

AD-A251 131



WL-TR-91-4125

**Thermal Exposure and Thermal Protection of a
Ceramic Fiber / Glass Matrix Composite**

R. C. Wetherhold

Department of Mechanical and Aerospace Engineering
State University of New York
Buffalo, NY 14260

L. P. Zawada

Materials Behavior Branch (MLLN)
Metals and Ceramics Division
Materials Directorate
Wright Laboratory
WPAFB, OH 45433-6533



March 1991

Interim Report for Period May 1988 - January 1990

Approved for Public Release; Distribution is Unlimited.

**MATERIALS DIRECTORATE
WRIGHT LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AFB, OHIO 45433-6533**

92-14271



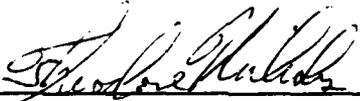
92 5 29 032

NOTICE

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely Government-related procurement, the United States Government incurs no responsibility nor any obligations whatsoever. The fact that the government may have formulated, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise in any manner construed, as licensing the holder of any other person or corporation, or as conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

This report is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.



Dr Theodore Nicholas
Project Engineer
Materials Behavior Branch



Katherine Williams
Technical Area Manager
Materials Behavior Branch



Allan Gunderson, Chief
Materials Behavior Branch

If your address has changed, if you wish to be removed from our mailing list, or if the addressee is no longer employed by your organization, please notify WL/MLLN, Wright-Patterson AFB, OH 45433-6533 to help us maintain a current mailing list.

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

| REPORT DOCUMENTATION PAGE | | | Form Approved OMB No. 0704-0188 | |
|---|--------------------------|--|------------------------------------|---------------------------|
| Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503. | | | | |
| 1. AGENCY USE ONLY (Leave blank) | 2. REPORT DATE Mar 91 | 3. REPORT TYPE AND DATES COVERED Interim Report May 88 to Jan 90 | | |
| 4. TITLE AND SUBTITLE Thermal Exposure and Thermal Protection of a Ceramic Fiber/Glass Matrix Composite | | 5. FUNDING NUMBERS PE 61102F PR 2302 TA P1 WU 01 Contract #F33615-88-C-5402 | | |
| 6. AUTHOR(S) R.C. Wetherhold and L.P. Zawada | | 8. PERFORMING ORGANIZATION REPORT NUMBER | | |
| 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Materials Behavior Branch (WL/MLLN) Wright-Patterson AFB OH 45433-6533 Dept. of Mech. & Aero. Engineering Sunny, Buffalo NY 14260 | | 10. SPONSORING / MONITORING AGENCY REPORT NUMBER WL-TR-91-4125 | | |
| 9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) Larry P. Zawada (513/255-1352) Materials Directorate (WL/MLLN) Wright Laboratory Wright-Patterson AFB OH 45433-6533 | | 11. SUPPLEMENTARY NOTES | | |
| 12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution is unlimited. | | 12b. DISTRIBUTION CODE | | |
| 13. ABSTRACT (Maximum 200 words) Thermal exposure in air has been shown to embrittle a ceramic fiber/glass matrix composite by the infiltration of oxygen to the fiber-matrix interface. Samples exposed at sufficiently high temperature demonstrate less embrittlement than those exposed at intermediate temperatures, due to a material flow which seals the surface. It is shown that the surface protection against embrittlement provided by a short, high-temperature thermal treatment remains in force for materials subsequently exposed under both loaded and unloaded conditions. In comparing results from plates processed under different conditions, it was found that the composites embrittled at two different rates - either quickly or slowly. The rate of embrittlement was not predictable from testing as-fabricated materials. A series of processing tests were carried out to determine the reason for the difference in the rapidity of embrittlement. The rapid embrittlement effects is believed to be attributable to an excess of carbon in the as-fabricated matrix. The thermal exposure treatment is effective with both types of composites (those which embrittle quickly and those which embrittle slowly). | | | | |
| 14. SUBJECT TERMS Ceramic Matrix Composites Fibers Interfaces | | Processing Thermal Protection Mechanical Behavior | | 15. NUMBER OF PAGES 28 |
| 17. SECURITY CLASSIFICATION OF REPORT Unclassified | | 18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified | | 16. PRICE CODE |
| 19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified | | 20. LIMITATION OF ABSTRACT UL | | |

FOREWORD

This Technical Report was prepared by the Department of Mechanical and Aerospace Engineering of the State University of New York for the Metals and Ceramics Division, Materials Directorate. Dr Theodore Nicholas, WL/MLLN, was the project engineer. The research was conducted under Task 30, "Thermal Fatigue in Ceramic Matrix Composites," and Task 125, "Thermal / Mechanical Fatigue of High Temperature Composites," Contract No: F33615-88-C-5402. In addition, research was also conducted under Task 2302P101, "Failure Prediction in Materials," and Air Force Materials Directorate contract No: F33615-87-C-5606. This report includes work performed during the period May 1988 to January 1991. The work was performed by Larry P. Zawada of the Air Force Materials Directorate, and Dr Robert C. Wetherhold of the State University of New York.

The authors gratefully acknowledge the valuable discussions with Dr T. Nicholas and Dr R. Kerans. Their comments during the project were very helpful. A sincere thanks is expressed to George Hartman of the University of Dayton Research Institute for his cooperation in setting up the experimental testing equipment. In addition, the authors appreciate the efforts of David Cartmel and Andrew Lackey in producing the CMC tiles.

| | |
|--|-----------------------------|
| Accession For | |
| <input checked="" type="checkbox"/> NTIS CRA&I | |
| <input type="checkbox"/> DTIC TAB | |
| <input type="checkbox"/> Unannounced | |
| Justification | |
| By _____ | |
| Distribution/ | |
| Availability Codes | |
| Dist | Avail and/or Special |
| A-1 | |

TABLE OF CONTENTS

| Section | | Page |
|---------|-------------------------------------|------|
| I | INTRODUCTION..... | 1 |
| II | PROCESSING AND INITIAL RESULTS..... | 3 |
| III | HEAT TREATMENT..... | 13 |
| IV | DISCUSSION AND CONCLUSIONS..... | 17 |
| | REFERENCES..... | 22 |

LIST OF ILLUSTRATIONS

| Figure | | Page |
|--------|---|------|
| 1 | HIGH STRENGTH FLEXURE FAILURE | |
| | a) TENSILE FACE VIEW..... | 6 |
| | b) EDGE VIEW..... | 7 |
| 2 | HIGH STRENGTH FAILURE LOAD TRACE..... | 8 |
| 3 | LOW STRENGTH FLEXURE FAILURE | |
| | a) TENSILE FACE VIEW..... | 9 |
| | b) EDGE VIEW..... | 9 |
| 4 | LOW STRENGTH FAILURE LOAD TRACE..... | 11 |
| 5 | AS-FABRICATED SURFACE..... | 12 |
| 6 | 800°C HEAT-TREATED, "SMOOTHED" SURFACE..... | 12 |
| 7 | TENSILE STRESS-STRAIN CURVE..... | 20 |

LIST OF TABLES

| Table | | Page |
|-------|-----------------------------------|------|
| 1 | PROCESSING SCREENING STUDIES..... | 5 |
| 2 | PLATE 88C14 STRENGTH RESULTS..... | 14 |
| 3 | PLATE 89C18 STRENGTH RESULTS..... | 15 |
| 4 | PLATE 89C45 STRENGTH RESULTS..... | 15 |
| 5 | PLATE 89C46 STRENGTH RESULTS..... | 16 |
| 6 | PLATE 89C47 STRENGTH RESULTS..... | 16 |
| 7 | PLATE 90C50 STRENGTH RESULTS..... | 16 |

SECTION I

INTRODUCTION

Ceramic Matrix Composites (CMC) composed of brittle fibers in a brittle matrix offer improvement in fracture toughness over monolithic ceramics. This is only true, however, if the fibers remain loosely bonded to the matrix, allowing crack bridging by unbroken fibers as well as crack blunting and branching. This is normally accomplished by the action of an easily debonded interface layer such as one rich in carbon, which either arises chemically during fabrication [1-4] or is directly applied [5,6]. The loss of this interface can result in a strong bonding between fiber and matrix, which returns the composite behavior to that of the monolith: straight crack paths with little energy absorption.

As seen in previous work [1,3,7-9], the infiltration of oxygen into a CMC may cause loss of the carbon-rich interface with subsequent embrittlement and loss of strength. Work with a Nicalon* fiber/alkaline earth alumino-silicate glass (Corning code 1723) composite has indicated that exposure to intermediate temperatures (650°C) can be more damaging for embrittlement than exposure to high temperatures (800°C) [8]. These effects were observed under both simple thermal exposure as well as under thermal fatigue, and have importance particularly for high temperature structural parts which will have intermediate temperature attachment points to the rest of the structure. The presumed mechanism for greater retained strength after 800°C exposure is that flow of matrix material smooths the surface and inhibits oxygen infiltration.

In this report, we focus on whether the high temperature smoothing may be used as a treatment to slow subsequent embrittlement under a variety of load and exposure conditions. In addition, some earlier production plates have

*TM Nippon Carbon Co. Ltd. Japan

shown a quick embrittlement, while in other later production plates the embrittlement is more gradual. Screening processing studies are performed to attempt to determine how the embrittlement differences arise. This difference is subtle, since no distinguishable differences appear in the strength or in the polished cross sections of the as-fabricated plates. The processing studies will also determine the values of leading process variables which will provide an improved composite strength.

SECTION II

PROCESSING AND INITIAL RESULTS

The fabrication process may be briefly described as follows. Fibers are drawn from spools through a sizing burn-off furnace, proceed through a bath of glass frit, polypropylene binder, and distilled water, and are wound onto an octagonal drum. After lamp drying, the layers are cut from the drum, stacked into a unidirectional laminate and loaded into a graphite die. Multiple plates in the same hot press run are separated by additional layers of graphite. Once loaded, the assembly is placed in a 375°C oven to burn off the binder and remove any remaining water. The graphite die is then placed in a vacuum hot press. The temperature is ramped to the maximum temperature, held for 10 minutes, and ramped down. Consolidation pressure of 1500 psi is applied at 700°C on upramp, and released at 400°C on downramp. Typical fiber volume fractions are 40-45 vol%, with well dispersed fibers.

The finished plates are cut into flexure specimens using an oil cooled diamond saw, and the cut edges are polished on a 45 μ m diamond wheel. Specimens are typically 5 mm wide by 2.5 mm thick, by 100 mm long. The 4-point flexure fixture has an 80mm outer span with 40mm inner span, and specimens were loaded in an Instron test machine at 5 mm/min. Sample failure was defined based on the maximum load consistent with tensile failure; the load-deflection curves of embrittled specimens were basically linear-to-failure, while non-embrittled specimens demonstrated an initial linear response followed by non-linearity.

The most critical processing variables were expected to be the maximum hot press processing temperature, the sizing on the fibers, and the sizing burn-off temperature. Nicalon* fibers have either "P" (modified epoxy) or "M" (polyvinylacetate) proprietary sizing intended for use in polymeric or ceramic matrices, respectively. The critical variables were varied systematically, and

the results appear in Table 1. The quantities of interest are the flexural strength of as-fabricated plates, and the percentage of strength retained after exposure to 650°C for 16 hours in air.

The use of P vs. M sizing was initially felt to be significant based on 89C18 and 88C14 results after standard embrittlement exposure. However, subsequent comparisons of 88C14 to 89C47 and 89C45 to 89C46 indicate no preference between the sizings. A further discussion of plate 88C18 is given in Section IV. The results of tests using various maximum pressing temperatures (88C18 to 89C46 to 89C47) give a good indication that the preferred temperature should be 1080-1100°C. No conclusions could be drawn for the importance of the burn-off temperature. All conclusions must be considered as preliminary, since the fiber strength may itself vary considerably.

The use of a flexural test to evaluate strength has been carefully considered. The fracture mode of interest is tensile, and flexure tests are only of use if they highlight this behavior. In the test results given in Table 1 and the following tables, the fracture initiates on the tensile side, within the central gauge section, with no compression failure features such as crushing or delamination under the loading pins or buckling of the compressive face. Cases of doubtful tensile failure (primarily failure outside the gauge section) comprised less than 15% of the total, and are not given in the tables. All tests were carried out at room temperature.

TABLE 1.
PROCESSING SCREENING STUDIES

| Plate | Sizing | Max Processing Temp (°C) | Sizing Burn-off Temperature (°C) | As-fabricated flex strength (MPa) | % Retained strength |
|---------|--------|--------------------------|----------------------------------|-----------------------------------|---------------------|
| 89C18 | M | 1100 | est. 725 | 1050 | 75% |
| 88C14 | P | 1080 | est. 700 | 1120 | 14% |
| { 89C45 | P | 1125 | 725 | 830 | 73% |
| { 89C46 | M | 1125 | 725 | 835 | 77% |
| 89C47 | M | 1081 | 700 | 1220 | 58% |
| 89C50 | M | 1086 | 660 | 1050 | 72% |

{ denotes common hot-pressing run

Typical failure modes are described as follows. High strength failures involve multiple cracks on both the tensile face and edges of the flex specimen. No cracks were observed on the compression face, even after failure. It is interesting to note that the cracks on the tensile face appear to first initiate near an edge and then propagate longitudinally along the gauge length, reversing direction several times in a tortuous, zigzag path. Several long interlaminar cracks also develop on the edges of the specimen. All of these interlaminar cracks on the edges propagate longitudinally along the gage length, with little through-the-thickness link-up. The overall fracture mode can be described as very fibrous, indicating a great deal of matrix damage and fiber debonding and pull-out; see Figures 1a and 1b. The strength values are based on the load at point "L" of the load-displacement curve typified by Figure 2, with visual inspection so that we are assured of obtaining a true tensile strength. Any subsequent interlaminar propagation is ruled out as of no interest. Specimens with lower strength generally had fewer and much straighter cracks.

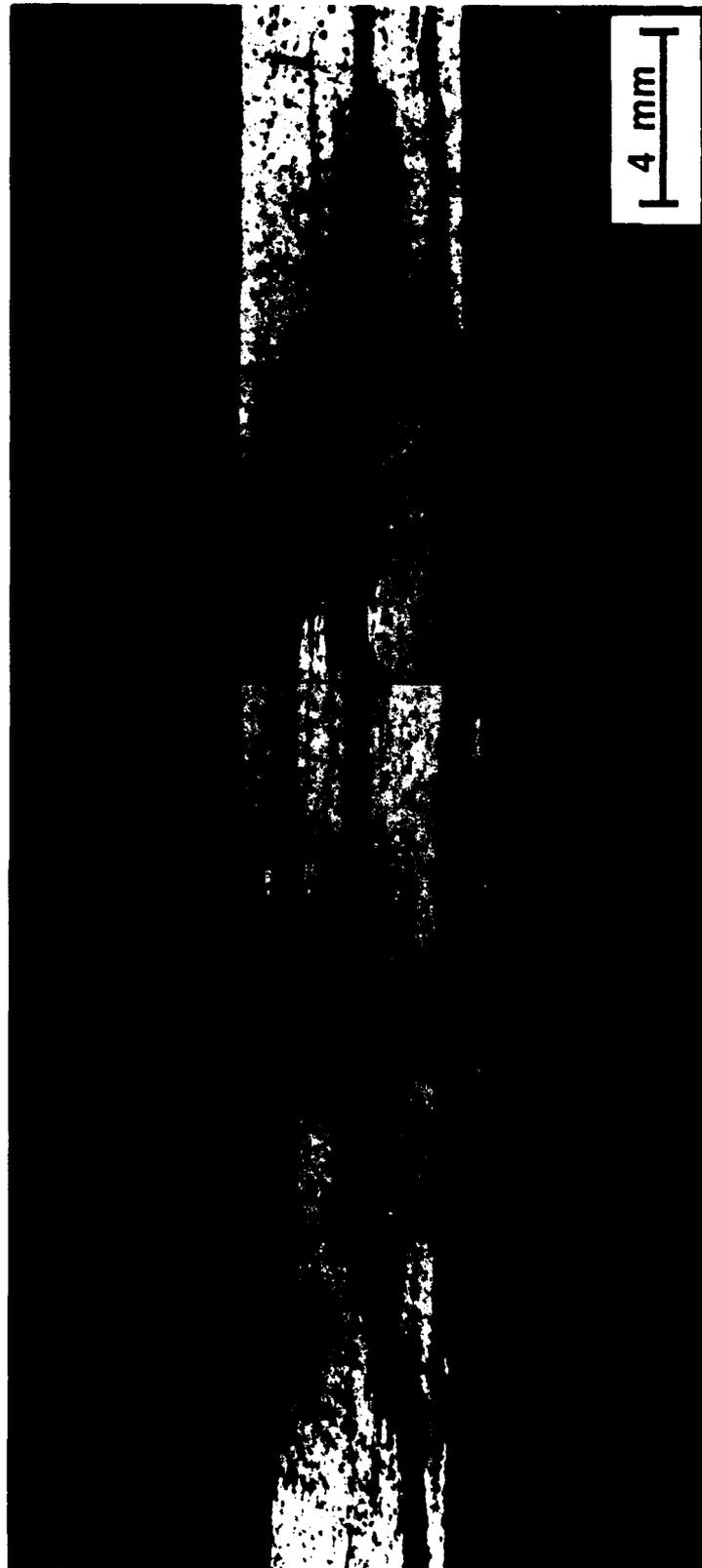


Figure 1a High Strength Flexure Failure - Tensile Face View

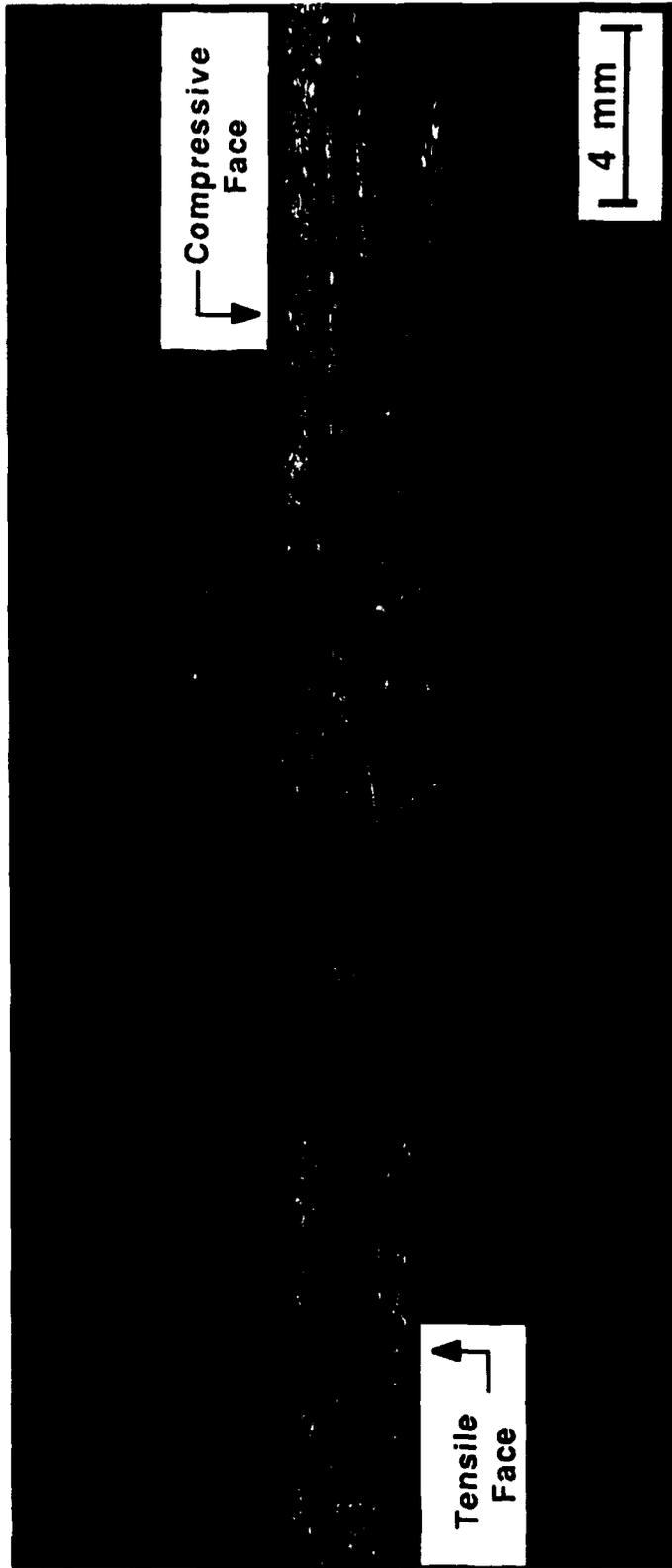


Figure 1b High Strength Flexure Failure - Edge View

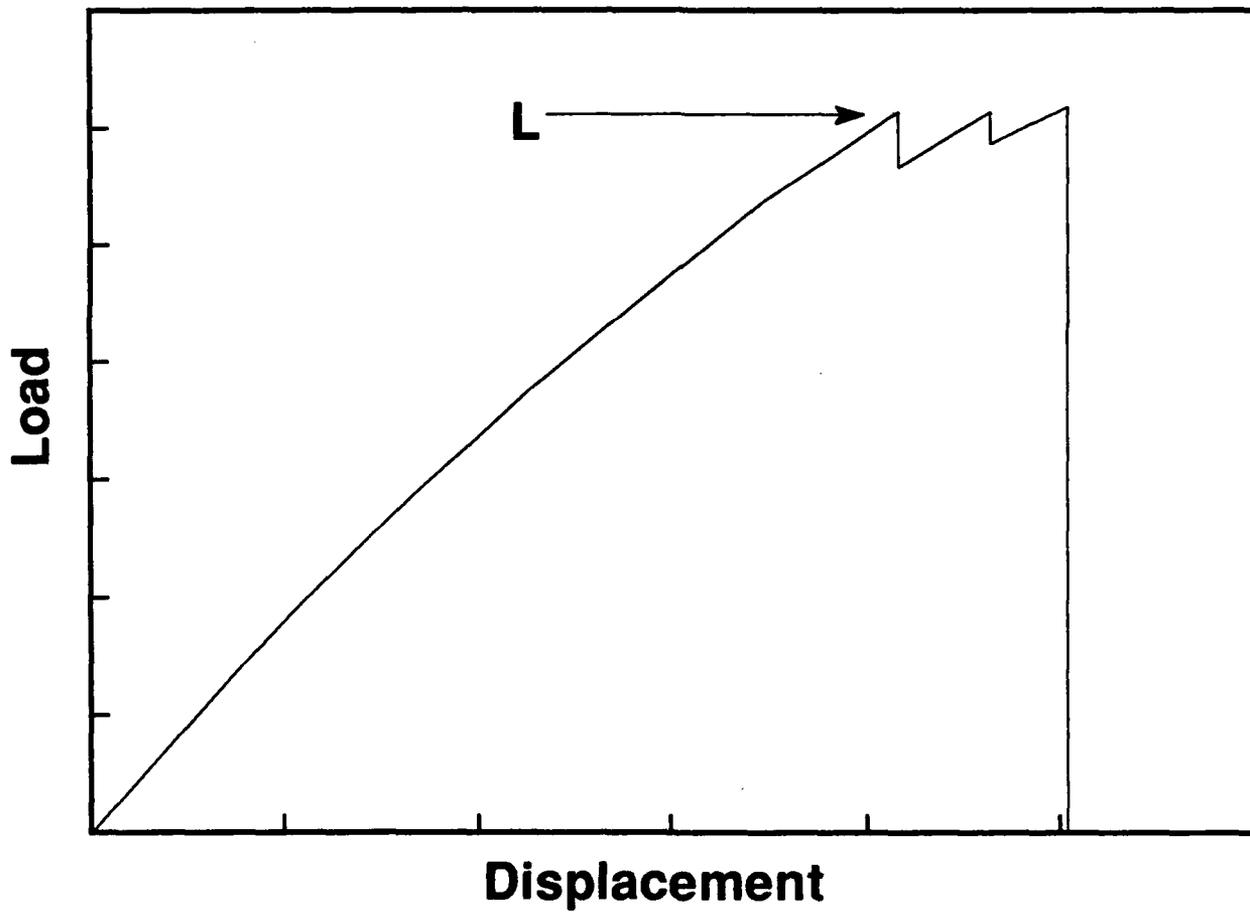


Figure 2 High Strength Failure Load Trace

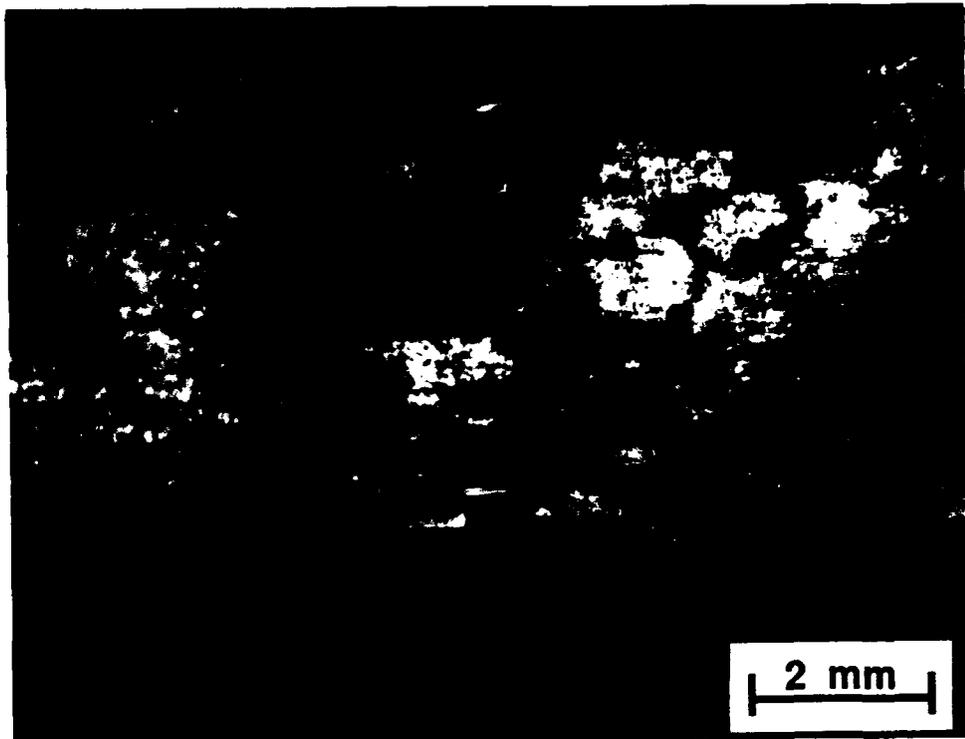


Figure 3a Low Strength Flexure Failure - Tensile Face View

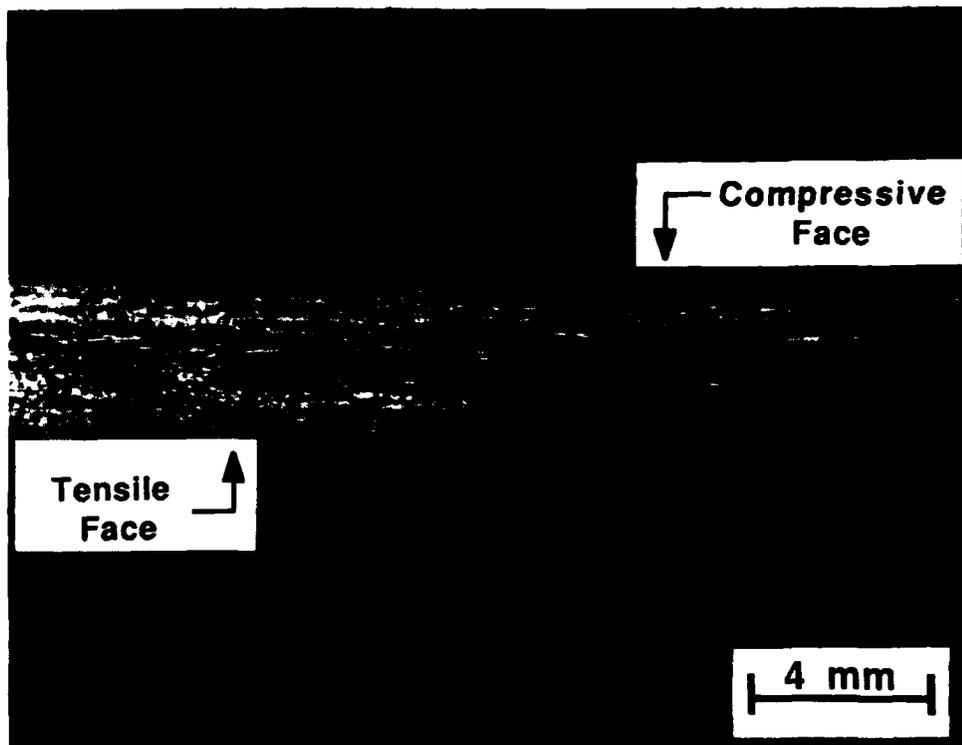


Figure 3b Low Strength Flexure Failure - Edge View

All of the lowest strength failures typically developed only one dominant crack. In these cases, the crack would develop essentially straight across on the tensile face and then propagate diagonally through the thickness of the flex specimen (a "toothpick" or wood-like failure); see Figures 3a and 3b. The strength values are based on the point "L" of Figure 4; there is little doubt that the value captured is the tensile strength before interlaminar propagation. Such failures resulted in flat fracture surfaces with greatly decreased fiber pull-out.

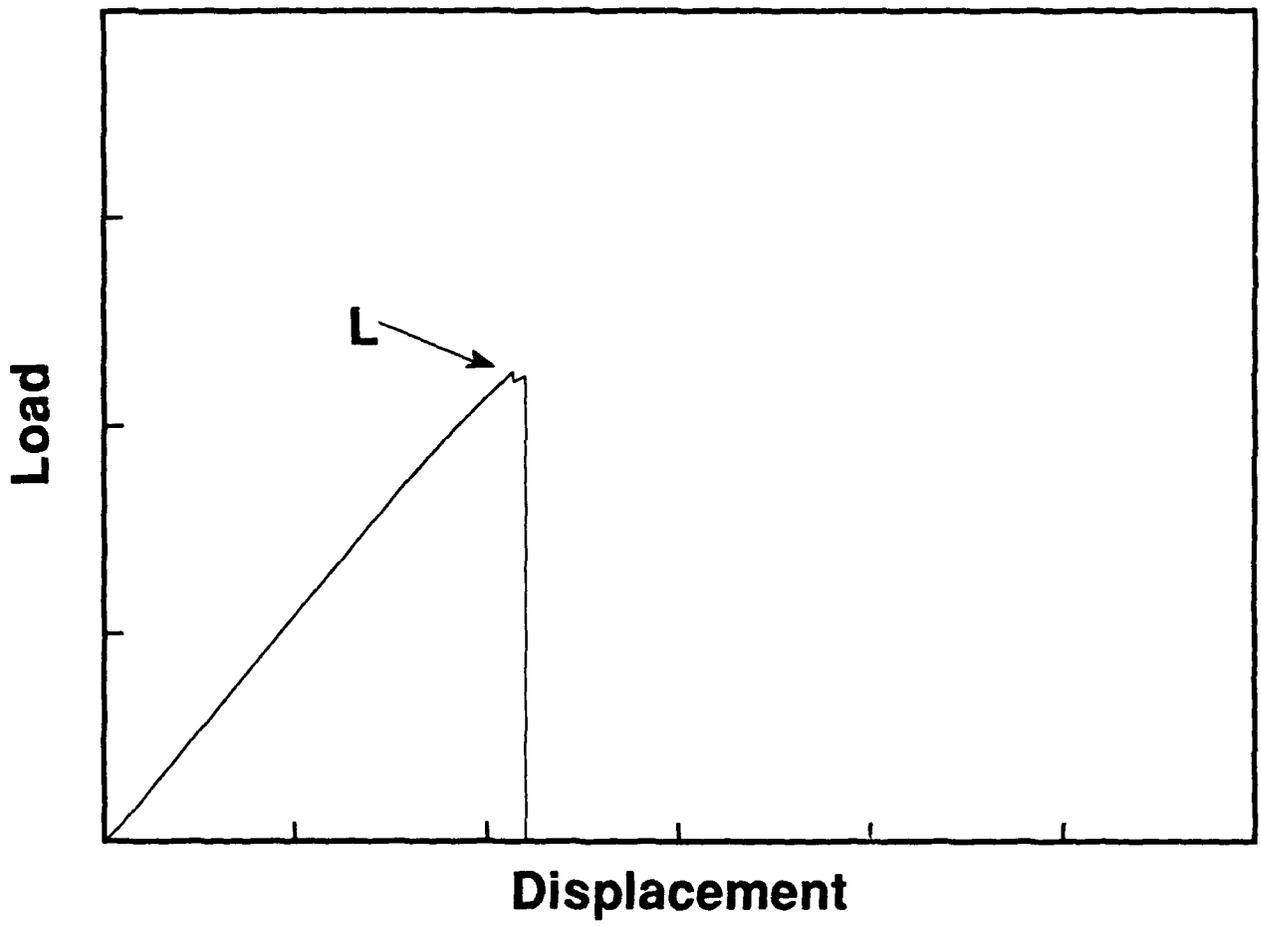


Figure 4 Low Strength Failure Load Trace

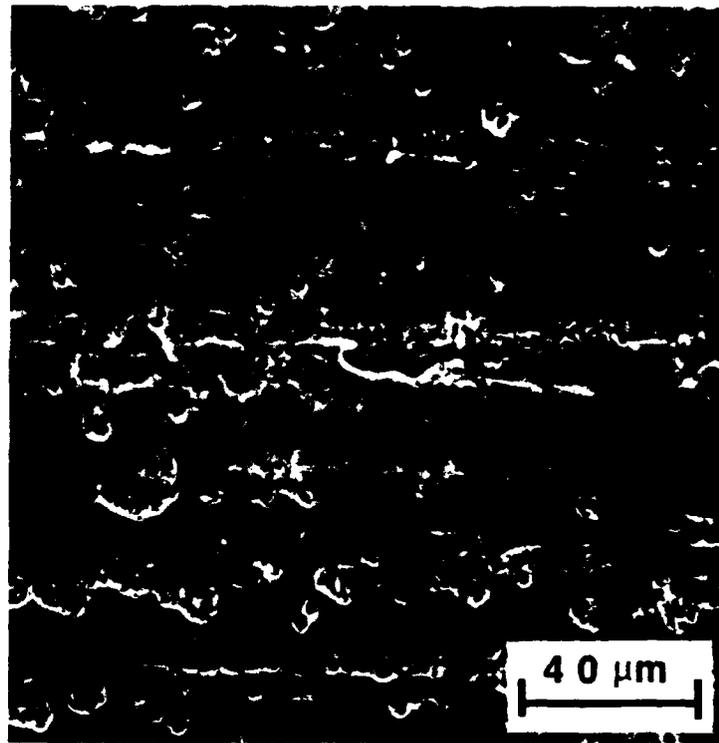


Figure 5 As-Fabricated Surface

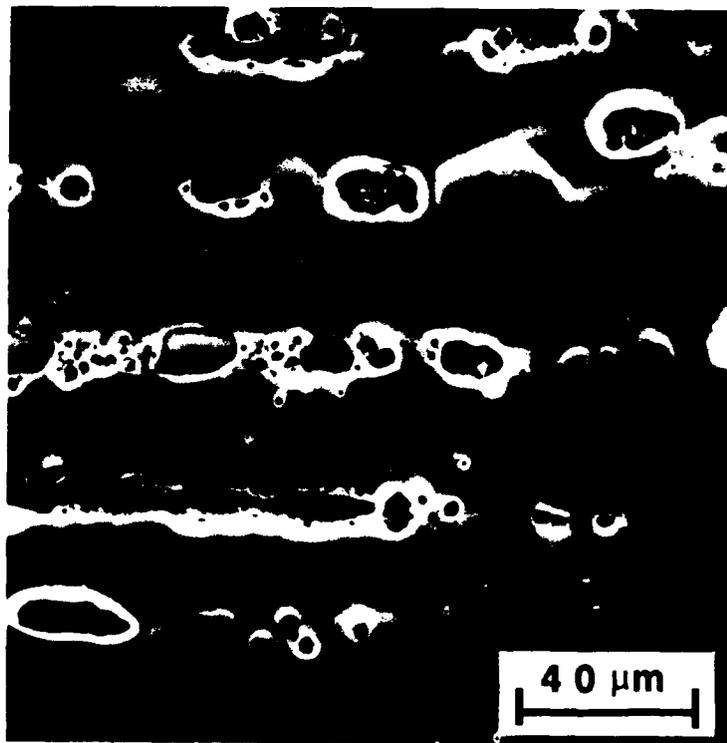


Figure 6 800°C Heat-Treated, "Smoothed" Surface

SECTION III

HEAT TREATMENT

Earlier tests had revealed that high temperature (800°C) exposure was less damaging (produced less embrittlement) than intermediate temperature exposure (650°C) in static air, since the higher temperatures produced a smoothing or healing of cracks in the surface by material flow [8]. See Figures 5,6. This smoothing causes a decrease in the oxygen infiltration from the surface and lessens the embrittlement. This first appeared as an "ordering effect," where an initial exposure to higher temperature lessened the embrittlement on subsequent exposure at intermediate temperature; see Table 2. This led to the proposal for a "heat treatment" at 800°C to improve properties during subsequent exposure at 650°C.

If the heat treatment is to be useful, we must understand its action under a variety of load and exposure conditions. In the following tables, the decision was made to explore the maximum number of conditions so as to screen for significant effects, with replicates only performed as needed to clarify the results. Every plate was tested in the as-fabricated and standard embrittlement exposure (650°C, 16 hr) conditions, since a baseline must be established for each plate. The baseline results differ sufficiently that the overall results from all plates cannot be combined (see Table 1). The trends, however, are consistent and clear; the number of replicates for each condition and the spread of data are given to aid the understanding of any overlap of results. All thermal exposures and heat treatments are done in air, with no forced ventilation. All plates are unidirectional 10 ply, 2.5 mm thick except plate 88C14, which was 6 ply, 1.5 mm thick.

The speed of embrittlement was investigated considering the standard

embrittlement exposure and a 9-times longer exposure; see Table 3. Since the material may be preloaded in a structural application, a tension sufficient to cause microcracking was applied. The embrittlement exposure was then carried out (see Table 3). Since it was uncertain how long a heat treatment must be applied to be effective, the results of a 16-hour 800°C treatment and a 15-minute 850°C treatment are compared (see Tables 4,5). The manner in which the treatment is applied is also considered. Normally, this is done in air in a box oven. Considering the positive effects of tempering in "sealing" a surface through compressive stress, samples were tested where the heat treatment was followed by quenching. See Table 5. Since air quench velocities for the proper Biot number would have been too high to achieve using standard cooling nozzles [11], an oil quench was used. The application of these materials will involve combinations of thermal exposure and load conditions. The combination of exposure with tensile load is given in Table 6, and exposure with compressive load in Table 7. The effect of the heat treatment before exposure under load is also found in Table 6.

TABLE 2.
PLATE 88C14 STRENGTH RESULTS

| Conditions (Number of Replicates) | σ_{ULT} , MPa | [% of MAX] |
|--|----------------------|------------|
| (3) Control | 1120 ± 35 | [100] |
| (2) 650°C, 16 hr | 160 ± 35 | [14] |
| (4) 800°C, 16 hr | 960 + 190 - 260 | [86] |
| (2) 800°C, 16 hr, then 650°C, 16 hr | 630 ± 40 | [56] |

TABLE 3.
PLATE 89C18 STRENGTH RESULTS

| Conditions (Number of Replicates) | σ_{ULT} , MPa | [% of MAX] |
|---|----------------------|------------|
| (2) Control | 1050 \pm 130 | [100] |
| (2) 650°C, 16 hr | 790 \pm 10 | [75] |
| (4) 650°C, 144 hr | 620 \pm 110 | [59] |
| | - 80 | |
| (2) Preload to 350 MPa | 950 \pm 20 | [90] |
| (2) Preload to 350 MPa; then 650°C for 16 hr, (no load) | 670 \pm 10 | [64] |

TABLE 4.
PLATE 89C45 STRENGTH RESULTS

| | | |
|---|--------------|-------|
| (2) Control | 830 \pm 30 | [100] |
| (2) 650°C, 16 hr | 610 \pm 20 | [73] |
| (2) 800°C, 16 hr; then 650°C, 16 hr | 720 \pm 20 | [87] |
| (2) 850°C, 15 min; then 650°C, 16 hr | 825 \pm 15 | [99] |

TABLE 5.
PLATE 89C46 STRENGTH RESULTS

| | | | |
|-----|--|------------------|-------|
| (3) | Control | 835 + 90 - 40 | [100] |
| (2) | 650°C, 16 hr | 645 ± 15 | [77] |
| (2) | 800°C, 16 hr; then 650°C, 16 hr | 830 ± 30 | [99] |
| (1) | heat to 800°C, 15 min, quench; then 650°C, 16 hr | 780 | [93] |
| (1) | heat to 850°C, 15 min, quench; then 650°C, 16 hr | 770 | [92] |

TABLE 6.
PLATE 89C47 STRENGTH RESULTS

| Conditions (Number of Replicates) | σ_{ULT} , MPA | [% of MAX] |
|--|----------------------|------------|
| (2) Control | 1220 ± 50 | [100] |
| (2) 650°C, 16 hr | 775 ± 60 | [64] |
| (2) 150 MPa, 16 hr, 650°C | 550 ± 35 | [45] |
| (2) 850°C, 15 min; then 150 MPa, 16 hr, 650°C | 735 ± 60 | [60] |

TABLE 7.
PLATE 89C50 STRENGTH RESULTS

| | | | |
|-----|-----------------------|------------|-------|
| (2) | Control | 1050 ± 100 | [100] |
| (2) | 650°C, 16 hr | 760 ± 60 | [72] |
| (2) | -60 MPa, 650°C, 16 hr | 785 ± 50 | [77] |

SECTION IV

DISCUSSION AND CONCLUSIONS

As a prefatory comment, we note that there is generally no overlap (or very little overlap) between the strength values produced by different conditions for a given plate. Although the number of replicates per condition is too small to draw conclusions based on statistics, there is little confusion of results, and the trends are clear. The limited number of data presented here all appear consistent with the accepted understanding of how strength varies as a function of interface behavior. The only numerical results presented are the strength values, since no standard exists for quantifying the fiber pullout or failure mode of a broken specimen. It can be stated, however, that there is an excellent correlation between strength, the extent of fiber debonding and pullout, and the amount of nonlinearity in the stress-strain response amongst all of the results presented. This is consistent with the description of Section II: high strength occurred with much fiber pull-out, crack tortuousness, and nonlinearity of stress-strain response. As we proceed to the lower strengths typical of embrittled composites, the cracks become straighter with less pull-out, and the degree of stress-strain linearity to failure increases.

A test grid was run to determine improved processing conditions for as-fabricated strength, and to gain insight into how these conditions affect the rapidity of embrittlement. Table 1 demonstrates the importance of controlling the maximum temperature during pressing. It was already known that Code 1723 is a "short" glass, whose viscosity-temperature relationship is steeply curved (strain, anneal, softening, and working temperatures of 665^o, 710^o, 908^o, 1168^oC, respectively [12]). It is also known that a too-high processing temperature can result in both fiber degradation [13] and a nonoptimal (too

thick) carbon interface [6]. A proper temperature value for this system should be 1080-1100°C for our consolidation pressure; higher temperatures produced lower strengths. The sizing type seems to make no appreciable difference for the test conditions applied. Other candidate process variables were also considered for controlling properties. The plate thickness is not felt to be important, since no exothermic reaction occurs during processing. The particular fiber lot will be important, since it can determine chemistry-influenced properties such as thermal stability [13]. Similarly, the lot of glass frit or polypropylene may have an influence, although these are long-time commercial products.

The heat treatment to lessen embrittlement is seen to be effective for a variety of plates and test conditions; see Tables 2, 4, 5, 6. The treatment need not be long; 15 minutes at 800°C or 850°C is sufficient to ensure an increased protection against embrittlement (Tables 4,6). Based on limited data, it can be said that quenching did not improve the strength. The treatment is effective for plates under a simultaneous thermal exposure and a 150-MPa applied tensile stress (Table 6). This stress is sufficient to open existing cracks and cause some limited crack propagation, as determined by surface replicates and acoustic emission [14]. The addition of a compressive stress at 650°C might be expected to lessen the embrittlement by mechanically closing surface cracks. At a moderate compressive stress level (60 MPa), however, there is no definitive effect (Table 7).

It has always been observed that any microcracking from tensile load smoothly closes upon removal of that load, especially for loads below the proportional limit. This has been reported for a unidirectional Nicalon*/LAS glass-ceramic composite [15]. We have seen in the Nicalon*/1723 glass material of this report that cracks after closure may not be visible even in SEM photographs. The result for a specimen which is preloaded in tension and then

exposed is still somewhat unexpected (Table 3). The stress-strain curve in tension (Figure 7) clearly shows that 350 MPa is sufficient for nonlinearity and cracking. When the plate is preloaded to 350 MPa, it loses about 10% of its strength, with the standard exposure causing an additional 26% loss. If the specimen undergoes only the standard exposure, it loses 25% of its strength. In this sense, then, little difference is seen in the strength loss attributable to embrittlement whether the plate has been preloaded or not.

A remarkable consistency was demonstrated for the strength results for the "89Cxx" plates exposed to the same conditions. The embrittlement experienced after the standard 16-hour, 650°C exposure produces plates with 75%, 73%, 77%, 72% and 64% of their original strengths, suggesting that a simple time-temperature degradation rule may be proposed. When plates are treated by a short 800°C or 850°C thermal treatment and then exposed 16 hours at 650°, retained strength values of 92% (one sample), 93% (one sample) and 99% are obtained after thermal exposure.

The rate at which specimens embrittle appears to slow as embrittlement proceeds (see Table 3). The rate of strength loss slows after the initial strength drop. A previous study [1,7] in a SiC/LAS glass-ceramic system attributed this loss to the replacement of interfacial C by amorphous SiO₂, with a large (80%) net positive volume change. This fills the space left by the disappearance of the interface, and "clamps down" or seals the interface against further oxygen infiltration. The data of Table 3 suggest that a similar mechanism may be operative in the SiC/glass system.

The curious difference in rapidity of embrittlement seen between "old" plates (88C14, from 1988) and other "new" plates (89Cxx, from 1989) remains unexplained. None of the variables systematically explored in Table 1 controls this behavior. It is clearly simply not the thickness of 88C14 (1.5mm vs.

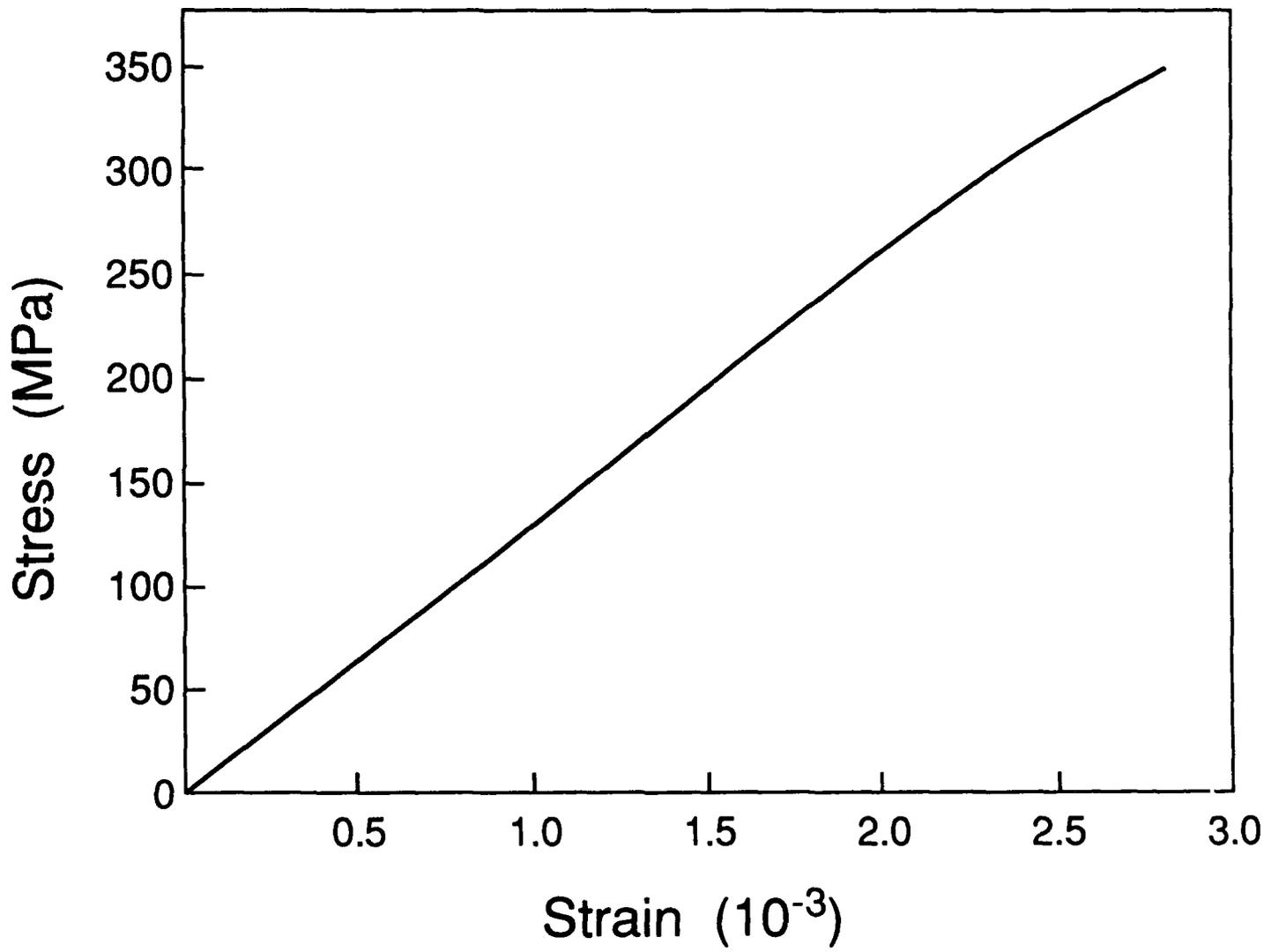


Figure 7 Tensile Stress-Strain Curve

other plates' 2.5mm) as may be seen from considering the diffusion of oxygen into the matrix. If it were simply a diffusion question, the 2.5mm composites would have reached an equivalent state of embrittlement with an exposure of $[(2.5/1.5)^2 \text{ times } 16 \text{ hour}]$, or 44 hours. (This corresponds to equivalent values of thickness/ time.) As seen in Table 3, the extreme embrittlement seen in 88C14 is not seen in the thicker 89C18, even after 144 hours. One possible explanation suggests that there was a chemical difference in the matrix, perhaps excess carbon from the binder, which caused the different behavior [16]. These islands of carbon in the binder could provide pipelines for quickly conveying the oxygen to the interface. Analytical SEM and TEM are being currently employed to describe this. However, the heat treatment is effective at limiting embrittlement of both types of plates (both quick and slow embrittling).

REFERENCES

1. E. Bischoff, M. Ruhle, O. Sbaizero, A.G. Evans, "Microstructural Studies of the Interfacial Zone of a SiC-Fiber-Reinforced Lithium Aluminum Silicate Glass-Ceramics," J Amer Cer Soc 72(1989) p. 741.
2. R.J. Kerans, et al., "The Role of the Fiber-Matrix Interface in Ceramic Composites," Ceram Bull 68(1989) p. 429.
3. J.J. Brennan, "Interfacial Chemistry and Bonding in Fiber-Reinforced Glass and Glass-Ceramic Composites," in Ceramic Microstructures '86, MRS V. 21, ed J.A. Pask, A.G. Evans, Plenum (1987).
4. R.F. Cooper, K. Chyung, "Structure and Chemistry of Fiber-Matrix Interfaces in SiC-Fiber-Reinforced Glass-Ceramic Composites: An Electron Microscopy Study," J Mat Sci 22(1987) p. 3148.
5. N.D. Corbin et al., "Microstructure/Structure Relationships for SiC Filament-Reinforced RBSN," Cer Eng Sci Proc 7(1986) p. 958.
6. E. Menessier, et al., "Thermo-mechanical Characterization of Ceramic Composites Made of A LAS Glass-Ceramic Matrix Reinforced with Silicon Carbide (Nicalon) Fibers," in Developments in the Science and Technology of Composite Materials (ECCM 3) ed AR Bunsell, et al., Elsevier, NY, (1989).
7. E.Y. Luh, A.G. Evans, "High-Temperature Mechanical Properties of a Ceramic Matrix Composite," J Amer Cer Soc 70(1987) p. 466.
8. L.P. Zawada, R.C. Wetherhold, "The Effects of Thermal Fatigue on a SiC Fiber/Aluminosilicate Glass Composite," J Mat Sci, in press (1990).
9. K.M. Prewo, B. Johnson, S. Starrett, "Silicon Carbide Fibre-Reinforced Glass-Ceramic Composite Tensile Behavior at Elevated Temperature," J Mat Sci 24 (1989) p. 1373.

10. R. Gardon, "Thermal Tempering of Glass," in Glass Science and Technology V.5, ed D.R. Uhlmann, N.J. Kreidl, Academic Press (1980).
11. R. Gardon, J. Cobonpue, "Heat Transfer Between a Flat Plate and Jets of Air Impinging on It," in International Developments in Heat Transfer (Proc Int Heat Transf Conf, 2nd), ASME, NY (1961).
12. Anon, Material Data Safety Sheet, Corning Glass, Corning, NY (1986).
13. T.-I. Mah, et al., "Thermal Stability of SiC Fibers (Nicalon)," J Mat Sci, 19 (1984) p. 1191.
14. R. Kim, N.J. Pagano, "Initiation of Damage in Unidirectional Brittle Matrix Composites," in Proc 4th US - Japan Conf on Compos Mat, Technomic (1988) p. 799.
15. D.B. Marshall, A.G. Evans, "Failure Mechanisms in Ceramic-Fiber/Ceramic-Matrix Composites," J Amer Cer Soc 68(1985) p. 225.
16. S.C. Farmer, personal communication, NASA-Lewis, (3/90).