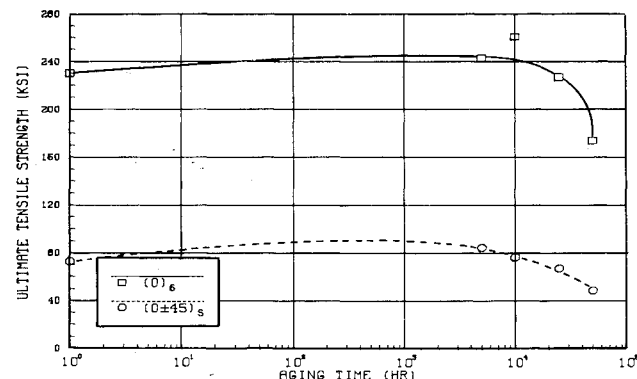


Fig. 4 Tensile strength of G/E at 450 K (350°F) after thermal aging at 450 K (350°F) in 0.014 MN/m² (2 psi) air.

specimens, indicating the beginning of matrix degradation. After 50,000 h, the tensile strength had decreased considerably, and the appearance of the failed specimens was much worse than the 25,000-h group. The resin loss and amount of graphite fibers exposed were about the same as those observed for the 5000-h, 450 K (350°F), 1-atm specimens after tensile testing. Figure 7 shows examples of the failed tensile specimens for various exposure periods. As stated, the first indication of matrix embrittlement can be seen in the 25,000-h specimen, and significant resin loss during tensile testing is visible in the 50,000-h specimen.

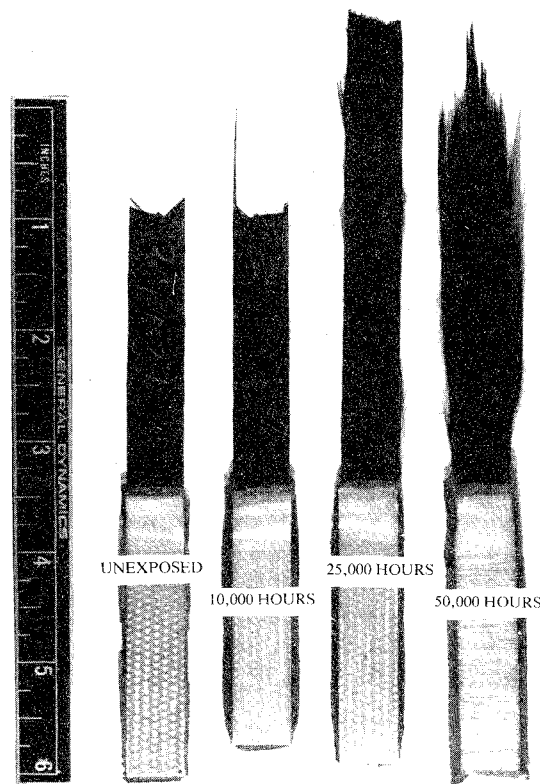


Fig. 5 G/E before and after thermal aging at 394 K (250°F) in 1-atm air and tensile testing at 450 K (350°F).

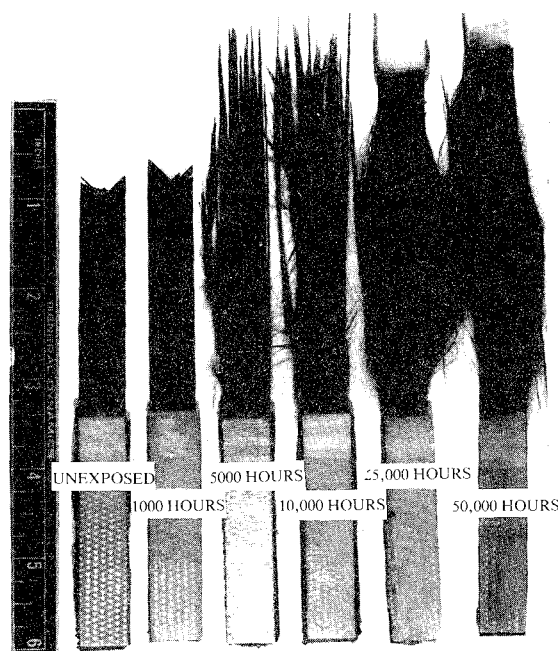


Fig. 6 G/E before and after thermal aging at 450 K (350°F) in 1-atm air and tensile testing at the same temperature.

Earlier work conducted after 10,000 h of exposure had shown that oxidation was responsible for the loss in tensile strength during thermal aging.³ For G/E, the extent of oxidation could readily be detected by metallographic techniques, especially with the SEM. Additional studies have now been made for aging time periods out to 50,000 h.

Figure 8 through 10 show the results of these examinations for crossplied specimens of G/E exposed to the three different aging conditions for 10,000, 25,000, and 50,000 h. The lighter cast, beginning at the specimen edges and spreading inward with exposure time, was shown earlier³ to be related to oxidation of the epoxy resin. The changes from dark to light were explained by the amount of relief polishing around the

individual fibers that increased with the degree of oxidation. When the epoxy resin oxidized, it was more prone to crumble, and differences in the amount of oxidation in the polished mounts could easily be detected using the SEM. The degree of oxidation, as shown in Figs. 8 and 9, is quite similar for the first two conditions, i.e., 394 K (250°F) and 1-atm air, and 450 K (350°F) and reduced pressure. Slight oxidation at the outer edges is visible after 10,000 h. At 25,000 h, considerable oxidation has occurred in the outer plies, and is beginning to extend into the second plies. The effect is somewhat more pronounced for the 450 K (350°F), 0.014 MN/m² (2 psi) exposure. After 50,000 h of aging, evidence of oxidation is visible in all the plies, with the effect again more pronounced for the 0.014 MN/m² (2 psi) specimen. The metallographic results show slight effects after 10,000 h, with major changes after 25,000 and 50,000 h. These results agree well with both the residual tensile data and the condition of the specimens after tensile testing discussed earlier. Photomicrographs of specimens exposed to the third and most severe aging condition, 450 K (350°F) and 1-atm air, are presented in Fig. 10. At 10,000 h, the effects of oxidation are visible throughout the thickness of the laminate. After 25,000 and 50,000 h, extensive matrix embrittlement has occurred. These effects

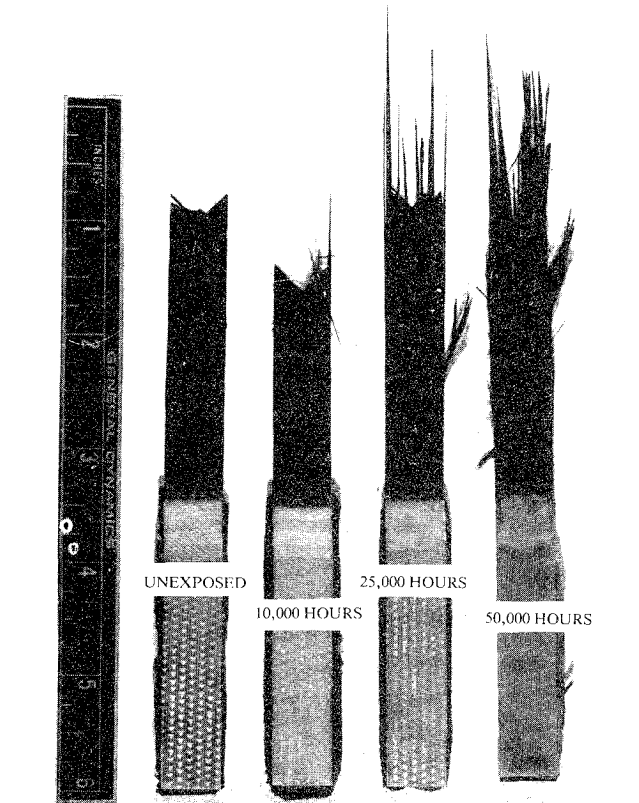


Fig. 7 G/E before and after thermal aging at 450 K (350°F) in 0.014 MN/m² (2 psi) air and tensile testing at the same temperature.

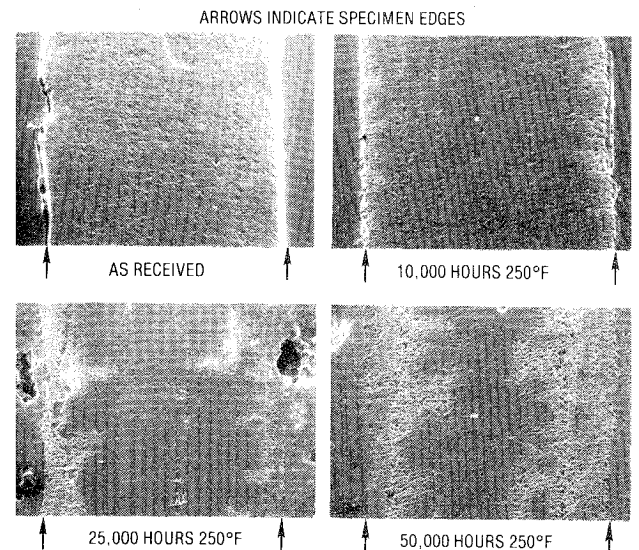


Fig. 8 Photomicrographs (SEM) of G/E after thermal aging in one-atmosphere air at indicated temperature (100X).

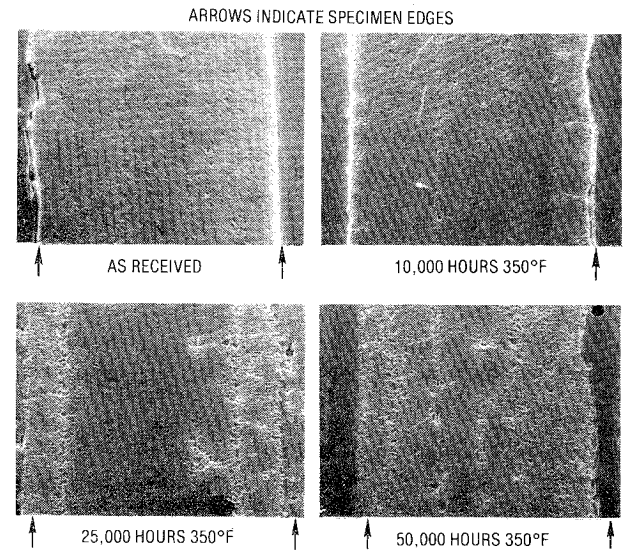


Fig. 9 Photomicrograph (SEM) of G/E after thermal aging in 0.014 MN/m² (2 psi) air at indicated temperature (100X).

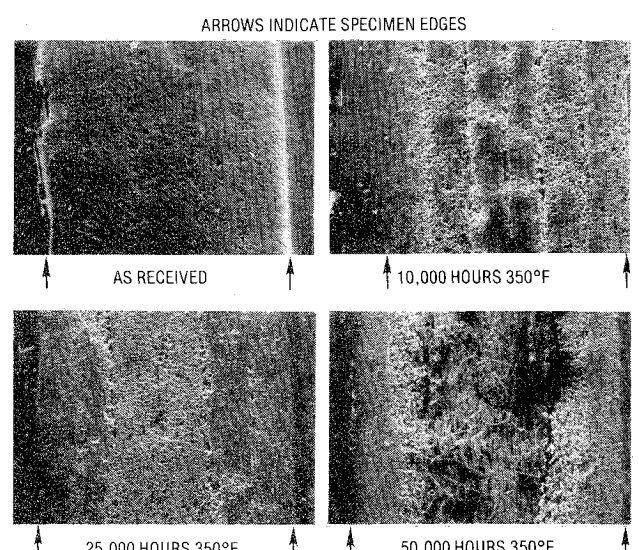


Fig. 10 Photomicrographs (SEM) of G/E after thermal aging in 1-atm air at indicated temperature (100X).

are particularly pronounced in the two middle plies, where resin loss is quite severe.

Graphite/Polyimide

Residual tensile-strength data for the G/PI system have been plotted as a function of aging time in Figs. 11 and 12 for ambient pressure, and in Fig. 13 for reduced pressure. Both unidirectional and crossplied specimens were aged at ambient pressure, whereas only longitudinal specimens were exposed at reduced pressure. Residual tensile-strength testing was performed at the same temperatures that were used for the thermal aging exposures.

505 K, 1-atm and 0.014 MN/m² (450°F, 14.7- and 2-psi) Exposures

Exposures of up to 25,000 h at both 1-atm and reduced pressure had essentially no effect on the 505 K (450°F) tensile strength (see Figs. 11 and 13). Compared with unexposed material, there was no apparent differences in appearance for the 1-atm specimens. The reduced-pressure specimens did show some small spots, but these did not appear to be either serious or significant. After tensile testing, the specimens again resembled unexposed specimens with no bare graphite fibers visible as a result of breaking up of an embrittled matrix.

After 50,000 h, significant decreases in tensile strength were found in specimens aged at both ambient and reduced pressure. Specimen condition, however, was still quite good. Before tensile testing, a small amount of edge cracking and a slightly burned appearance were observed. After tensile testing, a very slight loss of resin with a few bare graphite fibers could be seen, perhaps an indication of the start of matrix degradation. As shown in Fig. 14, however, the effects were minor.

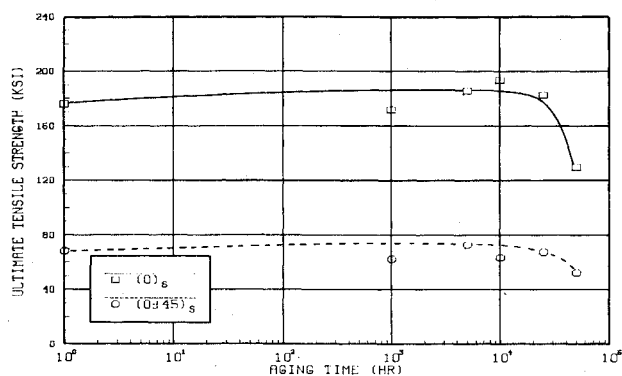


Fig. 11 Tensile strength of G/PI at 505 K (450°F) after thermal aging at 505 K (450°F) in 1-atm air.

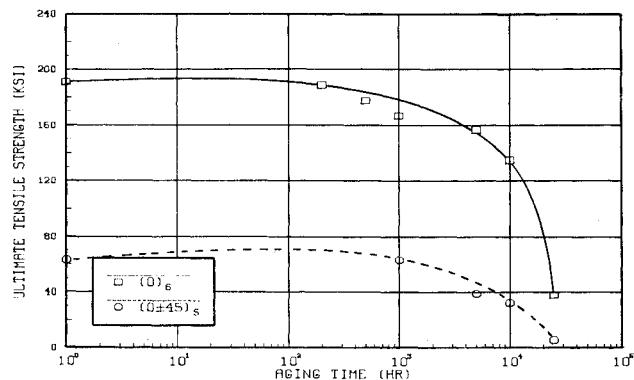


Fig. 12 Tensile strength of G/PI at 561 K (550°F) after thermal aging at 561 K (550°F) in 1-atm air.

561 K, 1-atm and 0.014 MN/m² (550°F, 14.7- and 2-psi) Exposure

In contrast to the lower temperature exposures, those at 561 K (550°F) produced significant decreases in tensile strength in times as early as 200 h for ambient pressure (Fig. 12). Although the strength decreases were quite large, very little change in the appearance of the specimens was observed after the first 10,000 h of aging. The color and surface texture remained almost unchanged. Only slight edge cracks and some small spots on the surface with a charred appearance were found. After tensile testing, the specimens aged for 10,000 h or less were almost identical to unexposed tensile specimens (Fig. 15) indicating no change in the failure mode as a result of the exposure.

A major change in specimen condition occurred after 25,000 h in 1-atm air. These specimens experienced considerable weight loss and, in the case of the crossplied material, severe edge delamination. Both orientations were warped and curved, badly discolored with a burned appearance, and were covered with orange and white spots.

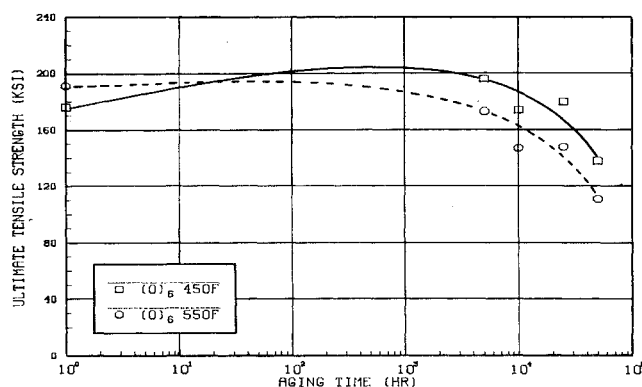


Fig. 13 Tensile strength of unidirectional G/PI at the indicated temperatures, after thermal aging in 0.014 MN/m² (2 psi) air at the same temperatures.

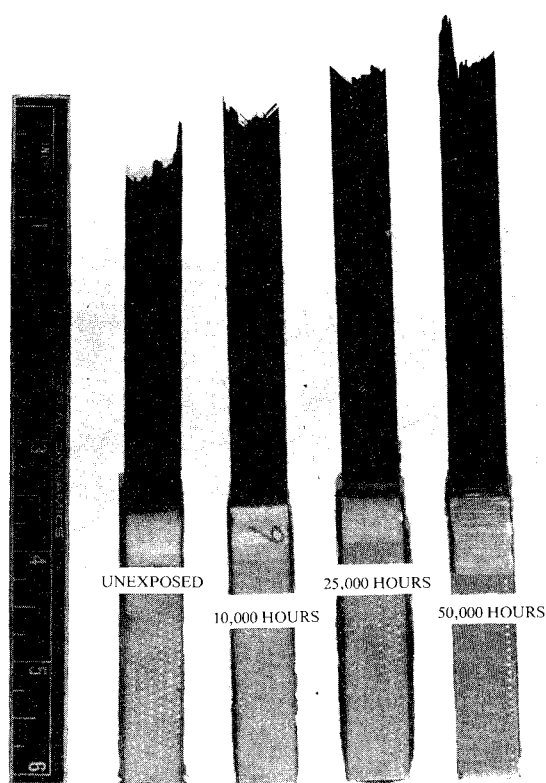


Fig. 14 G/PI before and after thermal aging at 505 K (450°F) in 1-atm air and tensile testing at the same temperature.

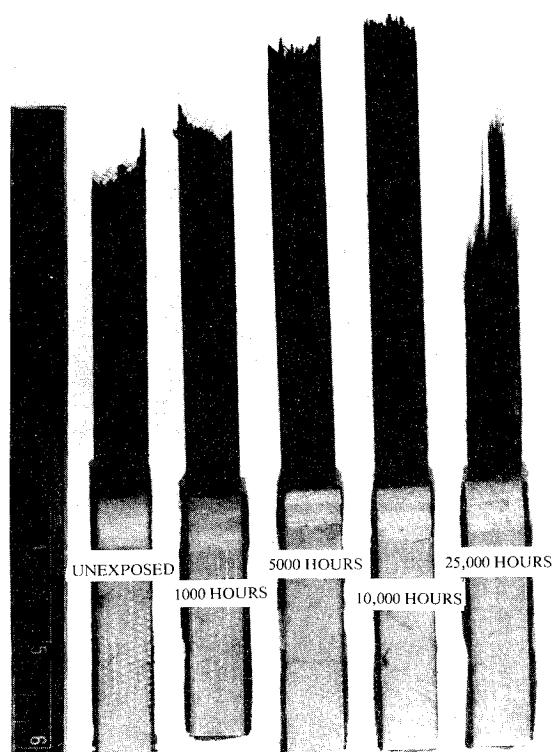


Fig. 15 G/PI before and after thermal aging at 561 K (550°F) in 1-atm air and tensile testing at the same temperature.

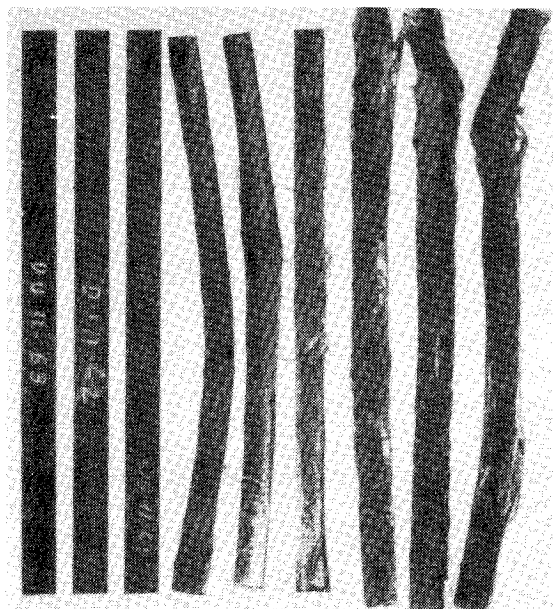


Fig. 16 G/PI after 50,000 h of thermal aging at 561 K (550°F) in 1-atm air.

Compared to the 1-atm specimens, the reduced-pressure specimens looked very good. They did, however, show some loss of resin with some exposed fibers, and were somewhat faded as compared to unexposed material. For the specimens aged in 1-atm air, 25,000 h of exposure severely degraded the tensile strength, with losses in both the crossplied and unidirectional material of 80% or more (Fig. 12). The specimens aged at reduced pressure showed approximately the same tensile strength (Fig. 13) as those aged for 10,000 h (75-80% of that for unaged material). After tensile testing, the 1-atm specimens were markedly different from those of material that had been aged for time periods up through 10,000 h. The polyimide matrix had broken up during test and

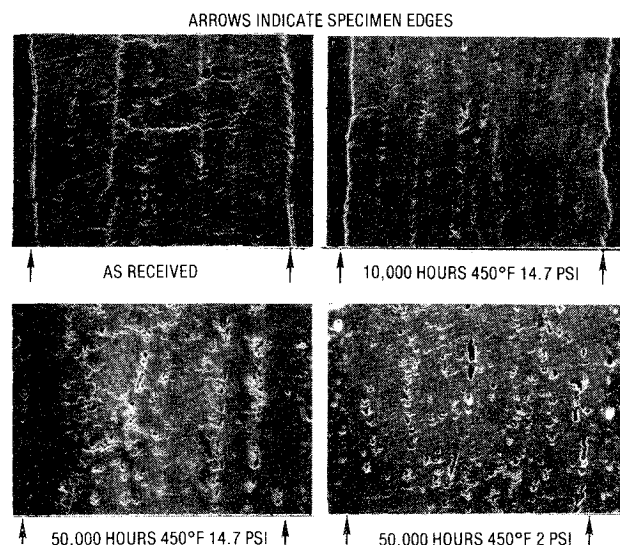


Fig. 17 Photomicrographs (SEM) of G/PI after thermal aging at indicated conditions (100X).

crumbled off revealing the graphite fibers. At failure, the graphite tows opened up and resembled pieces of yarn. Figure 15 shows the change in appearance in failed-tensile specimens after 25,000 h of exposure at 561 K (550°F). The reduced-pressure specimens were discolored after tensile testing, but showed very little resin loss or exposed graphite fibers.

After 50,000 h of aging in 1-atm air, almost no resin was left in the specimens, only graphite fibers. The specimens were badly warped and delaminated, and were no longer suitable for tensile testing. Figure 16 shows these specimens and the reduced-pressure specimens that were in considerably better condition. The reduced-pressure specimens were discolored, and showed some loss of resin and exposed surface fibers, but could be tested. The residual tensile strength, as shown in Fig. 13, was slightly less than 60% of the unexposed value. The failed specimens showed evidence of the beginning of matrix embrittlement, with loss of the matrix material and numerous exposed graphite fibers.

The results of the metallographic examinations of the thermal aging specimens of G/PI are shown in Figs. 17-19. The changes in appearance as a result of the thermal exposures were far less obvious for the G/PI system than for the G/E system. For specimens aged at 505 K (450°F), the only indication of matrix embrittlement was observed in specimens exposed for 50,000 h in 1-atm air. This specimen, Fig. 17, shows more relief polishing around the graphite fibers, more porosity, and more fiber-matrix separation than the specimens aged for shorter times or a reduced pressure. The effects, particularly the relief polishing, are more pronounced in the center than at the edges. Visual effects starting at the edges and moving inward, as seen in the G/E system, were not observed for the G/PI system. The effect of oxygen pressure can readily be seen, however, as evidenced by the two 50,000-h specimens in Fig. 17. The start of matrix degradation, as determined by the condition of the 50,000-h, 1-atm specimens after tensile testing, appears to be confirmed by the metallographic results.

Raising the aging temperature to 561 K (550°F) greatly increased the degree of matrix degradation of the G/PI system. Comparison of Figs. 17 and 18 shows that in 1-atm air, 10,000 h at 561 K (550°F) has a slightly greater effect on the microstructure than 50,000 h at 505 K (450°F). The amount of relief polishing around the graphite fibers is more pronounced, while the porosity and fiber-matrix separation are about the same. After 25,000 h at 561 K (550°F) in air, the matrix is severely embrittled with considerable loss of resin from the center plies. The final photomicrograph in Fig. 18,

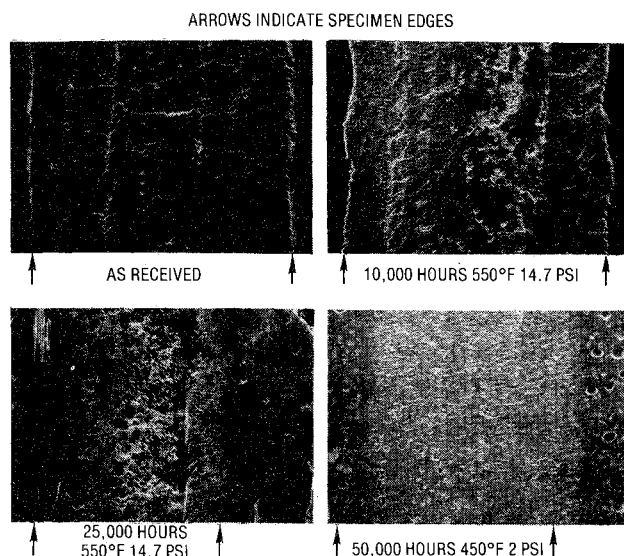


Fig. 18 Photomicrographs (SEM) of G/PI after thermal aging at indicated conditions (100X).

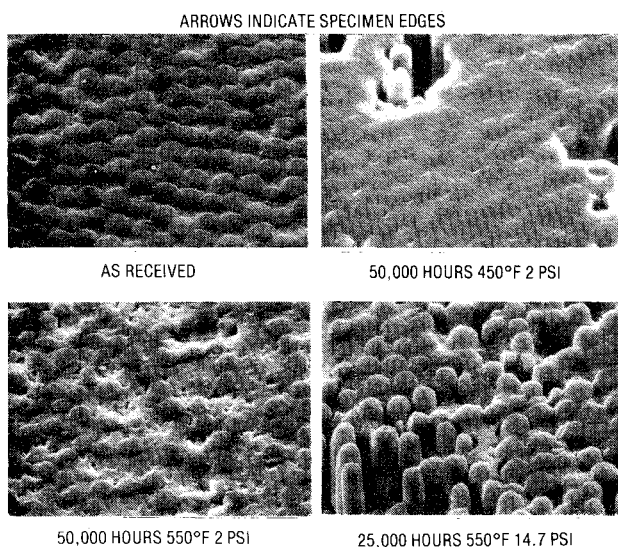


Fig. 19 Photomicrographs (SEM) of G/PI after thermal aging at indicated conditions (1,000X).

50,000 h at reduced pressure, shows little effect to the matrix. At higher magnification, however, (Fig. 19) considerable relief polishing has occurred. This metallographic evidence of the onset of matrix embrittlement agrees well with the tensile data and the appearance of the failed tensile specimens. The remaining photomicrographs in Fig. 19 clearly show the effect of matrix condition on the degree of relief polishing around the graphite fibers.

Conclusions

For the G/E system, thermal aging at 394 K (250°F) and 1-atm pressure produced no effects for the first 10,000 h. Matrix degradation began between 10,000 and 25,000 h, and was severe after 50,000 h. The fiber-controlled tensile properties, however, showed almost no change. Aging at 450 K (350°F) and 1-atm pressure was more damaging, with matrix degradation beginning between 1000 and 5000 h. After 5000 h, the matrix was severely embrittled and crumbled away during tensile testing, leaving many bare fibers. Tensile properties were considerably reduced for aging times of 5000

h or longer. Reduced pressure exposures at 450 K (350°F) delayed the effects, but did not eliminate them.

The G/PI system survived 25,000 h of thermal aging at 505 K (450°F) and 1-atm pressure with no effects. Some decrease in tensile strength was measured after 50,000 h, but matrix degradation was not observed. At 1-atm pressure, raising the aging temperature to 561 K (550°F) reduced the time at which tensile strength decreases were observed to 10,000 h. In like manner to the 505 K (450°F) exposures, this initial fall off in tensile strength was not accompanied by matrix embrittlement. After 25,000 h, the material was partially delaminated and showed high weight loss. The tensile strength was greatly reduced and severe matrix embrittlement had occurred. Degradation after 50,000 h was such that the specimens could not be tested. Again, aging effects were less severe for exposures conducted in a reduced pressure environment.

Matrix degradation by oxidation was shown to be the primary cause of mechanical property losses during thermal aging. For G/E, the extent of oxidation could readily be detected by metallographic techniques, especially with the SEM. Similar studies of G/PI revealed increased porosity and fiber-matrix separation accompanied by numerous fine cracks at the fiber-matrix interface. However, visual effects starting at the edges and moving inward as seen in the G/E system were not observed for the G/PI system.

Current Studies

The results of this program have shown that the loss in mechanical properties of G/E and G/PI advanced composites during thermal aging are related to both degradation of the resin matrix and, to a less extent, the graphite reinforcing fiber. Since tensile strength is a fiber-dominated property, a post-exposure tensile test was probably not the best choice for evaluating the effects of thermal aging. As discussed earlier, the relatively high residual tensile strengths obtained after many of the exposures were somewhat misleading as to the actual quality of the material. A test that measured the matrix strength would undoubtedly have given results more indicative of the degree of material degradation.

To better understand the effects of long-term thermal exposure on mechanical properties that are highly matrix dependent, an additional program was initiated. This thermal aging study is being conducted at 373 and 394 K (212 and 250°F) for G/E, and 450 and 505 K (350 and 450°F) for G/PI. The exposures, all at 1 atm, are for 1000, 5000, 10,000 and 25,000 h. Post-aging evaluation consists of weight-change measurements; room-temperature compressive, interlaminar shear, and tension-compression fatigue tests, glass transition temperature tests; and optical and SEM examinations. Insufficient data are available at this time to be included in this paper.

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