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EFFECTS OF TEMPERATURE AND HUMIDITY CYCLING ON THE STRENGTHS OF
TEXTILE REINFORCED CARBON/EPOXY COMPOSITE MATERIALS

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ABSTRACT

Results are presented from an experimental evaluation of the combined effects of temperature and humidity cycling on AS4/3501-6 composites (unstitched, Kevlar 29 stitched, and S-2 glass stitched uniweave fabric) and AS4/E905L composites (2-D, S-2 glass stitched 2-D, and 3-D braided fabric). The AS4/3501-6 uniweave material had a quasi-isotropic layup, whereas the AS4/E905L materials were braided in a $(\pm 30^\circ/0^\circ)_s$ orientation. Data presented include compression strengths and compression-compression fatigue results for uncycled composites and cycled composites (160, 480, 720, and 1280 cycles from 140°F at 95 percent relative humidity to -67°F). To observe the presence of microcracking within the laminates, photomicrographs were taken of each material type at the end of each cycling period. Microcracks were found to be more prevalent within stitched laminates, predominantly around individual stitches. The glass stitched laminates showed significant microcracking even before cycling. Less microcracking was evident in the Kevlar stitched materials, whereas the unstitched uniweave material developed microcracks only after cycling. The 3-D braid did not develop microcracks. The static compression strengths of the unstitched and Kevlar stitched uniweave materials were degraded by about 10% after 1280 temperature/ humidity cycles, whereas the reduction in compression strength for the glass stitched uniweave was less than 3%. The reduction in compression strength for the glass stitched 2-D braid was less than 8%. The unstitched 2-D and 3-D braids did not lose strength from temperature/ humidity cycling. The compression-compression fatigue properties of all six material types were not affected by temperature/ humidity cycling.

INTRODUCTION

Textile composite materials which incorporate through-the-thickness (TTT) reinforcement (stitching, braiding, or weaving) have demonstrated excellent damage tolerance (references 1, 2, and 3), an important criteria for their use in primary aircraft structures. The development of microcracking in or near stitching threads of textile reinforced composites is an issue of concern. These microcracks are caused by a mismatch in thermal expansion coefficient of the resin and fibers during cool-down from fabrication temperature. The potential for increased microcracking as a result of thermal and moisture cycling during service is an additional concern. The extent to which the presence and growth of these microcracks affects material performance is, therefore, an area of great interest. Thus, a need exists to determine the effects of temperature and humidity cycling on the mechanical properties and fatigue response of textile composites.

This study presents the results of an experimental evaluation of the effects of temperature and humidity cycling on stitched and unstitched AS4/3501-6 uniweave and braided AS4/E905L composite materials. Data presented include compression strength and compression-compression fatigue results for uncycled and cycled (160, 480, 720, and 1280 temperature/ humidity cycles from 140°F at 95% relative humidity to -65°F) quasi-isotropic uniweave and ($\pm 30^\circ/0^\circ$)_S braided laminates. To observe the presence and development of microcracks within the laminates, photomicrographs were taken of each material type before and at the end of each cycling period.

MATERIALS

Uniweave fabric made from Hercules AS4 carbon fiber was used to make 32 ply quasi-isotropic (0/45/90/-45)_{4S} preforms. These preforms were then stitched (3/16 inch rows by 1/8 inch step along the 0° direction) with Kevlar 29 (1000 denier thread) and with S-2 glass (449-1500 thread). All stitching was performed using a 2-end 200 denier twisted Kevlar 29 needle thread. Unstitched, Kevlar 29 stitched, and S-2 glass stitched composite laminates were then fabricated by McDonnell Douglas Aircraft with Hercules 3501-6 epoxy resin using resin transfer molding (RTM). The unstitched and Kevlar stitched uniweave composite materials were approximately 0.18 inches thick, whereas the glass stitched uniweave laminates were approximately 0.19 inches thick. Unstitched and S-2 glass stitched 2-D fabric, braided by Fiber Innovations, and 3-D fabric, braided by Atlantic Research, were also fabricated into composite panels. Using RTM, Fiber Innovations fabricated the three braided panel types with British Petroleum E905L epoxy resin. All of the braided laminates were around 0.24 inches thick.

TEST PROCEDURES

To simulate the extremes of conditions that a primary structural component on a commercial transport aircraft may be expected to encounter, the test materials were cycled between 140°F, 95% relative humidity and -65°F, no humidity (figure 1) in a programmable temperature/ humidity chamber. The specimens were exposed to 16 cycles per day.

A 120-kip hydraulic testing machine was used to determine the compression properties of the materials studied. All compression values were determined from 1.5-inch wide by 1.75-inch long specimens (3 replicates per test) using the short-block fixture shown in figure 2. Load was applied at a displacement rate of 0.05 in./min. Baseline room temperature (RT) properties were determined from as-fabricated specimens with a moisture content resulting from normal laboratory exposure. Specimens were weighed prior to cycling and upon removal from the chamber to determine the increase in weight due to water absorption. Cycled specimens were strain-gaged immediately upon removal from the temperature/ humidity cycling chamber and tested at room temperature. All compression testing was performed at NASA Langley.

Fatigue properties were determined using the fatigue fixture shown in figure 3. All the fatigue properties were determined using 1-inch wide by 4-inch long test coupons (6 to 8 specimens for each material at each testing condition), a maximum/ minimum stress ratio of 10 and a frequency of 5 hertz. The uniweave fatigue specimens were dried to their RT-equilibrated weight prior to testing. The braided fatigue specimens, however, were tested with their full moisture content from temperature/ humidity cycling (because prior compression testing had shown minimal effect from moisture as well as a lack of material which prevented the planning of a further moisture testing). All fatigue testing was performed at RT at Virginia Polytechnic Institute and State University.

RESULTS AND DISCUSSION

Compression Results

AS4/3501-6 Uniweave

The compression results for the AS4/3501-6 uniweave materials are presented in table 1 and plotted in figures 4 and 5. In figure 4, the RT baseline compression strengths are compared to the compression strengths obtained from cycled specimens. Figure 5 presents the percent retention of baseline strength for the three uniweave materials as a function of temperature/ humidity cycles. All three materials showed a decrease in compression strength compared to the RTD strengths as a result of temperature/ humidity cycling. The unstitched and Kevlar stitched material had initial decreases of 6.4 and 4.8 percent after 160 temperature/ humidity cycles. Although the strength retention continued to decrease with temperature/ humidity cycling, the reductions appear to be leveling off at 1280 temperature/ humidity cycles. The unstitched material and Kevlar stitched uniweave material retained 89.7% and 90.4% of their baseline strengths, respectively, after 1280 cycles. The glass stitched material had an initial reduction in compression strength of 2.5 percent at 160 temperature/ humidity cycles and no further reduction with additional cycling. The glass stitched uniweave material had a lower RTD compression strength, but it retained 97.6% of its compression strength after 1280 cycles. Since the properties of 3501-6 are known to be adversely affected by moisture, the water absorption (table 1) of these materials is most likely the cause of the decrease in compression strength for the unstitched and Kevlar stitched materials. Even though the glass stitched uniweave absorbed similar amounts of water as the Kevlar stitched material, the compression strengths were not degraded. The initiation of failure in the glass stitched material may be dominated by a mechanism which is insensitive to moisture.

The moduli of the uniweave materials (table 1) were not affected by temperature/ humidity cycling which is not surprising since modulus is a fiber-dominated property. All the modulus values for each material are essentially the same within the scatter of the data.

AS4/E905L Braids

The compression results for the AS4/E905L braided materials are presented in table 2 and plotted in figures 6 and 7. In figure 6, the RT baseline compression strengths from reference 4 are compared to the compression strengths obtained from cycled specimens. As shown in figure 6, only the stitched 2-D braided material showed a reduction in compression strength. Although it only retained 91.7% of the RTD compression strength, the scatter in the data (standard deviations of 4 to 6 %) is large enough to suggest that the reduction may not be significant. The unstitched 2-D and 3-D braids were not affected by the temperature/humidity cycling, retaining 99% percent and 100%, respectively, of their RTD compression strength after 1280 cycles. These results are not surprising since the moisture absorption of the AS4/E905L braided material (table 2) was significantly lower than that for the AS4/3501-6 uniweave materials. As for the uniweave materials, the moduli of the braided materials (table 2) were not affected by temperature/ humidity cycling. All the modulus values for each material are essentially the same within the scatter of the data.

Compression-Compression Fatigue

AS4/3501-6 Uniweave

The fatigue data obtained for the three uniweave materials are presented in figures 8-11. The trends of the data are represented by logarithmic regression curves calculated for each material. Although the glass stitched material did have slightly lower fatigue properties compared to the unstitched material (figure 8), on the whole stitching did not significantly affect the fatigue properties of the AS4/3501-6 uniweave. This result agrees with previous fatigue data (references 5 and 6) on stitched uniweave material which also showed no adverse affects from stitching. All three materials showed a reduction in compression strength with cycling (reductions of approximately 50% after 1 million fatigue cycles).

Figures 9 through 11 compare the fatigue properties of the cycled uniweave specimens to the baseline (0 temperature/ humidity cycles) data. As shown in figure 9, temperature/ humidity cycling did not significantly affect the fatigue properties of the unstitched laminates. The glass stitched material, figure 10, similarly appeared unaffected by temperature/ humidity cycling. The Kevlar stitched material, figure 11, also did not appear to be adversely affected by temperature/ humidity cycling. Overall, the compression strengths of all the uniweave materials were reduced to around 50 ksi after 1 million fatigue cycles.

AS4/E905L Braids

The fatigue data obtained for the three braided materials are presented in figures 12 through 15. The baseline (0 temperature/ humidity cycles) fatigue results (figure 12) indicate that the 3-D braid showed better fatigue response in terms of ultimate stress with fatigue cycles than the 2-D and stitched 2-D braids which showed very similar results. As evidenced in figures 13, 14 and 15, temperature/ humidity cycling did not affect the fatigue response of the braided materials. The compression strengths of the braided materials were reduced by 40-45% after 1 million fatigue cycles.

Microcracking

AS4/3501-6 Uniweave

Photomicrographs of each of the three uniweave materials are presented in figures 16-18, respectively. For each material type, a photomicrograph is presented for an uncycled specimen and a specimen after 1280 temperature/ humidity cycles. For the stitched materials, one individual stitch is shown in each photomicrograph. The unstitched material, figure 16, did not show any microcracks in the uncycled specimens. Microcracking between some of the outer plies did develop after temperature/ humidity cycling. It is interesting to note that cracks only developed on one side of the laminate, indicating a possible dependence on the temperature gradients developed within the specimens as they were heated or cooled in the temperature/ humidity environmental simulation chamber.

As shown in figure 17, the glass stitched material had significant microcracking around each individual stitch. These microcracks did not appear to grow with temperature/ humidity cycling as evidenced by the photomicrograph of the glass stitched specimen after 1280 cycles. The microcracks in the specimen with 1280 temperature/ humidity cycles appear to be similar in severity to the uncycled specimen. The severity of these microcracks may dominate the initiation

of failure of these specimens which would explain the lower baseline compression strength compared to other uniweave materials as well as the constant compression strength with increasing moisture content (see figure 4 and table 1). The Kevlar stitched material, figure 18, also showed microcracking around the stitching but not to the degree that the glass stitched material microcracked. Similar to the glass stitched material findings, the microcracks in the Kevlar stitched material did not appear to increase in severity with temperature/ humidity cycling.

AS4/E905L Braids

Photomicrographs for the braided materials are presented in figures 19-21. Similar to the glass stitched uniweave, the glass stitched 2-D braid (figure 19) suffered from significant microcracking around the stitches before and after cycling. The unstitched 2-D material, figure 20, and the 3-D braided material, figure 21, did not appear to develop any microcracking from temperature/ humidity cycling.

Implications

Although the elimination of microcracking is a desirable goal, it appears that their presence in at least these particular textile reinforced composite materials is not detrimental to their potential use in terms of strength. As evidenced by the glass and Kevlar stitched laminates, microcracking did not appear to increase in severity nor did the compression properties of these materials significantly degrade (some effect on compression for the Kevlar stitched materials; no effect on fatigue) after temperature/ humidity cycling. In addition, it does appear possible, by appropriate selection of reinforcement geometry and matrix material, to avoid microcracking altogether (e.g. as was observed to be the case for the 2-D and 3-D AS4/E905L braided composites). The next phase of this work will address the effects of increasing moisture content without temperature cycles as well as the effects of temperature cycling alone on the strengths of textile composites.

CONCLUSIONS

The effects of temperature/ humidity cycling on the mechanical properties, fatigue response, and microcracking of textile reinforced AS4/3501-6 composites (unstitched, Kevlar 29 stitched, and S-2 glass stitched uniweave fabric) and AS4/E905L composites (2-D, S-2 glass stitched 2-D, and 3-D braided fabric) were investigated. Compression strengths and compression-compression fatigue behavior were determined for cycled and uncycled quasi-isotropic uniweave and ($\pm 30^\circ/0^\circ$) braided laminates. The results obtained in this investigation support the following conclusions:

1. Temperature/ humidity cycling reduced the static compression properties of the unstitched and stitched AS4/3501-6 up to 10 percent.
2. Temperature/ humidity cycling reduced the compression properties of the AS4/E905L 2-D stitched braid by 8 percent but did not affect the compression properties of the unstitched 2-D and 3-D braids.
3. Compression-compression fatigue properties for all the materials were not significantly affected by temperature/ humidity cycling.
4. Microcracks were predominant around individual stitches in both the stitched uniweave and braided materials.

5. Microcracks around glass stitches were more predominant than for Kevlar stitches.
6. The presence of microcracks does not appear to be a significant concern in terms of the compression and compression-compression fatigue properties after temperature/ humidity cycling of the textile composite materials studied.

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Table 1. Averaged Properties for AS4/3501-6 Uniweave.

Material	Number of Cycles	Water Absorption, wt%	Compression Strength, ksi	Modulus, Msi
Unstitched	0	0.000	96.7 ± 2.0*	6.76 ± .67
	160	0.169	90.5 ± 1.5	6.48 ± .09
	480	0.251	95.2 ± 3.1	6.47 ± .08
	720	0.357	88.4 ± 2.1	6.46 ± .02
	1280	0.510	86.8 ± 1.8	6.47 ± .05
Glass Stitched	0	0.000	80.9 ± 0.8	6.28 ± .44
	160	0.300	78.9 ± 0.5	6.13 ± .12
	720	0.617	79.2 ± 1.4	6.03 ± .13
	1280	0.783	79.0 ± 0.8	5.75 ± .12
Kevlar Stitched	0	0.000	92.1 ± 1.0	6.34 ± .28
	160	0.231	87.7 ± 1.8	6.48 ± .07
	720	0.583	84.3 ± 2.0	6.37 ± .16
	1280	0.748	83.3 ± 1.0	6.14 ± .11

* ± indicates standard deviation

Table 2. Averaged Properties for AS4/E905L Braids.

Material	Number of Cycles	Water Absorption, wt%	Compression Strength, ksi	Modulus, Msi
2-D Braid	0	0.000	58.6 ± 1.0*	8.8 ± .5
	160	0.091	56.0 ± 0.7	8.4 ± .5
	480	0.122	58.6 ± 1.2	8.8 ± .7
	1280	0.100	58.0 ± 2.2	8.2 ± .5
	0	0.000	55.5 ± 2.3	8.3 ± .5
2-D Stitched	160	0.158	50.2 ± 1.9	7.8 ± 1.0
	480	0.175	52.1 ± 1.3	8.8 ± .8
	1280	0.143	50.9 ± 2.9	7.4 ± .6
	0	0.000	64.8 ± 1.9	7.3 ± .6
3-D Braid	160	0.097	67.4 ± 5.8	9.9 ± 1.0
	480	0.176	70.1 ± 1.5	8.7 ± .3
	1280	0.206	68.4 ± 1.5	8.7 ± .7

* ± indicates standard deviation

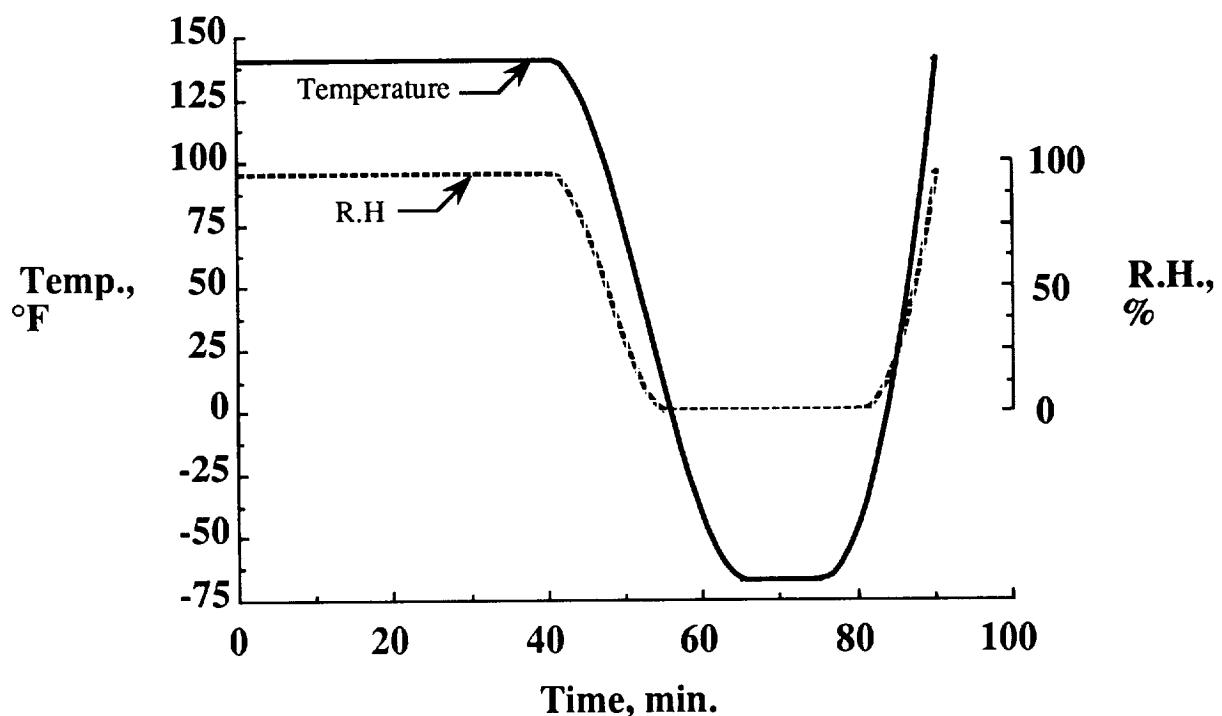


Figure 1. Temperature/ humidity cycle.

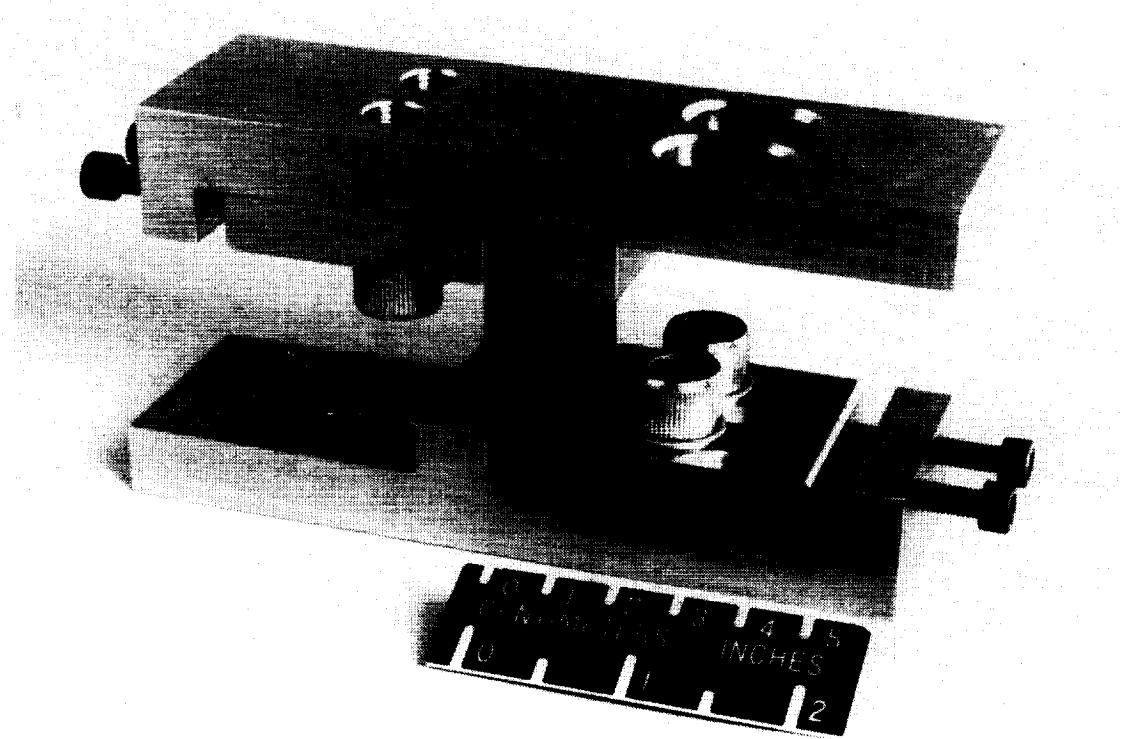


Figure 2. Short-block compression fixture.

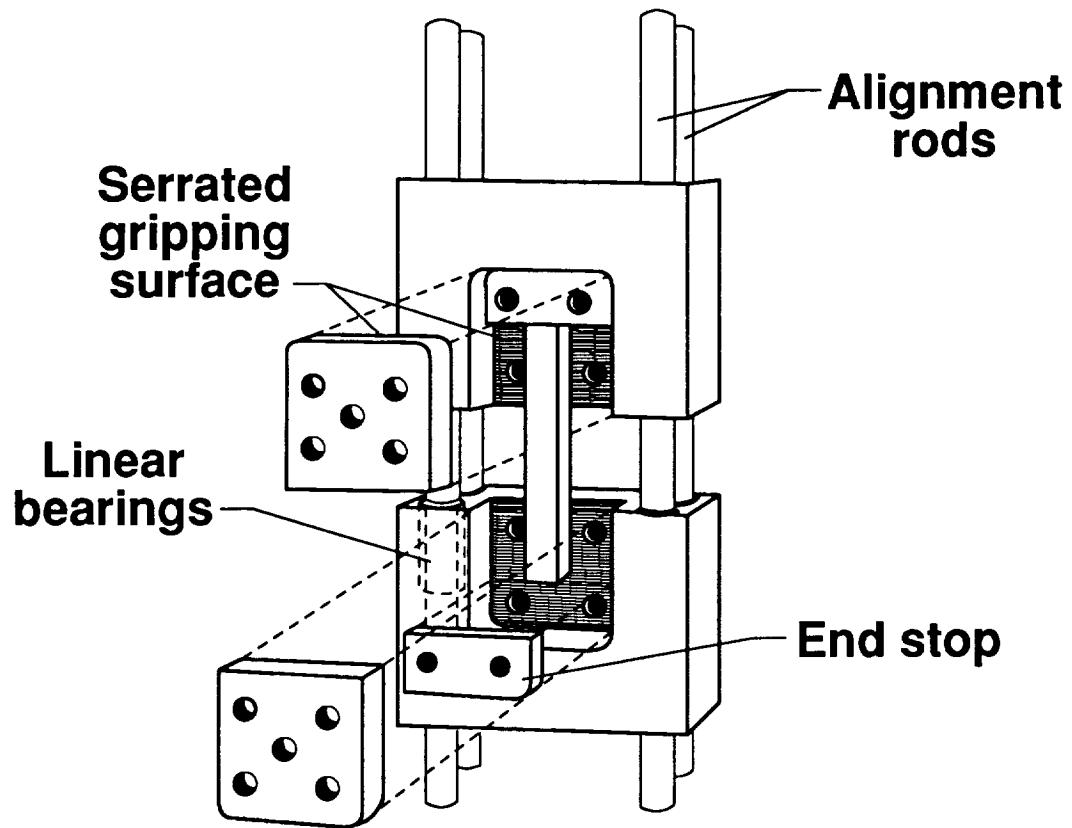


Figure 3. Compression-compression fatigue test fixture.

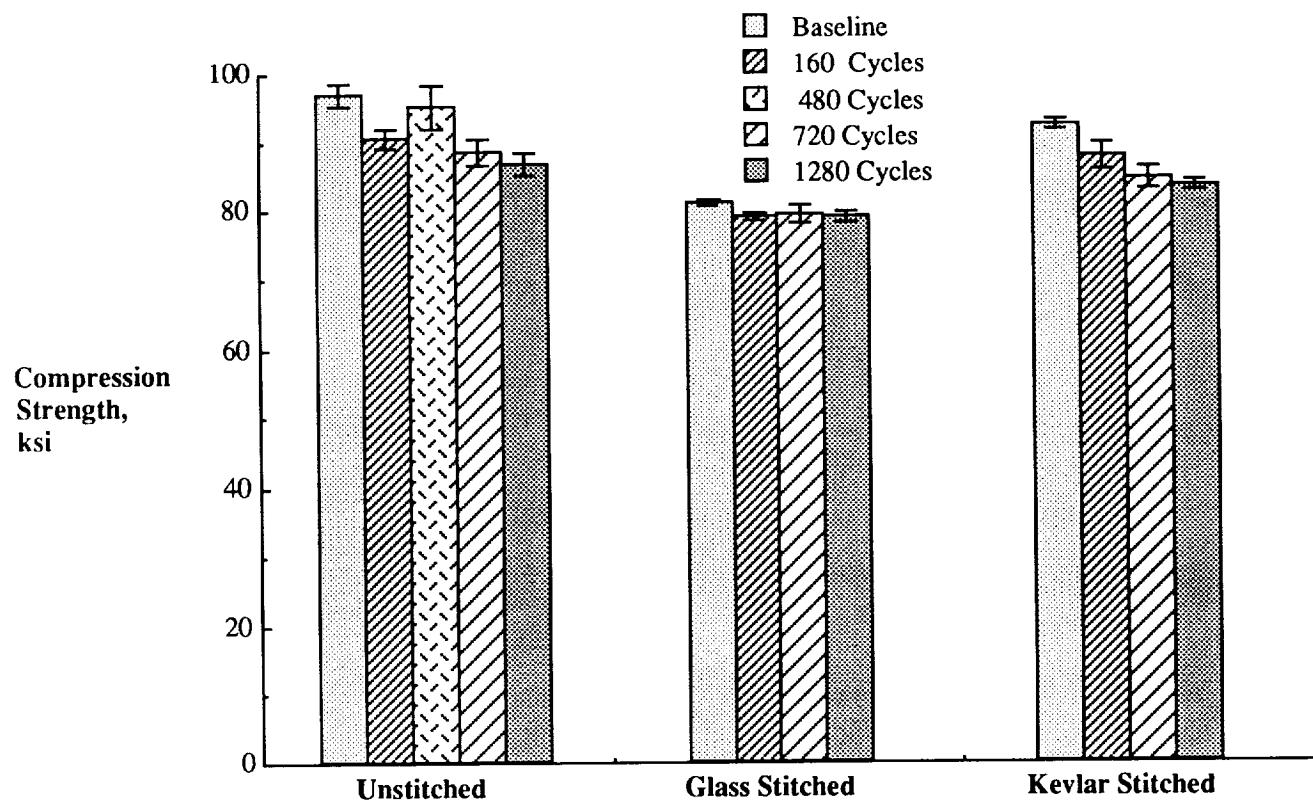


Figure 4. Compression strength for uncycled and cycled AS4/3501-6 uniweave.

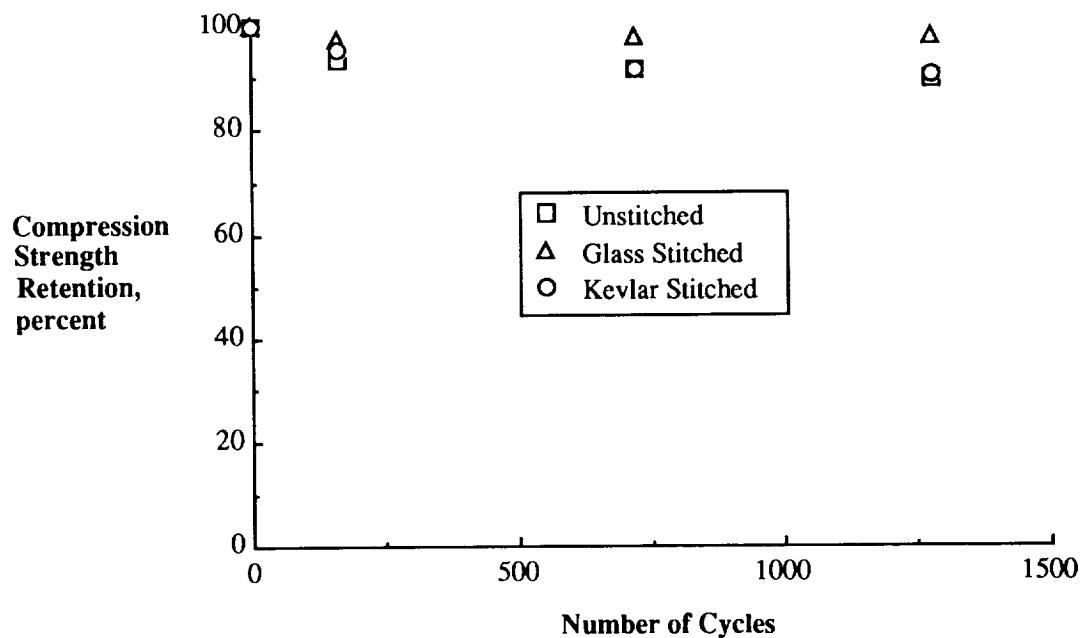


Figure 5. Strength retention of AS4/3501-6 uniweave composites.

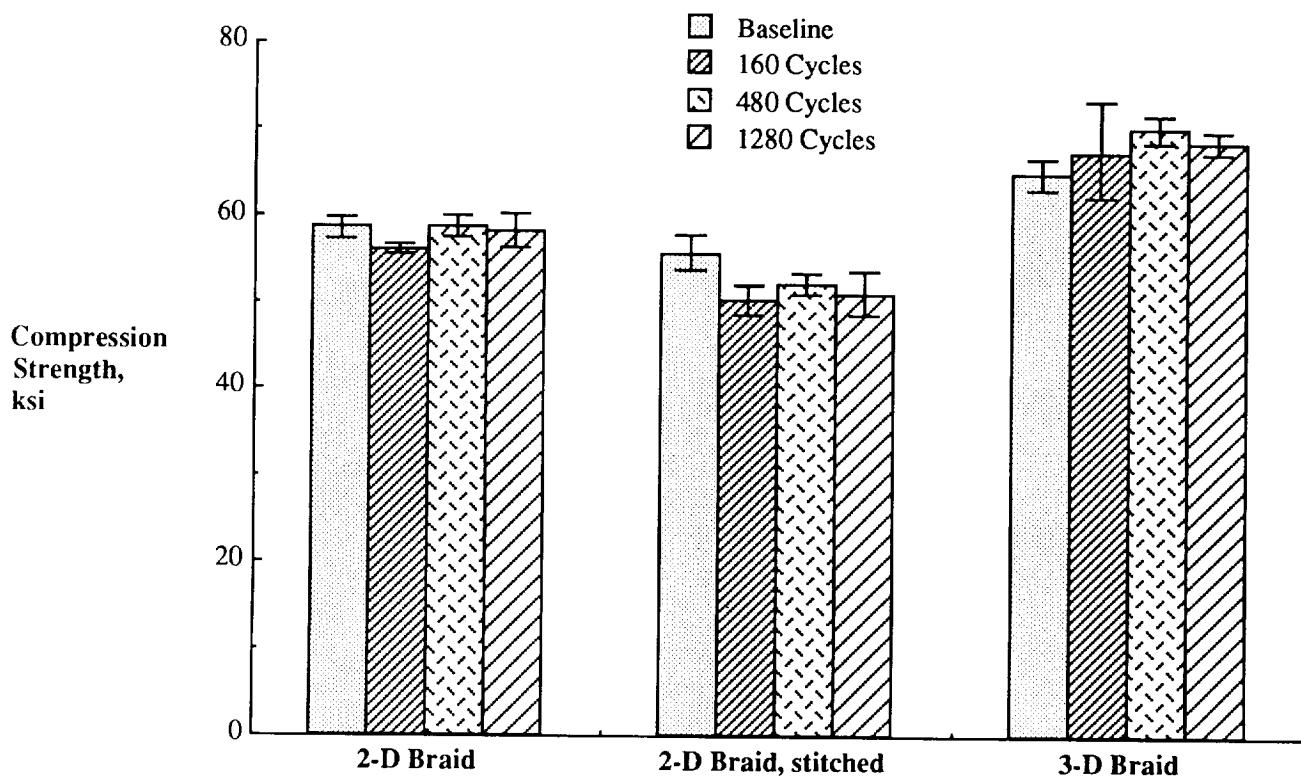


Figure 6. Compression strength for cycled and uncycled AS4/E905L braids.

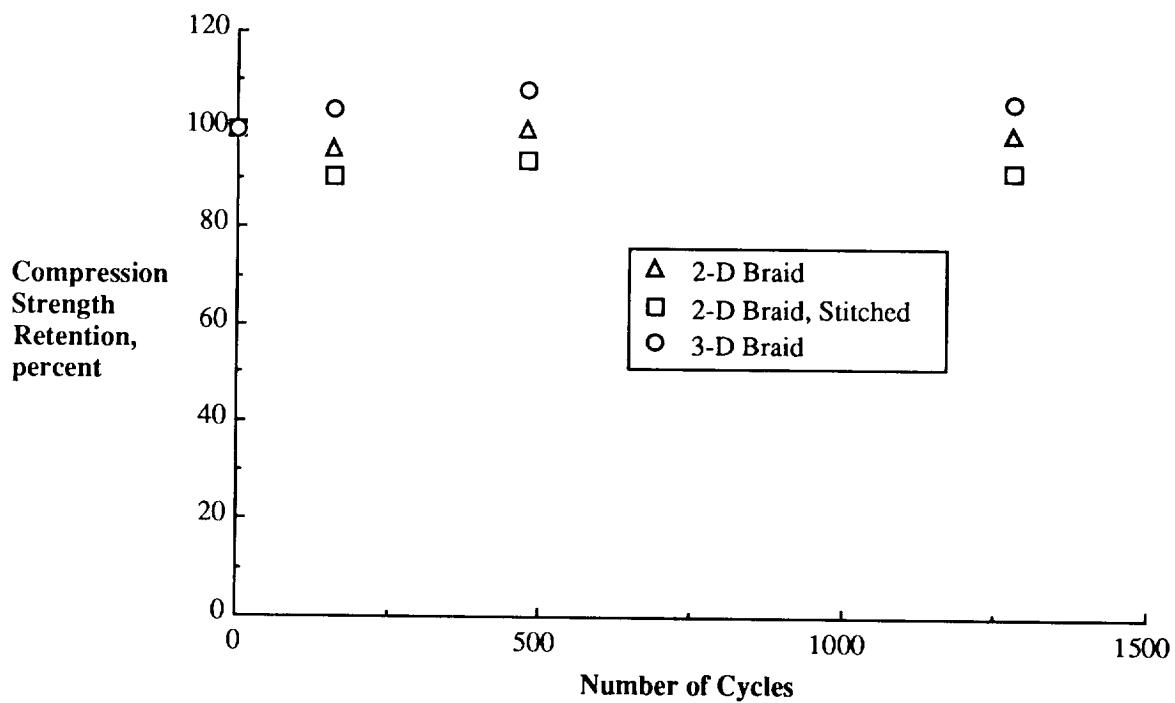


Figure 7. Strength retention of AS4/E905L braided composites.

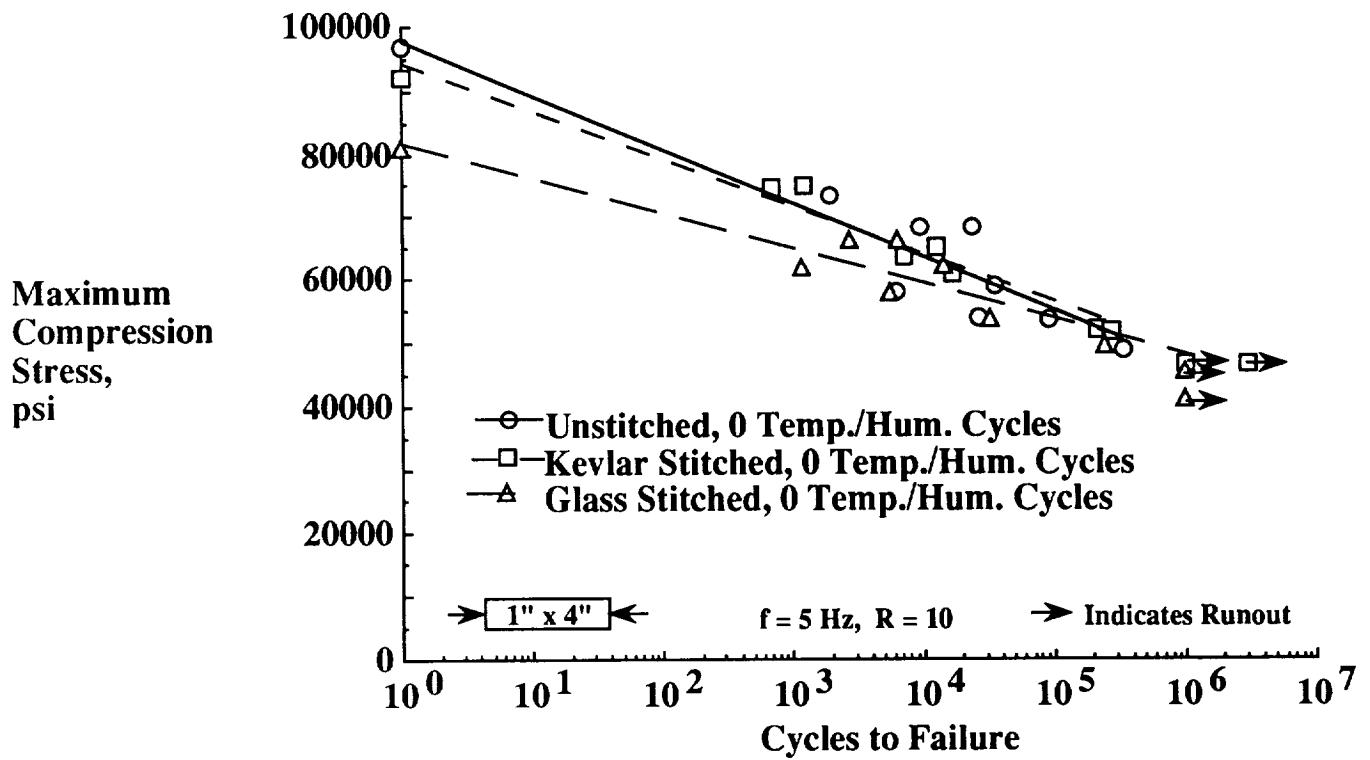


Figure 8. Fatigue data for AS4/3501-6 uniweave laminates without temperature/humidity cycles.

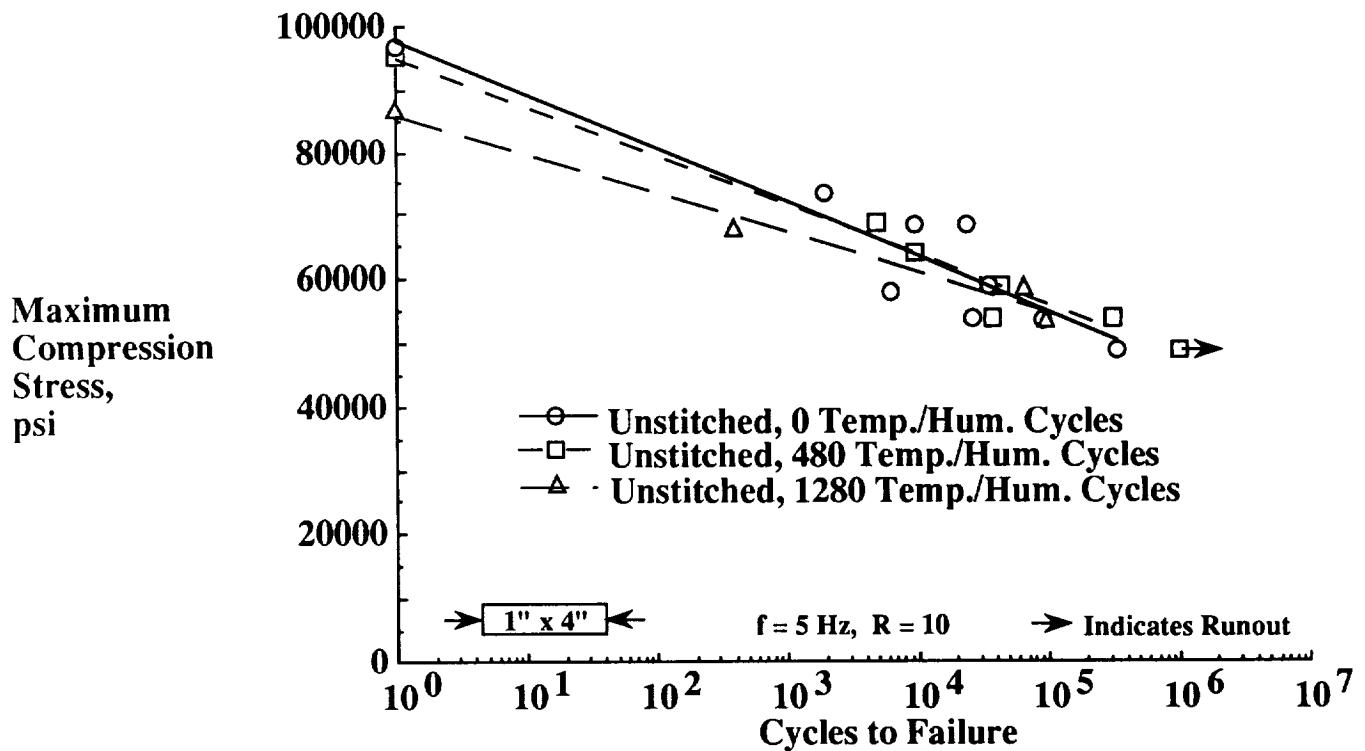


Figure 9. Fatigue data for unstitched AS4/3501-6 uniweave laminates with and without temperature/humidity cycles.

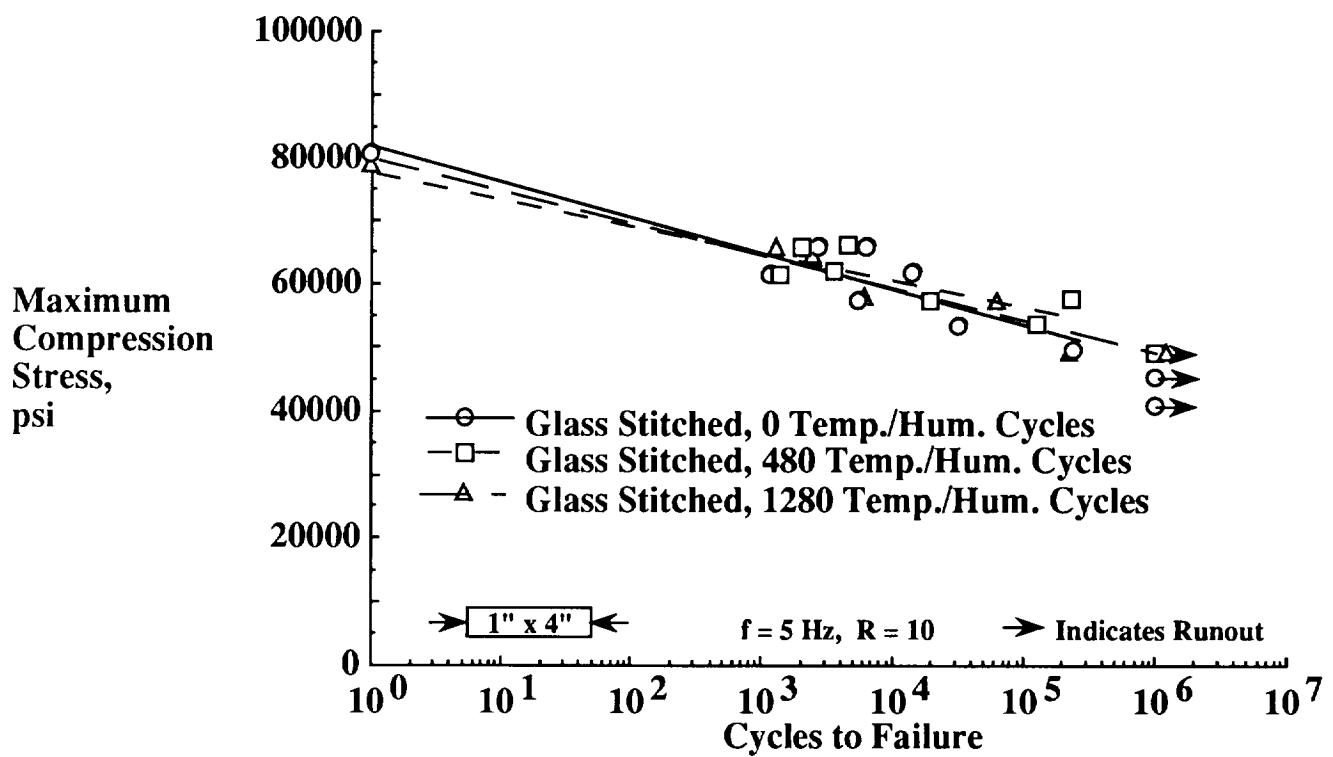


Figure 10. Fatigue data for glass stitched AS4/3501-6 uniweave laminates with and without temperature/humidity cycles.

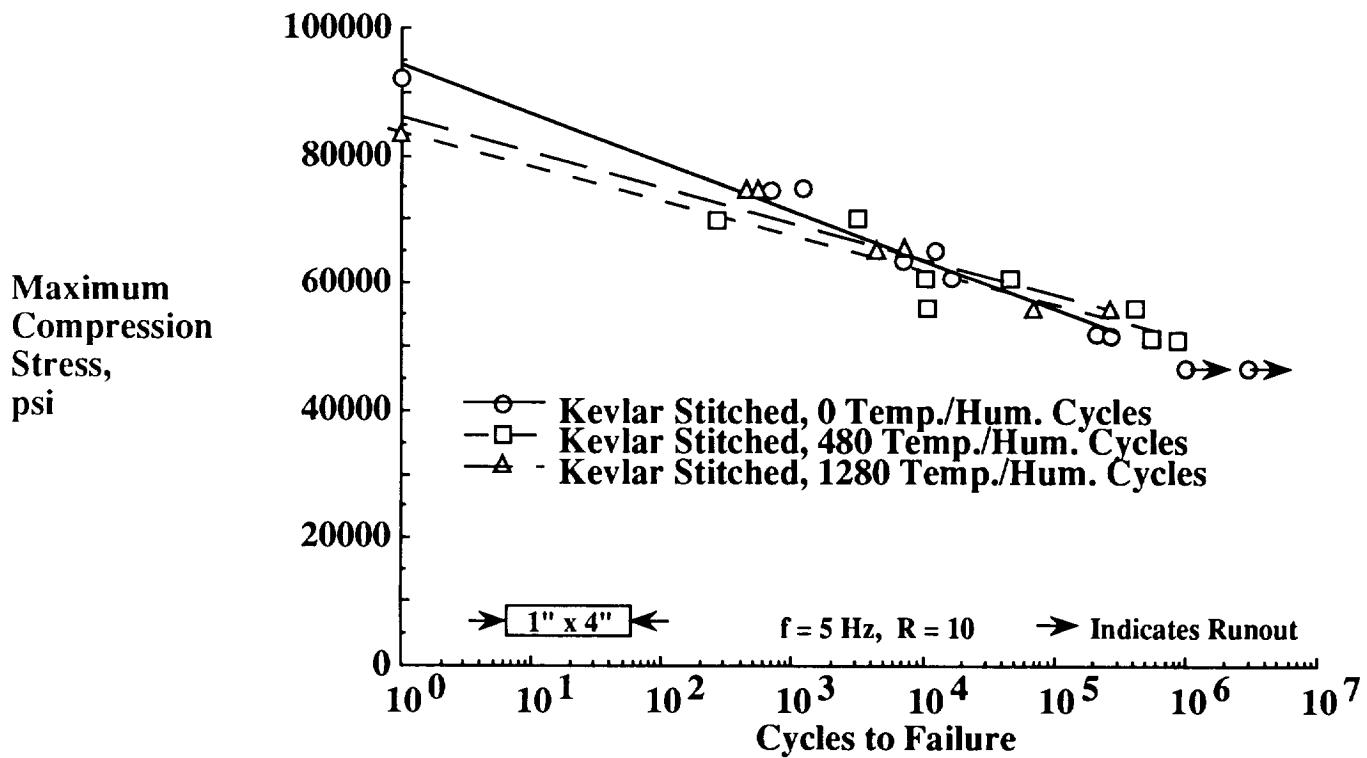


Figure 11. Fatigue data for Kevlar stitched AS4/3501-6 uniweave laminates with and without temperature/humidity cycles.

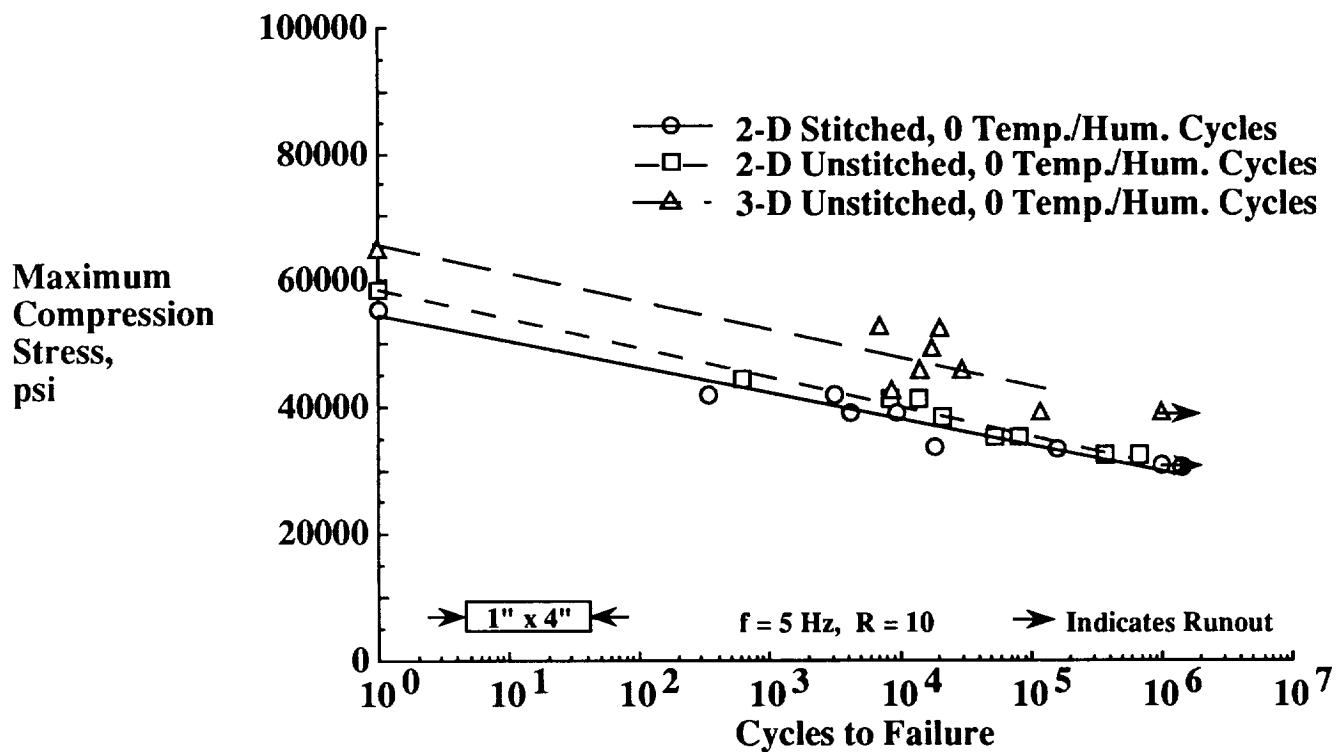


Figure 12. Fatigue data for AS4/E905L braided laminates without temperature/humidity cycles.

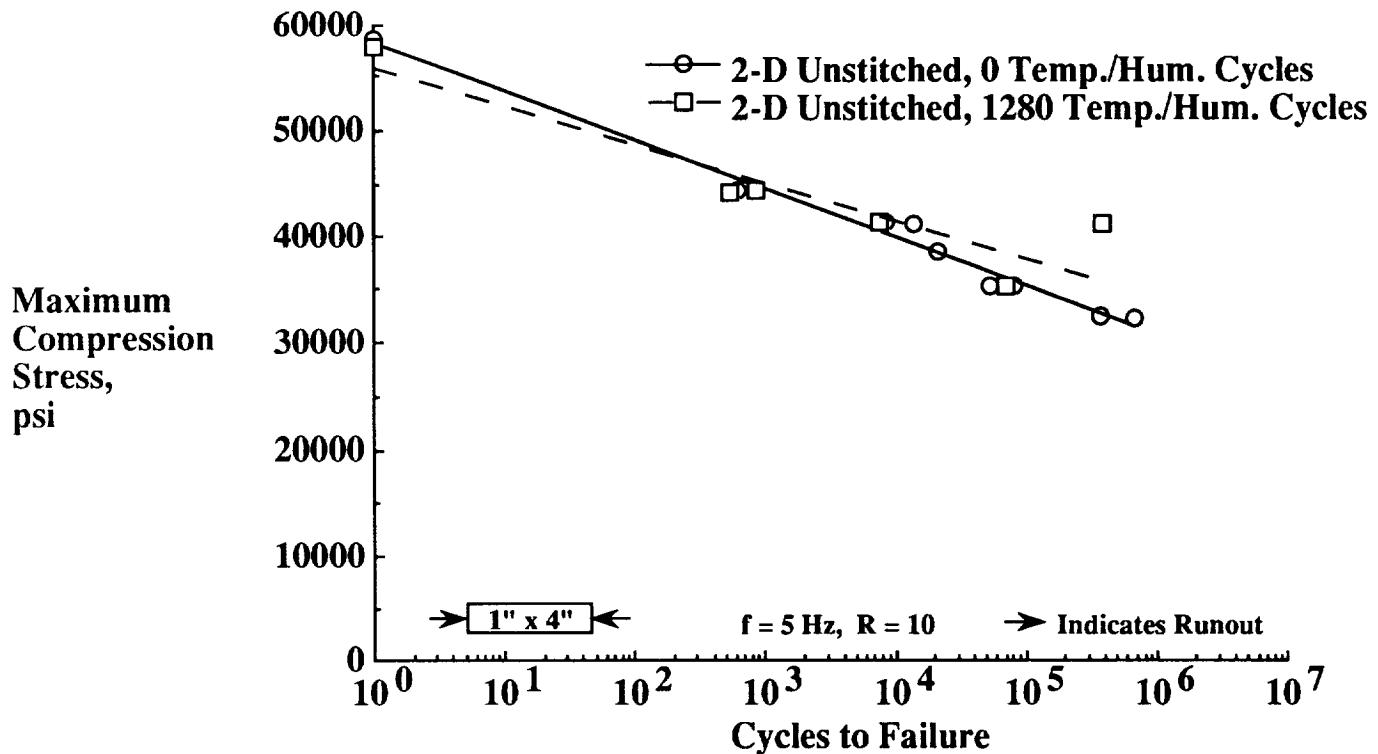


Figure 13. Fatigue data for 2-D unstitched AS4/E905L braided laminates with and without temperature/humidity cycles.

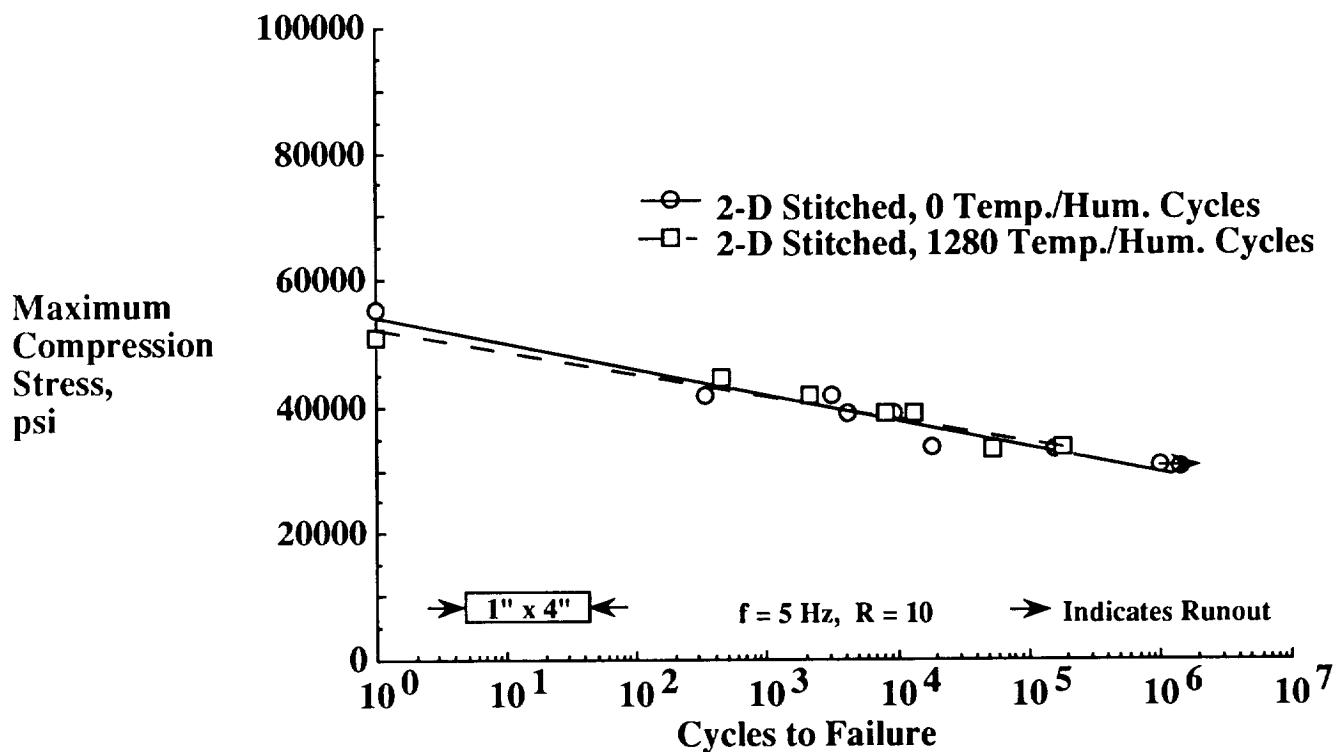


Figure 14. Fatigue data for 2-D stitched AS4/E905L braided laminates with and without temperature/humidity cycles.

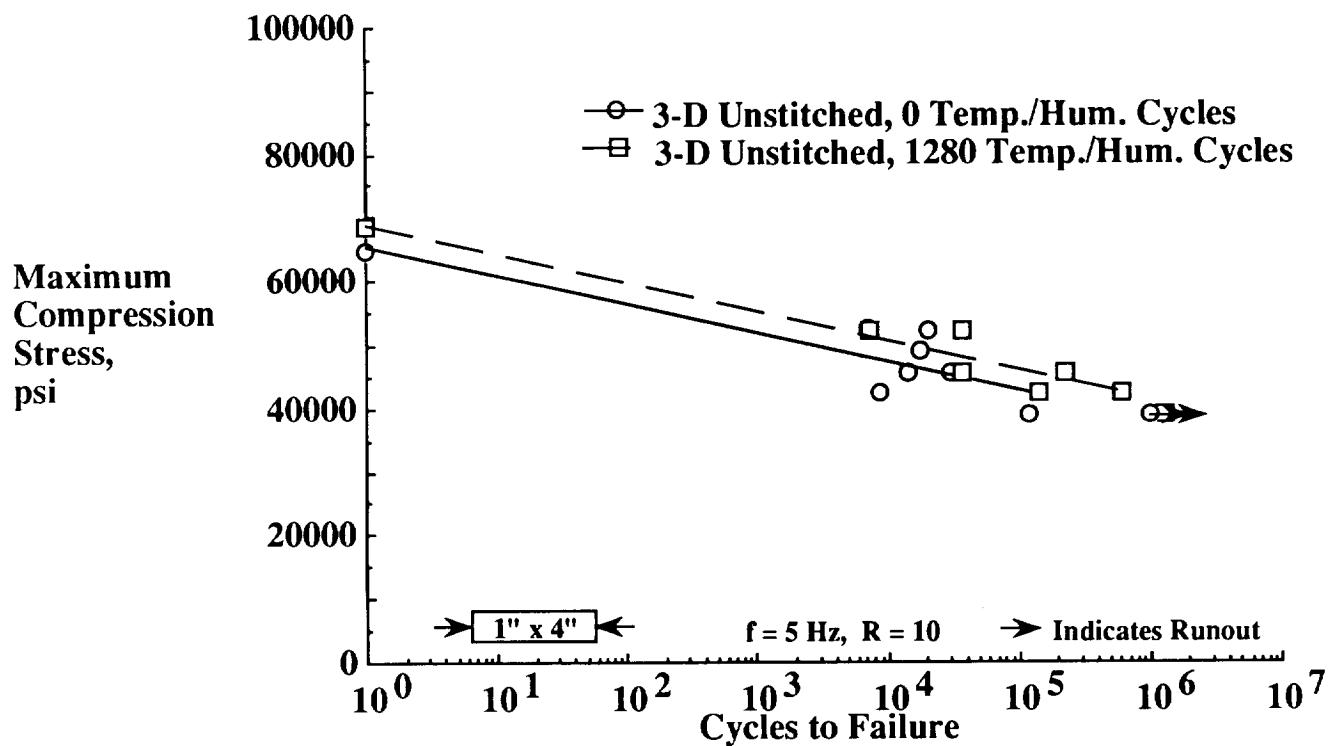


Figure 15. Fatigue data for 3-D unstitched AS4/E905L braided laminates with and without temperature/humidity cycles.

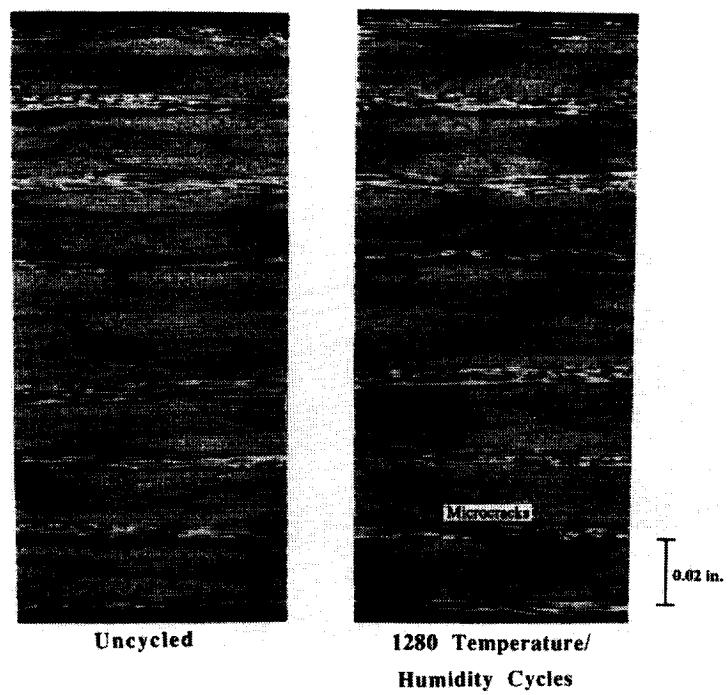


Figure 16. Photomicrographs of unstitched AS4/3501-6 uniweave.

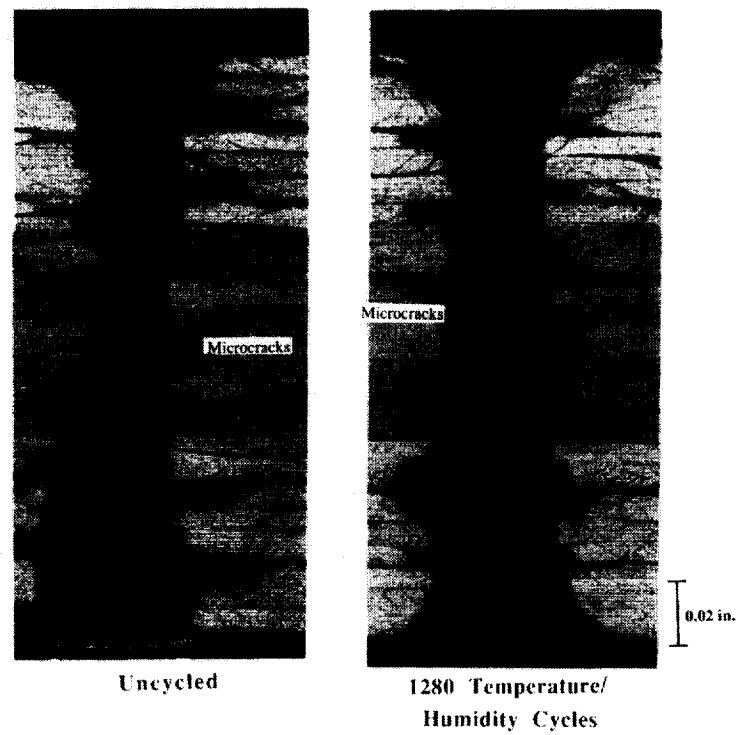


Figure 17. Photomicrographs of S-2 glass stitched AS4/3501-6 uniweave.

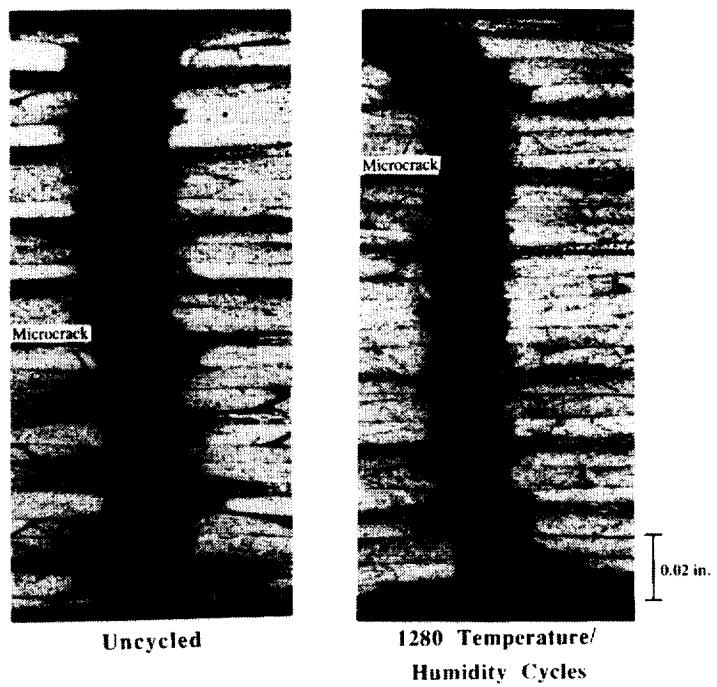


Figure 18. Photomicrographs of Kevlar 29 stitched AS4/3501-6 uniweave.

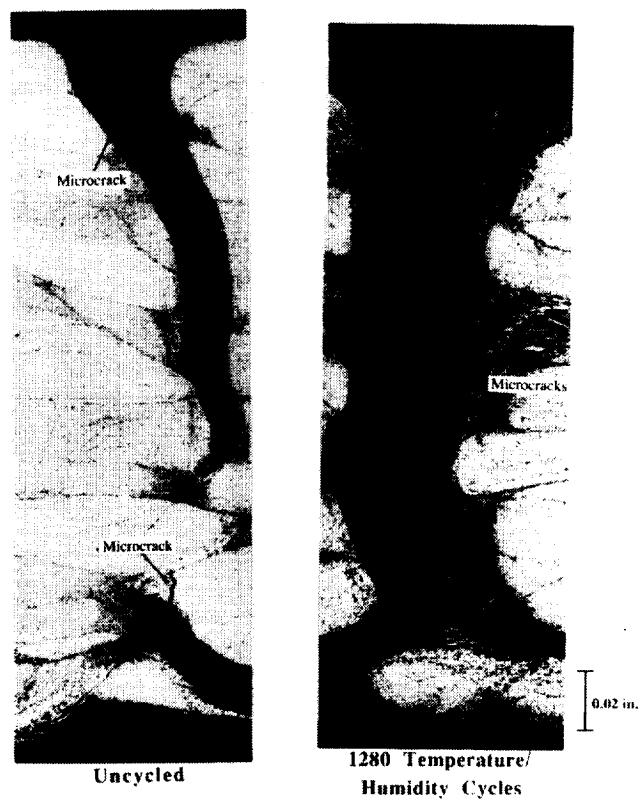


Figure 19. Photomicrographs of S-2 glass stitched AS4/E905L 2-D braid.

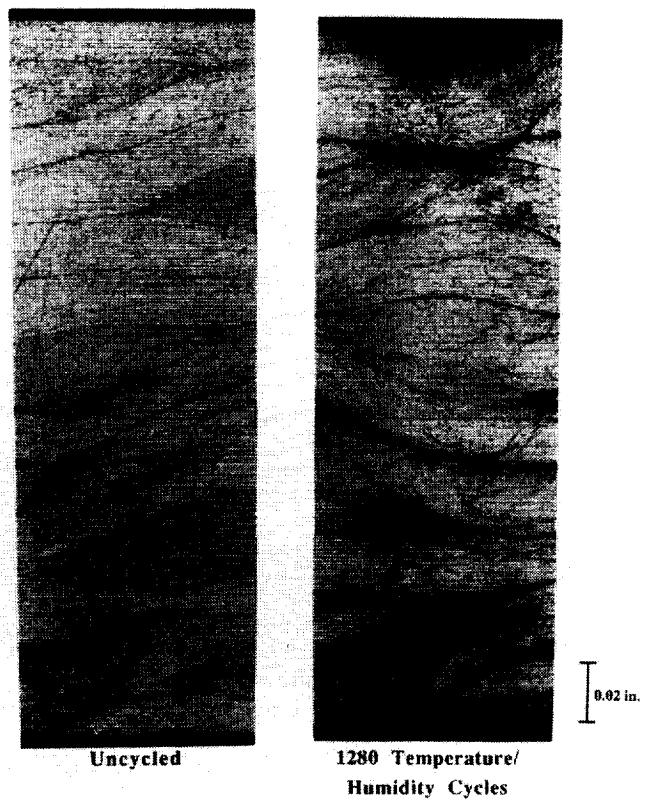


Figure 20. Photomicrographs of unstitched AS4/E905L 2-D braid.

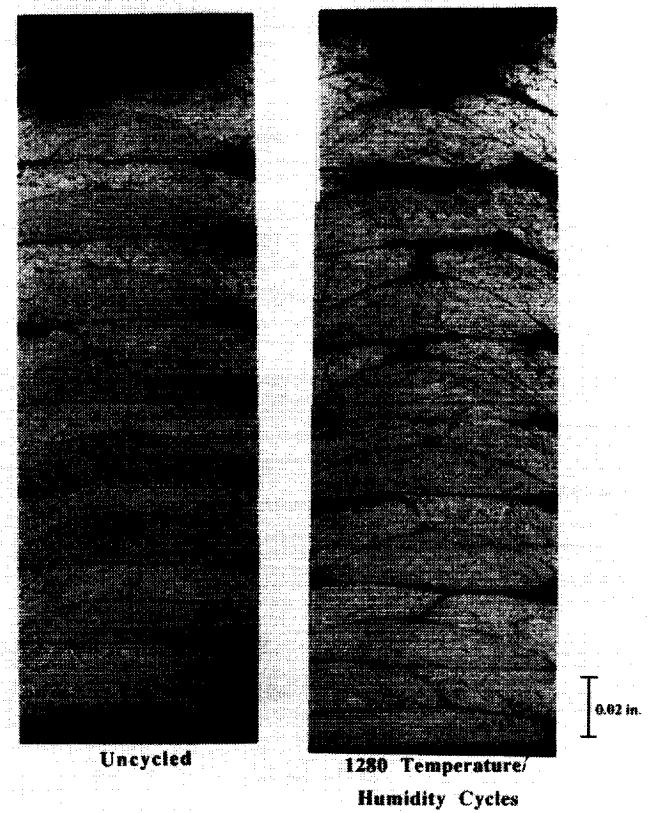


Figure 21. Photomicrographs of Kevlar 29 stitched AS4/E905L 3-D braid.