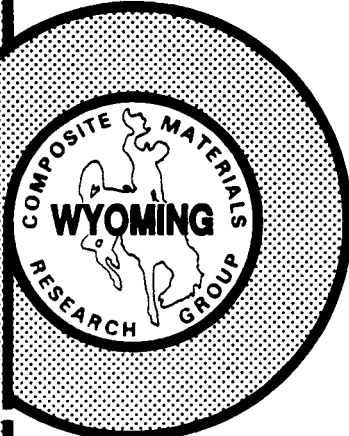


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# STATIC TENSILE AND TENSILE CREEP TESTING OF FIVE CERAMIC FIBERS AT ELEVATED TEMPERATURES



Richard S. Zimmerman  
Donald F. Adams

December 1988

FINAL REPORT

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## PREFACE

This technical report presents the results of a ten-month study, sponsored by NASA-Ames Research Center under Purchase Order No. A39603C, dated April 22, 1986. Dr. Demetrius A. Kourtides served as the NASA-Ames Technical Monitor.

All work was conducted by the Composite Materials Research Group (CMRG) within the Department of Mechanical Engineering at the University of Wyoming. Co-Principal Investigators were Mr. Richard S. Zimmerman, Staff Engineer, and Dr. Donald F. Adams, Professor. Making significant contributions to this program were Messrs. Byron Johnson and Hal Radloff, undergraduate students in Mechanical Engineering and members of the Composite Materials Research Group.

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## SECTION 1

### INTRODUCTION

Five types of ceramic fibers were static tensile tested at room temperature and four elevated temperatures, and creep tested at four elevated temperatures. To do so, single fibers were separated from tows supplied by NASA-Ames. Initially, the five ceramic fibers chosen for this program were J.P. Stevens Co. Astroquartz 9288 glass fiber, Nippon Carbon, Ltd. (Dow Corning) Nicalon NLM-102 silicon carbide fiber, and 3M Company Nextel 312, Nextel 440, and Nextel 480 alumina/silica/boria fibers. It proved to be impossible to separate single Nextel 440 fibers from the tows supplied because of the fiber sizing present and the brittle nature of these fibers. Therefore, Nextel 380 was substituted for the Nextel 440 in this program.

The complete test matrix is given in Table 1. ASTM Standard Test Method D3379-75 was used as the reference test procedure for all testing [1].

Each type of fiber was initially static tensile tested at three gage lengths, viz., 1, 2, and 4 inches, at room temperature, to determine the magnitude of end effects from the paper end tabs and grips. No creep testing was performed at room temperature. Each type of fiber was also initially static tensile tested at two gage lengths, viz., 8 and 10 inches, at one of the elevated test temperatures, to determine end effects for the elevated temperature static and creep testing. An end effect correction was found to be unnecessary for the

Table 1

## Test Matrix for Each Type of Ceramic Fiber

Fiber Type	Test Method	Test Temperature (°C)	Gage Length (inches)	No. of Replicates
Astroquartz 9288	Static	25	1	10
			2	10
			4	10
		500	8	5
			10	5
			8	5
			8	5
	Creep	400	8	3
			8	3
			8	3
			8	3
		500	8	3
			8	3
			8	3
Nicalon NLM-102	Static	25	1	10
			2	10
			4	10
		1000	8	5
			10	5
			8	5
			8	5
		1100	8	5
			8	5
			8	5
	8		5	
	Creep	800	8	3
			8	3
		900	8	3
8			3	
8			3	
Nextel 312	Static	25	1	10
			2	10
			4	10
		400	8	5
			8	5
			10	5
			8	5
		500	8	5
			8	5
			8	5
	8		5	
	Creep	400	8	3
			8	3
		500	8	3
8			3	

Table 1 (continued)

## Test Matrix for Each Type of Ceramic Fiber

Fiber Type	Test Method	Test Temperature (°C)	Gage Length (inches)	No. of Replicates	
Nextel 380	Static	25	1	10	
			2	10	
			4	10	
		600	8	5	
			10	5	
			8	5	
			8	5	
	Creep	500	8	3	
		600	8	3	
		700	8	3	
		800	8	3	
	Nextel 480	Static	25	1	10
				2	10
4				10	
900			8	5	
			8	5	
1000			10	5	
			8	5	
Creep		700	8	3	
		800	8	3	
		900	8	3	
		1000	8	3	

elevated temperature testing. All subsequent elevated temperature testing, both static and creep, was thus performed utilizing only 8 inch long specimens.

As will be noted in Table 1, the creep test temperatures used were slightly lower than the static tensile test temperatures. This was to compensate for the much longer time the fibers being creep tested spent at the elevated temperatures, these high temperatures for long times causing severe degradation of the fibers. Static testing nominally subjected each fiber to the elevated temperature for only 1 to 3 minutes. The creep test duration was nominally four hours. Exposure in an air atmosphere at the highest temperatures for this comparatively long time was found to degrade the ceramic fibers too much. Thus, lower test temperatures for creep were utilized to ensure properties could be measured.

A summary of all of the test results is presented in the next section. Specimen fabrication techniques and descriptions of the test apparatuses used are included in Section 3. Test methods and detailed experimental results are given in Section 4. Conclusions are given in Section 5. Appendix A contains the individual single fiber static tensile test results, while Appendix B contains individual tensile creep test results. Appendix C contains additional SEM photographs taken by the Composite Materials Research Group and NASA-Ames Research Center and sent to the CMRG for inclusion in this report.



## SECTION 2

### SUMMARY OF RESULTS

Single fiber static tensile and tensile creep testing at various elevated temperatures was performed on five types of ceramic fibers supplied by NASA-Ames. All fibers were supplied as untwisted rovings, both sized and unsized. The single fibers used for all testing were carefully separated by hand from the unsized rovings. It was found that sized fibers could not be utilized since the fibers adhered to each other to such a great degree that individual fibers could not be extracted from the fiber bundles without damaging them.

Static testing was performed on each of the five types of ceramic fibers at five temperatures, viz., room temperature and four elevated temperatures, to provide baseline data for each fiber type. Creep testing revealed that the fibers degraded in only a short time (5 to 20 minutes) during air atmosphere exposure at the higher temperatures. Thus, the creep testing was performed at lower temperatures than those used for static testing. Because of the differences in temperature sensitivity of the different types of fibers, each type was tested at a slightly different set of elevated temperatures, selected to be consistent with the sensitivity of that particular fiber. It should be noted that those fibers that performed the best at the highest elevated temperatures when tested in static tension also exhibited the lowest creep rates. All creep tests were conducted at a stress level approximately equal to 80 percent of the corresponding static tensile strength measured at each fiber's highest test temperature. This creep stress level stress level was set by NASA-Ames. The strain measurement

limit was 2 percent, dictated by the small LVDTs used to measure fiber displacements and the long specimen gage lengths.

Data are presented in graphical form in this summary, to provide the reader with relative magnitudes and temperature capabilities for the five different types of ceramic fibers. As will be noted, the test temperatures were quite different. Thus, comparisons of fiber properties should be made carefully, to avoid improper conclusions as to the capability of each fiber at elevated temperature. For each of the types of fibers, testing was performed up to the highest temperature that still yielded reasonable mechanical properties. It was found that properties fell dramatically above the highest temperatures reported here.

Average static tensile properties are shown in Figures 1 through 3 while average tensile creep rates are shown in Figure 4. As discussed in the Introduction, the maximum tensile creep test temperatures were slightly lower than those used for static testing. The average data presented here in graphical form are presented again in tabular form in Section 4. Individual test specimen results are presented in Appendices A and B.

Some experimental work had been performed previously by other researchers using fiber tows to determine similar properties for the five materials tested in this study [2-4]. Considerable effort was expended here in developing the required test apparatus for performing single fiber static tensile and tensile creep tests.

Single fiber testing requires a great deal of patience and care in specimen preparation due to the fragile nature of the fibers and the affinity of the fibers for other fibers in the tow. Careful handling is

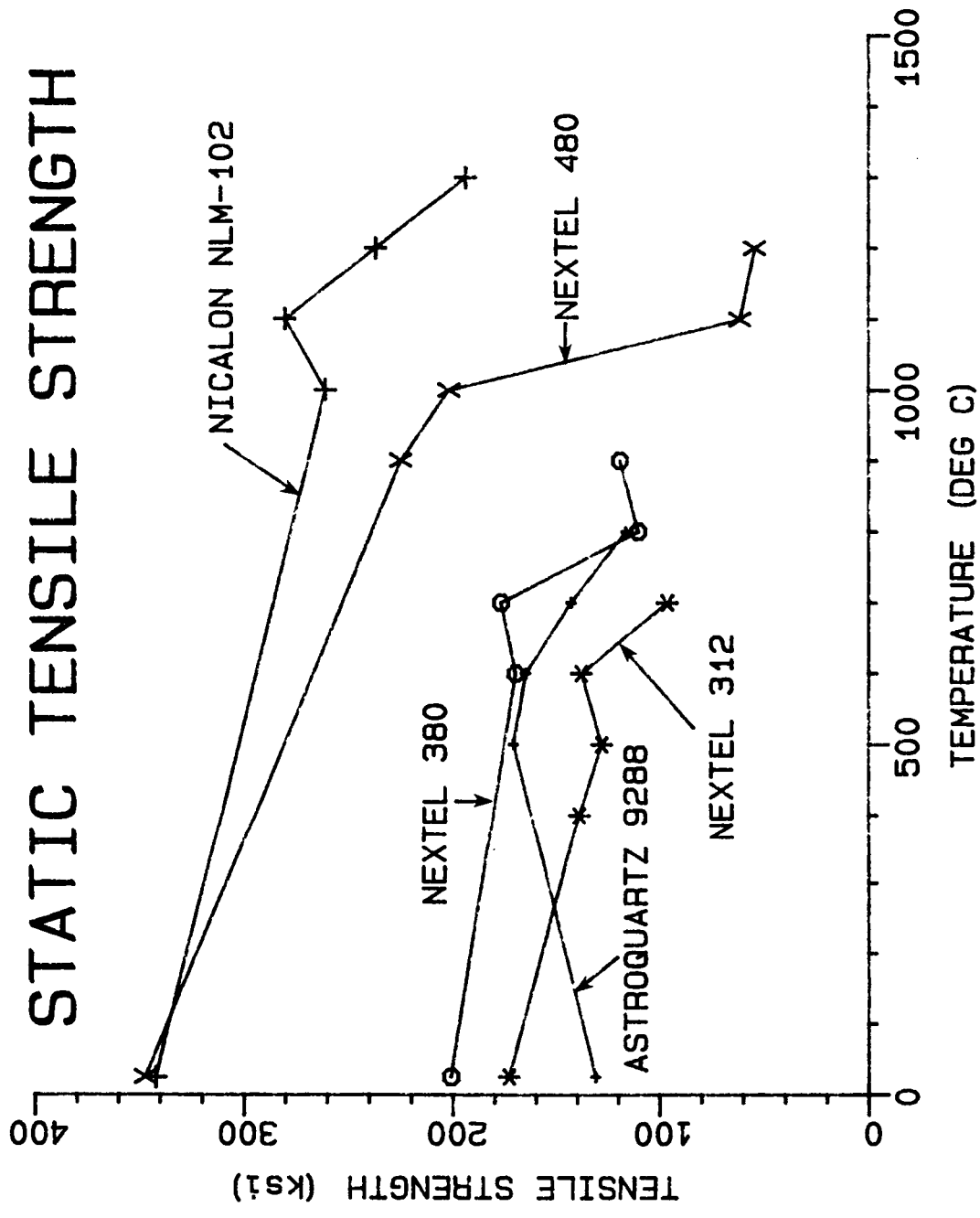


Figure 1. Tensile Strengths of Ceramic Fibers as a Function of Test Temperature.

# STATIC TENSILE MODULUS

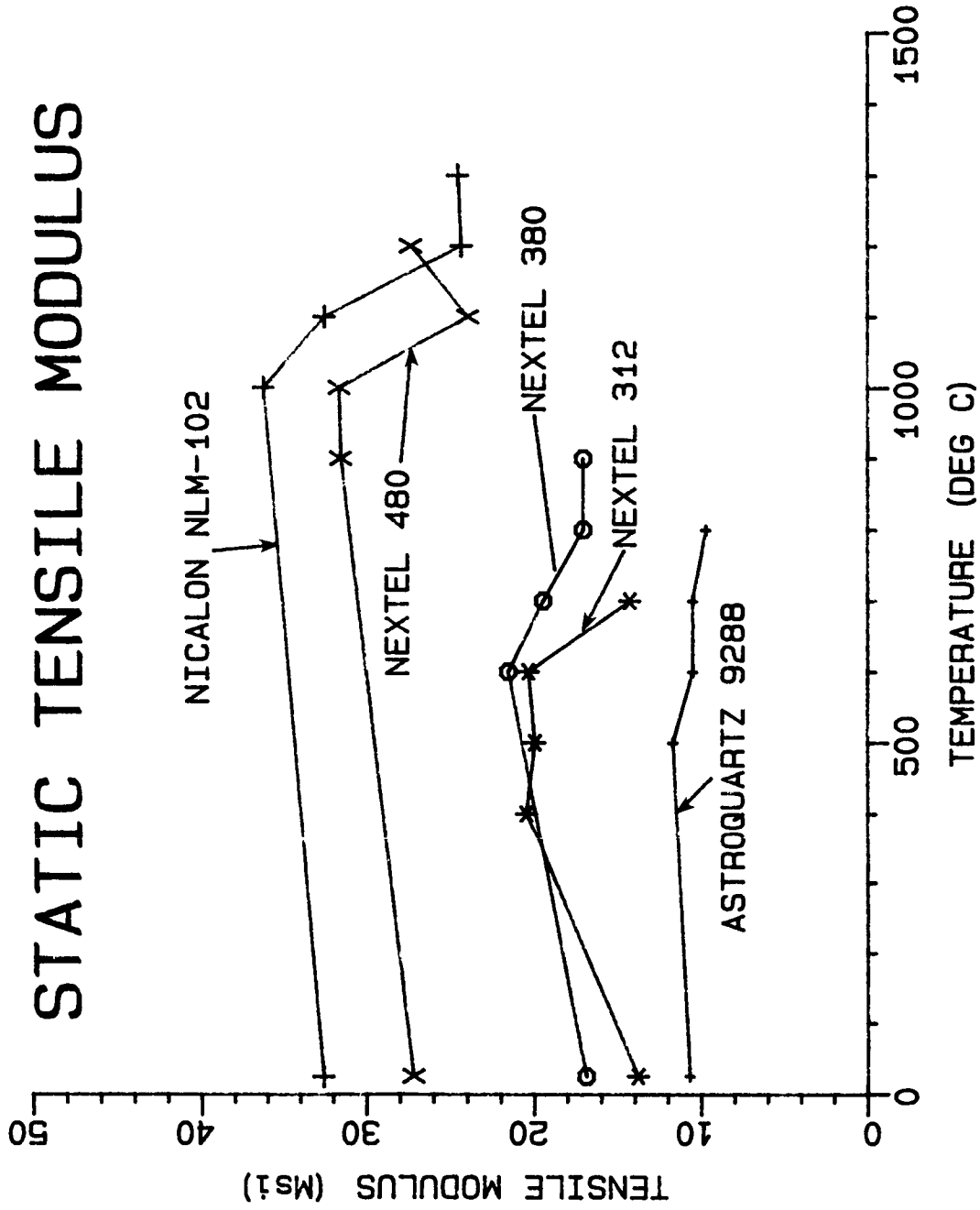


Figure 2. Tensile Moduli of Ceramic Fibers as a Function of Test Temperature.

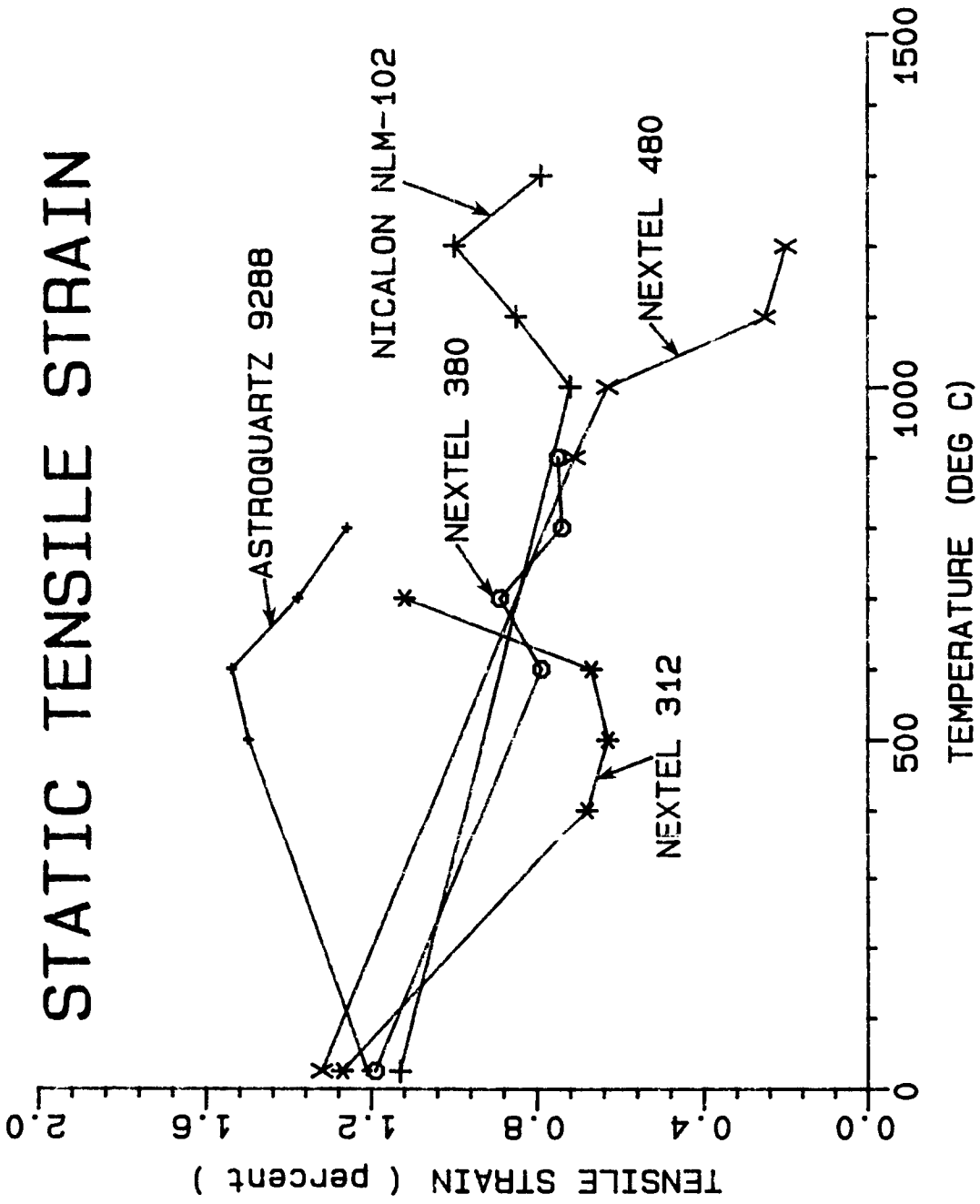
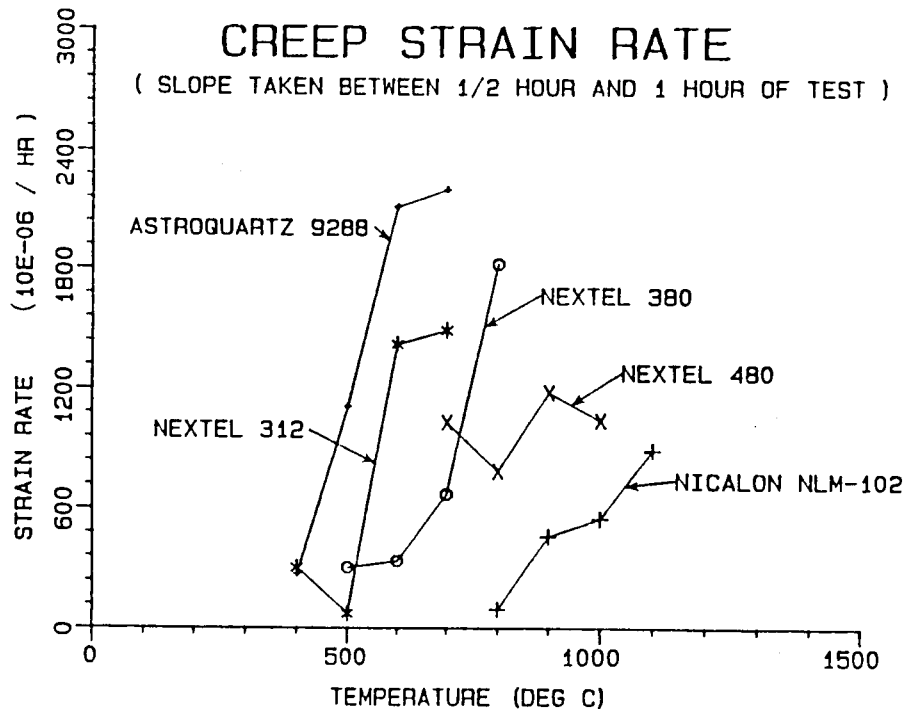
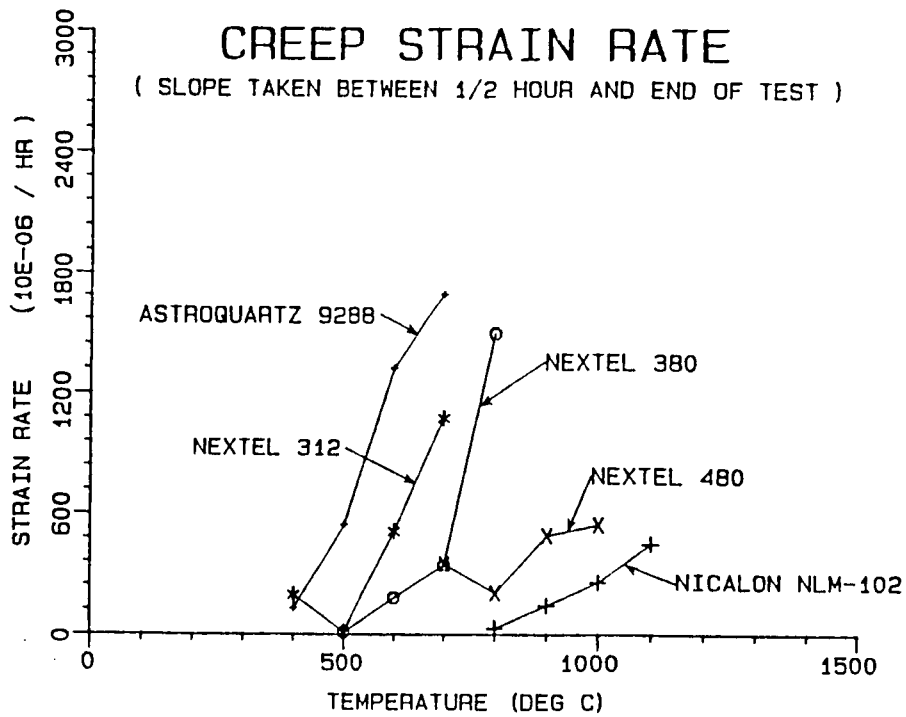


Figure 3. Ultimate Tensile Strain of Ceramic Fibers as a Function of Temperature.



a) Tensile creep rates calculated between one-half hour and one hour.



b) Tensile creep rates calculated between one-half hour and end of test.

Figure 4. Tensile Creep Rates of Ceramic Fibers as a Function of Temperature.

also critical once specimens are fabricated, to ensure the fiber specimens are not damaged prior to testing.

After testing, each specimen was examined in an optical microscope to determine the fiber diameter and cross-sectional shape. Also, a scanning electron microscope (SEM) was used to photograph groups of fibers, to provide an overall indication of the fiber diameters and shapes, and their variations.

The test specimen configuration and test apparatuses used for static tensile and tensile creep are described in detail in the next section.

## SECTION 3

### SPECIMEN FABRICATION AND TEST METHODS

#### 3.1 Specimen Fabrication

Single fibers were carefully separated from tows supplied by NASA-Ames, using a lighted magnifying lens. Small tweezers and gentle pulling with fingers were necessary to remove single fibers from the tows. Due to the delicate nature of the ceramic fibers, often many attempts were necessary before an intact fiber was successfully separated from a tow. Also, as previously noted, only unsized tows could be used; the fibers stuck to each other too firmly in the sized tows.

Single fiber test specimens were prepared from the five different types of ceramic fibers following the guidelines in ASTM Standard Test Method D3379-75 [1]. Figure 5 is a photograph of typical single fiber specimens of different gage lengths used in the room temperature testing.

A test specimen was prepared as follows. A stiff paper card (e.g., an index card) was cut into one-inch wide pieces, of a length appropriate for the gage length of the particular fiber to be tested. A diamond-shaped cutout was then made in the center region of this card, leaving a narrow strip of paper at each side of the one-inch width. A single fiber was stretched along the centerline of the length of the cutout, and temporarily held in place with masking tape near each outer end of the card. A small drop of adhesive was then placed at each end of the cutout, to permanently hold the fiber in place on the card. Thus, the length of the diamond-shaped cutout in the card defined the



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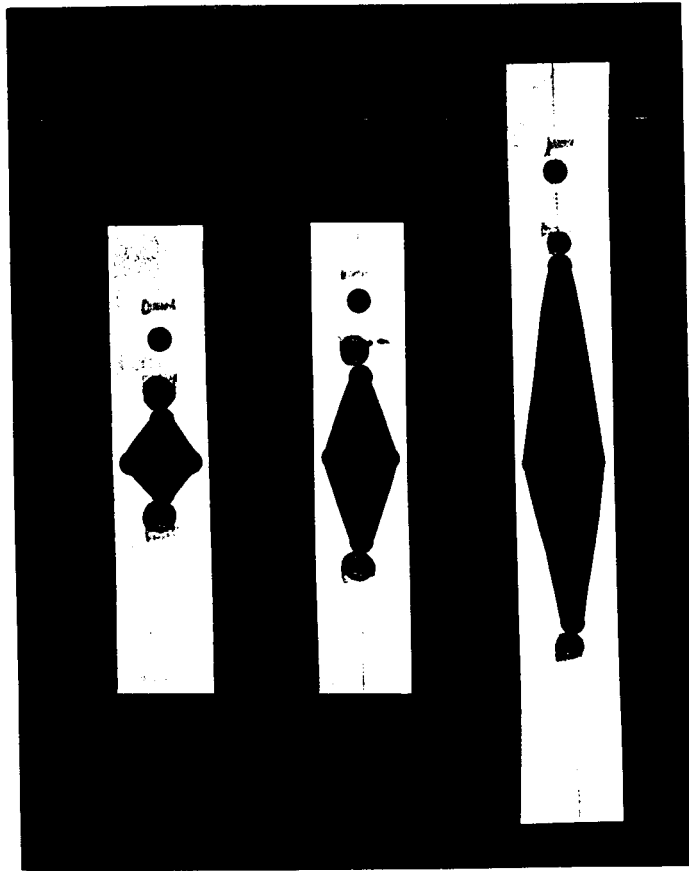


Figure 5. Typical Single Fiber Test Specimens Used for All Room Temperature Static Tensile Testing.

gage length for the fiber test specimen. For example, the test specimens used in the room temperature static tensile testing were prepared by making the length of the diamond-shaped cutout either 1, 2, or 4 inches, as shown in Figure 5.

The adhesive used was Type A-12 manufactured by Techkits, Inc. of Demarest, New Jersey. This adhesive is a two-part epoxy adhesive used extensively for bonding tabs onto composite material specimens, and has good mechanical properties up to 177°C. This adhesive could be used for all testing, including that at elevated temperatures, since the bond areas were outside the furnace and thus remained well below 177°C.

The 8 and 10 inch gage section specimens used for the elevated temperature static tensile and tensile creep testing were fabricated as follows. First, two pieces of the same paper card material used for the room temperature static testing, each piece being approximately one-inch square, were taped onto a large piece of corrugated cardboard, at a distance of 8 or 10 inches apart, depending upon the test specimen gage length desired. A single fiber was then temporarily held in place between the two pieces of card, using masking tape near the outer end of each card. The fiber was then bonded to each card using a small drop of the Techkits A-12 epoxy. Many of these single fiber specimens could be mounted on one large piece of corrugated cardboard, for safe storage prior to testing.

A special clip assembly consisting of a stiff metal rod separating two small alligator clips was used when transferring each single fiber test specimen from the corrugated cardboard storage board into the grips of the testing machine. This permitted the insertion of the single fiber specimen into the grips of the testing machine without damaging

the fiber. The clip assembly was removed just prior to testing, leaving the fiber to carry the applied force. Figure 6 shows a typical single fiber test specimen being held by the special clip assembly used for all elevated temperature testing.

### 3.2 Room and Elevated Temperature Static Tensile Test Apparatuses

All room temperature static tensile testing was performed using an Instron Model 1125 electro-mechanical testing machine with a 20 Newton load cell and special spring-loaded fiber grips manufactured by Instron Corporation. A test specimen such as one of those shown in Figure 5 was placed in the grips and an Engel Type HSGO heat cutter, with a Type 100R cutting blade, was used to burn through the two narrow sides of the paper card just prior to testing. Force versus crosshead displacement data were acquired using the integral strip chart recorder on the Instron Model 1125 testing machine.

Elevated temperature static tensile testing was performed using an MTS Model 810 servo-hydraulic testing machine, and the same 20 Newton load cell and fiber grips used for the room temperature testing in the Instron testing machine. The test specimen configuration was as shown in Figure 6. A Hewlett-Packard Model 7044A X-Y recorder coupled to the MTS Model 810 testing machine was used to record load versus crosshead displacement data for each test specimen.

Temperature chambers fabricated by the CMRG specially for this testing program were used to heat the test section of the single fiber specimen. Considerable consultation and assistance relative to temperature chamber size, shape, and configuration was provided by Dr. Demetrius Kourtides of NASA-Ames.

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Figure 6. Single Fiber Test Specimen and Special Clip Assembly Used for All Elevated Temperature Testing.

Two pieces of Greenlite 33 refractory brick, purchased from A.P. Green of San Fransisco, California, were bolted together, with a spacer between each half to form a vertical slot. Cavities were machined in the refractory brick to provide spaces for wire-wound heating elements. Kanthal A-1 18-gauge wire was purchased from Duralite, Inc., Riverton, Connecticut, for use as the heating element wire. Initially, short circuits and heater burnouts within the temperature chambers were common occurrences, usually due to the heater elements distorting and sagging since no support cores were used. Machinable ceramic rod was then purchased from McMaster-Carr, Chicago, Illinois, and used to provide support for the heating coils, as found to be particularly necessary when testing at the highest test temperatures. A spiral groove was machined around the ceramic rod circumference to hold the heater wire in place. Figure 7 shows a typical heater assembly with heater wire and ceramic core.

The temperature chamber was mounted on a slide attached to the MTS Model 810 testing machine, which allowed it to be pushed back out of the load train between tests, making installation of the next specimen much easier. The slide assembly also permitted vertical adjustment of the temperature chamber to accommodate test specimens of different lengths, by the use of spaced holes in its vertical support columns. Figure 8 shows a typical temperature chamber mounted on the slide assembly, with a single fiber specimen in place. The upper paper tab and fiber grip is hidden by an aluminum foil radiation shield.

A thermocouple was used to monitor temperature in each temperature chamber. A Type "S," platinum/platinum-5% rhodium, thermocouple was used, which provided good resolution in the temperature range from 500

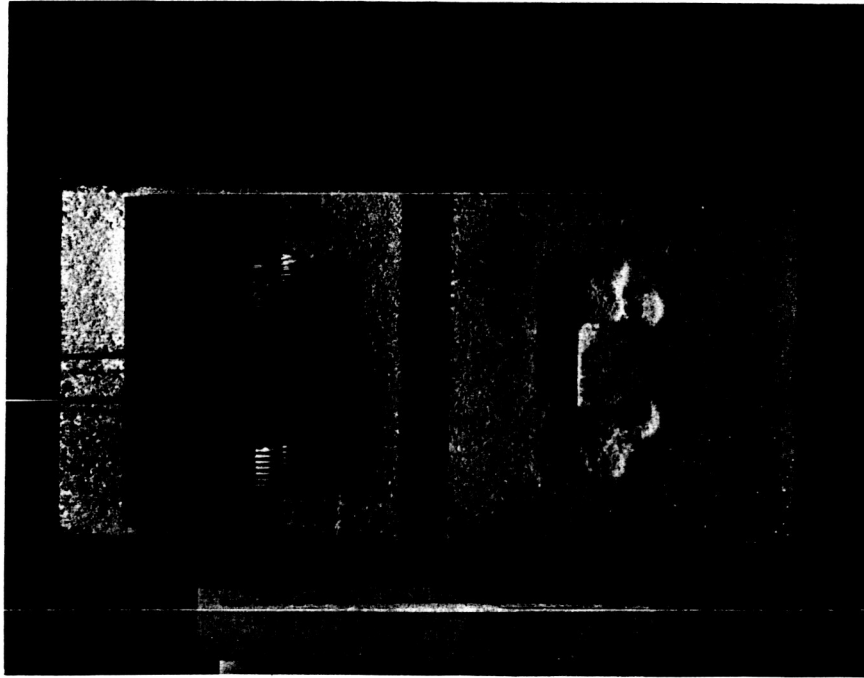


Figure 7. Two Halves of High Temperature Furnace, Showing Heater Elements, Grooved Ceramic Cores, Power Leads, and Machined Cavities for Heater Elements.

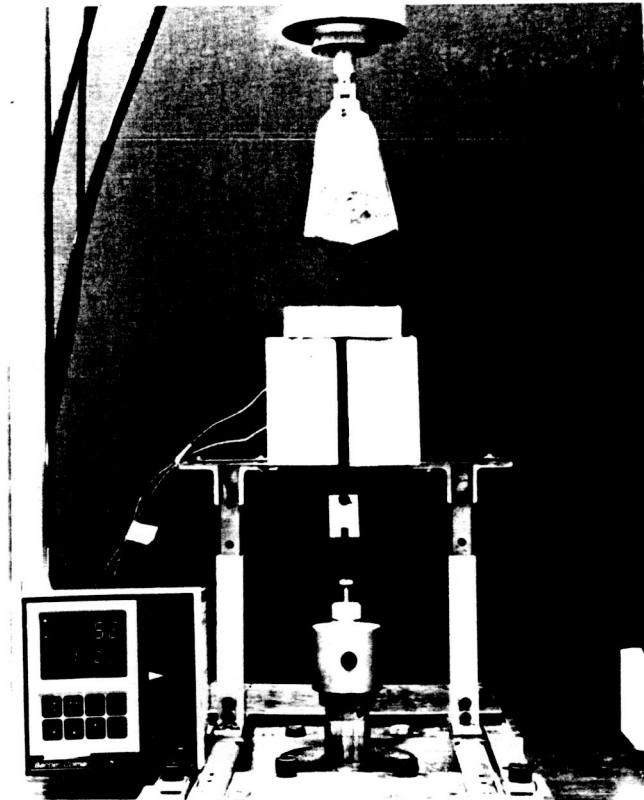


Figure 8. Single Fiber Tensile Test Setup, Showing the Temperature Chamber, Controller, Lower Grip, and Aluminum Heat Shield Around the Upper Grip.

to 1300°C. The thermocouple was mounted in the temperature chamber at the middle of the heated zone, to monitor the temperature and thus to allow for controlling the furnace temperature. The temperature was controlled using a Barber-Coleman Model 5643 digital temperature controller calibrated for the Type "S" thermocouple. The temperature controller was capable of maintaining the temperature in the chamber to within 5 to 10°C of the setpoint value, as determined by temporarily installing and simultaneously monitoring two additional thermocouples.

High amperage (25 amp) silicon controlled rectifiers (SCR's) were coupled to the controllers to actually supply power to the heating elements. Auxiliary heater elements wired in series with the temperature chamber elements were necessary to lower the amperage passing through the circuit since the heating elements were of too low resistance to limit the current by themselves. Electric water heater elements were utilized as the auxiliary heater elements. The auxiliary heaters were housed in water jackets to remove the heat generated when operating the heaters.

A top cover plate for the chamber was constructed using one-inch thick rigid insulation. This cover plate was used to minimize convective air currents within the temperature chamber, and thus help minimize any thermal gradients within the chamber. This cover plate contained a small slot, to permit it to pass around the fiber specimen. The cover was then rotated 90°, to minimize the opening. This resulted in a small hole at the top of the chamber for the fiber to pass through. A 30 to 50°C thermal gradient from the top to the bottom of the chamber typically still existed even with the cover plate in place, as again

determined using two auxiliary thermocouples positioned away from the primary sensor.

As shown in Figure 8, the the top and bottom paper tabs of the single fiber test specimen, held in the testing machine by the spring-loaded grips, were well outside of the heat zone created by the temperature chamber. The top grip and top paper tab region were further protected from convective heat currents by using an aluminum foil heat shield positioned between the furnace and the top paper tab area, as shown in Figure 8. This heat shield contained a small slot to allow the fiber to pass through untouched.

### 3.3 Creep Test Apparatus

A special creep test facility was designed and fabricated, to permit the testing of single fibers at very high temperatures while accurately measuring fiber extension (creep) throughout the nominal four-hour duration of a typical creep test. Figure 9 shows the single-fiber creep frame assembly, with controllers, temperature chambers, grips, linear variable differential transformers (LVDT'S), dead weights, wind shields, and auxiliary heaters in place. Four independent creep frames were used, to allow for multiple specimen testing.

The four Barber-Coleman Model 5643 temperature controllers shown mounted on top of the creep apparatus in Figure 9 were of the same type used with the static testing apparatuses described in the previous subsection. The temperature chambers were also of the same construction as used for the static tensile testing.



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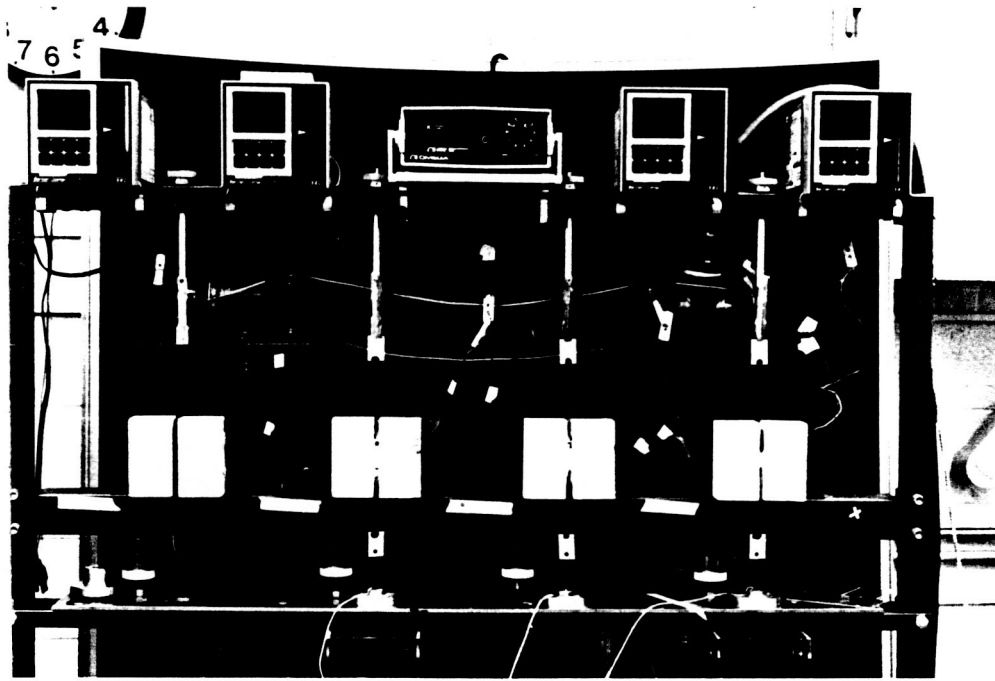


Figure 9. Four-Station Single Fiber Creep Apparatus Showing Temperature controllers, Temperature Chambers, Wind Shields, and LVDT Instrumentation.

The bottom grip assembly was different, however, to permit the hanging of dead weights at the bottom of the load train, and to permit the measurement of fiber extension (creep) by the use of LVDT's mounted between the fibers and the dead weights. An adjustable hook was used to hold the lower fiber tab stationary while hanging the dead weights and adjusting the vertical position of the upper load train to zero the LVDT's, to prevent premature loading of the fiber. Wind shields were used to surround the dead weights, to prevent natural air currents in the testing laboratory from inducing any movement of the lower portion of the load train.

Data were acquired using a Northstar Z-80 computer. Temperature readings from the thermocouples and extensions from the LVDT's were recorded by the computer and stored on a floppy disk for later retrieval and transfer. After completion of a test, the data were transferred to a Control Data Corporation Cyber 760 mainframe computer for reduction and plotting. All design and construction of both the static test apparatus and the creep apparatus was performed by the Composite Materials Research Group at the University of Wyoming.

### **3.4 Room and Elevated Temperature Static Tensile Test Methods**

The guidelines included in ASTM Test Standard D3379-75 [1] were followed in performing all of the single fiber testing in this study.

Room temperature static tests were performed using the Instron Model 1125 testing machine with spring-loaded single fiber grips previously described. Grip spacing was set to the appropriate distance to accommodate the three different gage length specimens used in the room temperature testing, viz., 1, 2, and 4 inches. A specimen was

placed in the grips and the narrow sides of the paper card were carefully burned through using the heat cutter. This left the single fiber unsupported, free to carry the applied force directly. Loading rate was set at 1 mm/minute (0.04 inch/minute). Force-displacement plots were obtained using the chart recorder built into the Instron testing machine.

After failure, each fiber was examined in an optical microscope to determine its diameter and cross-sectional shape, for comparison with scanning electron microscope (SEM) measurements. Individual fiber tensile strength, modulus, and strain to failure was calculated, using the average fiber cross-sectional areas determined from the SEM photographs.

A static tensile test procedure for elevated temperature testing was developed using the experience gained while testing at room temperature. The MTS Model 810 load frame was set up with the single fiber grips and the special temperature chamber previously described. A single fiber test specimen was then transferred to the testing machine using the two clips and rod assembly. The paper tabs were mounted in the grips with as little initial tension in the fiber as possible. Some slight tension was necessary, however, to prevent the fiber from moving due to the convective currents generated in the temperature chamber. This slight preload was equivalent to the weight of the bottom paper tab, i.e., about 0.3 grams, which was sufficient to prevent the fiber from accidentally contacting the heating elements prior to testing.

The temperature chamber was slid forward along the support slides until the thermocouple in the chamber was as close as possible to the fiber without making contact. The one-inch thick block insulation with

a slot in one side was then placed around the fiber at the top of the temperature chamber and rotated 90°. The slotted heat shield of aluminum foil was placed as close as possible around the fiber above the furnace to protect the top paper tab and grip. Loading was begun when the furnace stabilized at the desired test temperature. Temperature chamber heat-up typically took only 1 to 3 minutes.

Crosshead rate was set at 1 mm/minute (0.04 inch/minute). Load-displacement plots were made directly on a Hewlett-Packard 7044-A X-Y recorder. Tensile strength, modulus, and strain were calculated using average fiber diameter measurements taken from SEM photographs.

### 3.5 Single Fiber Creep Test Method

Single fiber creep testing, only performed at elevated temperatures, was achieved utilizing the special single fiber creep frame designed and constructed for this program. The creep tests were more complicated than the static tensile tests, due to the need to ensure zero applied force at the start of the creep test, and the need to accurately monitor the displacement for a period of four hours. Exposing the ceramic fibers to high temperatures in an air environment for four hours presented problems for the fibers as well as the heating elements in the temperature chambers.

The fibers were severely degraded at the higher temperatures used for the static testing and thus were necessarily creep tested at lower temperatures. The heating elements required replacement periodically due to their excessive creep characteristics at the high test temperatures, which caused them to sag excessively, and fail.

Before beginning a test, the support arm and wire hook assembly of the fiber loading system was raised until the LVDT core was at the top of the calibrated range. The top fiber tab was then mounted in the top grip. The bottom tab was placed over the bottom wire hook using the vertical adjustment nut. The appropriate dead weight for the test was hung on the bottom hook under the load frame and the wind shield was placed around the weight and lower hook and wire. No force was yet sensed by the fiber since the bottom tab was still held by the support arm hook. The top grip was moved back and forth to position the fiber adjacent to the thermocouple in the temperature chamber.

The one-inch thick insulation block was then placed on top of the chamber and rotated 90° to block off the top around the fiber. The data acquisition computer program was activated, to record temperature and displacement for the duration of the creep test. The support arm was then lowered away from the lower fiber tab and the fiber position was checked to ensure it was still located adjacent to the thermocouple in the furnace. Slight adjustments were sometimes necessary to center the fiber in the chamber at the beginning of the creep test.

Creep tests were run for four hours, with the computer acquiring and recording the data on a 5 1/4 " floppy disk. Load, temperature, and displacement data were then transferred to the Control Data Corporation Cyber 760 mainframe computer for later plotting and reduction.

Calculations of fiber stress, strain, and modulus were performed using the average fiber cross-sectional area measurements obtained using the SEM, as discussed in Section 4.1. Two discrete creep rates were calculated for each fiber by calculating the slope of the strain versus

time curve between  $\frac{1}{2}$  hour and one hour of creep and between  $\frac{1}{2}$  hour and the end of each creep test, as discussed in Section 4.3.

After each test, the fiber was removed and examined in an optical microscope. Fiber geometry data were compared to average SEM measurements for each fiber type.

## SECTION 4

### EXPERIMENTAL RESULTS

The five fibers tested were J.P. Stevens Astroquartz 9288 glass fiber, Nippon Carbon, LTD. (Dow Corning) Nicalon NLM-102 silicon carbide fiber, and 3M Company Nextel 312, Nextel 380, and Nextel 480 alumina/silica/boria fibers. These five ceramic fibers were static tensile tested at room temperature and four elevated temperatures, and tensile creep tested at the four elevated temperatures listed in Table 1 of Section 1. No creep testing was performed at room temperature.

Mechanical properties as a function of temperature, viz., Young's modulus, tensile strength, tensile strain, and creep strain, for the five types of fibers were then determined at each test temperature.

Average test results are presented here while individual test data are given in Appendices A and B.

#### 4.1 Optical and Scanning Electron Microscope Observations

All fiber types were photographed using a scanning electron microscope (SEM) to determine their typical shapes and diameters. This determination was very important since it was necessary to calculate the fiber cross-sectional areas. Optical microscope measurements were also made for all types of fibers, and the results compared with the SEM measurements. The optical and SEM measurements were in reasonable agreement, with the optical measurements typically being slightly smaller. This may have been due to the lower magnification used and some lighting problems encountered with the optical microscope measurements. For the present study, all data were reduced using the

average cross-sectional area values determined from the SEM photographs. Due to fiber to fiber size variations, this introduced some scatter in the data, particularly for the types of fibers that exhibited greater geometric variations.

The five types of fibers were found to conform closely to the corresponding sizes and shapes published in the literature [3-5, 7]. For example, the Astroquartz 9288 glass fibers were observed to be round in cross-section, with an average diameter of 8.75 microns. A typical literature value was 9.0 microns [5]. Figure 10 shows an SEM photograph of a group of Astroquartz fibers.

The Nicalon NLM-102 fibers were also round in cross-section, with an average diameter of 10-15 microns, comparing reasonably well with the range of 10-20 microns given in the literature [3]. Figure 11 is an SEM photograph of a group of Nicalon fibers, showing the variability in size observed.

The three types of Nextel fibers tested were found to all be roughly elliptical in shape, with consistent dimensions between the three types. That is, they had similar minor and major diameters, varying from 8 to 13 microns, which were consistent with the values published in the literature [4].

Reference [8] contains an equation, repeated below as Eq. (1), used by 3M Company to calculate the approximately elliptical area of the Nextel fibers.

$$\text{Area} = \{\text{major dia.}\} \times \{\text{minor dia.}\} \times (0.87) \quad (1)$$



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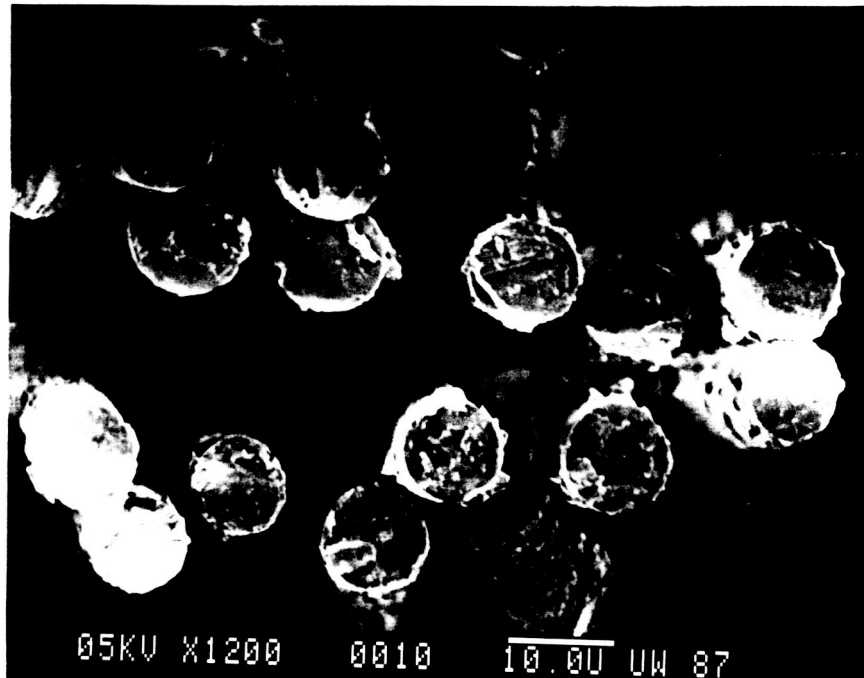


Figure 10. Scanning Electron Microscope Photograph of a Group of Astroquartz 9288 Fibers.

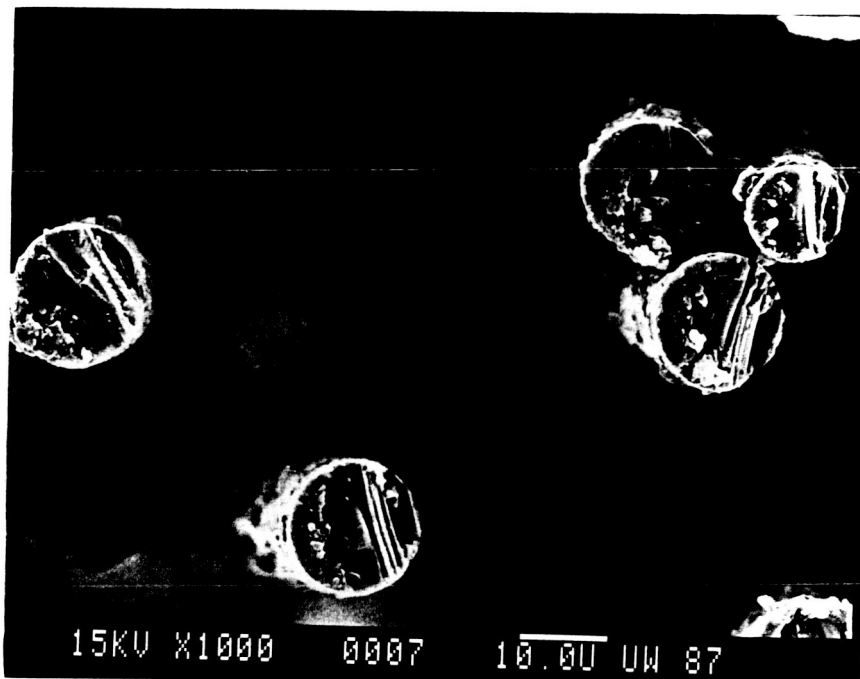


Figure 11. Scanning Electron Microscope Photograph of a Group of Nicalon NLM-102 Fibers.

The equation for a true ellipse is:

$$\text{Area} = (\text{major dia.}) \times (\text{minor dia.}) \times (\pi/4) \quad (2)$$

Following the recommendation of the manufacturer of the Nextel fibers, viz., the 3M Company, Eq. (1) was used in making the calculations of the present study.

Figures 12, 13, and 14 are SEM photographs of groups of Nextel 312, Nextel 380, and Nextel 480 fibers, respectively.

NASA-Ames performed additional scanning electron microscope photography of some of these same fibers, photographs which they provided being included in Appendix C along with additional SEM photographs taken at the University of Wyoming.

Comparisons of fiber sizes and shapes obtained from the various microscope studies performed are given in Table 2. As will be noted,

Table 2  
Average Fiber Dimensions as Measured from  
Optical and Scanning Electron Microscope Photographs

Fiber Type	Fiber Shape	UW Optical ( $\mu\text{m}$ )	UW SEM ( $\mu\text{m}$ )	NASA-Ames SEM ( $\mu\text{m}$ )
Astroquartz 9288	round	6.7	8.75	8.84
Nicalon NLM-102	round	11.7	13.8	-
Nextel 312	ellip.	7.5 by 11.9	7.8 by 12.5	9.2 by 13.9
Nextel 380	ellip.	7.2 by 12.3	8.2 by 13.2	-
Nextel 480	ellip.	6.7 by 11.1	7.2 by 11.5	-

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Figure 12. Scanning Electron Microscope Photograph of a Group of Nextel 312 Fibers.



Figure 13. Scanning Electron Microscope Photograph of a Group of Nextel 380 Fibers.

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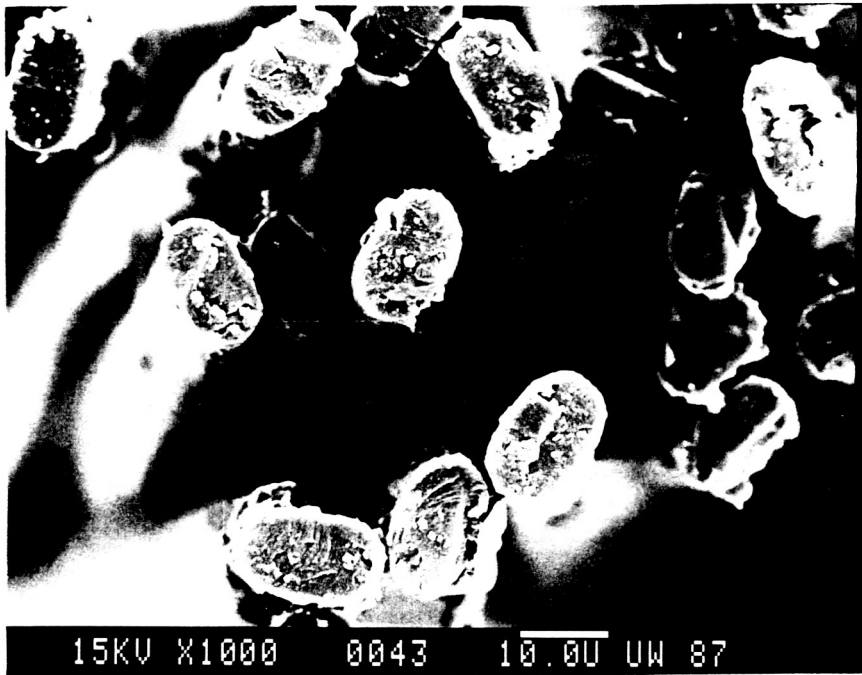


Figure 14. Scanning Electron Microscope Photograph of a Group of Nextel 480 Fibers.

photographs of only two of the five types of fibers were provided by NASA-Ames.

#### 4.2 Static Tensile Test Results

Table 3 presents average static tensile strengths, moduli, and strains to failure for the five types of ceramic fibers tested. The five types of fibers were tested at the highest temperatures that still gave reasonable mechanical properties for that type of fiber. Each fiber was tested at slightly different temperatures, as indicated in Tables 1 and 3.

Fiber strength was the major parameter used in establishing the highest temperatures the fibers were to be tested at. It will be noted in Table 3 that the fiber modulus values do not show as much of a drop as the fiber strengths. All of the fibers suffered losses in tensile strength at the higher test temperatures well before they exhibited much loss of stiffness.

Table 3 also presents average static tensile strains to failure for the five types of ceramic fibers tested in this program. As will be noted, the average ultimate strains for all of the fibers were less than two percent, and did not vary significantly as a function of test temperature.

#### 4.3 Creep Test Results

Table 4 presents average creep rates calculated in the time intervals between one-half hour and one hour, and between one-half hour and the end of the test (normally four hours). Tests were conducted at each of four elevated test temperatures. It will be noted that the four

Table 3

Average Tensile Properties for the Five Types of Ceramic Fibers  
Tested at Five Test Temperatures

Fiber Type	Gage Length (inches)	Test Temperature (°C)	Tensile Strength (ksi) (GPa)		Tensile Modulus (Msi) (GPa)		Tensile Strain (percent)
Astro-quartz 9288	1	25	131	0.90	10.7	74	1.21
	2		113	0.78	10.6	73	1.06
	4		97	0.67	11.4	79	0.85
	10	500	214	1.48	12.5	86	1.69
	8		171	1.19	11.6	80	1.47
	8	600	165	1.14	10.5	72	1.54
	8	700	143	0.99	10.5	72	1.38
	8	800	116	0.80	9.7	67	1.26
Nicalon NLM-102	1	25	349	2.40	32.6	225	1.13
	2		406	2.80	34.1	235	1.23
	4		361	2.49	39.2	270	0.91
	10	1000	215	1.48	32.1	221	0.66
	8		261	1.80	36.3	250	0.72
	8	1100	280	1.93	32.6	225	0.85
	8	1200	237	1.63	24.4	169	1.00
	8	1300	104	1.28	24.6	170	0.79
Nextel 312	1	25	173	1.19	13.8	95	1.27
	2		156	1.07	14.9	103	1.02
	4		111	0.76	17.4	120	0.63
	8	400	139	0.96	20.5	141	0.68
	10		500	128	0.88	18.9	130
	8	600	128	0.88	20.0	138	0.63
	8		138	0.95	20.4	140	0.67
	8		700	96	0.67	14.3	98
Nextel 380	1	25	201	1.39	16.9	116	1.19
	2		173	1.20	19.1	132	0.90
	4		151	1.04	18.0	124	0.83
	10	600	179	1.24	20.5	141	0.88
	8		170	1.17	21.6	149	0.79
	8	700	177	1.22	19.5	135	0.89
	8	800	110	0.76	17.1	118	0.74
	8	900	119	0.82	17.1	118	0.75
Nextel 480	1	25	347	2.39	26.3	181	1.33
	2		265	1.83	29.0	200	0.89
	4		256	1.77	30.0	207	0.86
	8	900	225	1.55	31.6	218	0.71
	10		1000	149	1.03	30.9	213
	8	1100	202	1.39	31.7	219	0.63
	8		61	0.42	24.0	166	0.25
	8		1200	54	0.37	27.4	189

Table 4

Average Creep Rates for the  
Five Types of Ceramic Fibers Tested

Fiber Type	Test Temperature (°C)	Applied Stress* (ksi)	Strain Rate from ½ Hour to 1 Hour (10 <sup>-6</sup> /hour)	Strain Rate from ½ Hour to End of Test** (10 <sup>-6</sup> /hour)
Astroquartz 9288	400	95	271	125
	500		1110	547
	600		2110	1320
	700		2200	1690
Nicalon NLM-102	800	162	94	29
	900		461	139
	1000		548	255
	1100		890	452
Nextel 312	400	77	301	190
	500		72	10
	600		1420	513
	700		1490	1080
Nextel 380	500	91	302	9
	600		335	177
	700		669	345
	800		1820	1490
Nextel 480	700	43	1030	350
	800		783	201
	900		1180	493
	1000		1040	547

\*Creep Stresses were 80% of ultimate stress at highest test temperature

\*\*Typically four hours

elevated test temperatures used for the creep testing of the five types of ceramic fibers were typically different than those used for the static tensile testing of each type of fiber, for the reasons previously discussed.

A typical strain versus time curve generated from the creep testing is presented in Figure 15, to illustrate the creep response observed for the ceramic fibers tested in the present study. The remainder of the creep curves are included in Appendix B. All fibers were loaded to 80 percent of the static ultimate stresses measured at the test temperatures given in Table 4. This 80 percent stress level was set by NASA-Ames, to provide creep data at a stress as close as practical to the ultimate strength of each type of ceramic fiber. Each creep test was terminated after four hours; recovery strain was not monitored.

The creep rates presented here (Table 4) were calculated using a linear regression technique to calculate the slopes between the two discrete time intervals. One time interval was between one-half hour and one hour elapsed time (i.e., near the beginning of each creep test). The second interval was between one-half hour and the end of each test, typically four hours total creep duration. Figure 15 illustrates the two slopes calculated from the strain versus time creep curves. The shorter time interval yielded a higher creep rate, as expected and as observed in Table 4 and on Figure 15. Some creep rates were actually measured as small negative values indicating the strains being measured were very small. These small negative values were not used in calculating the average values listed in Table 4.



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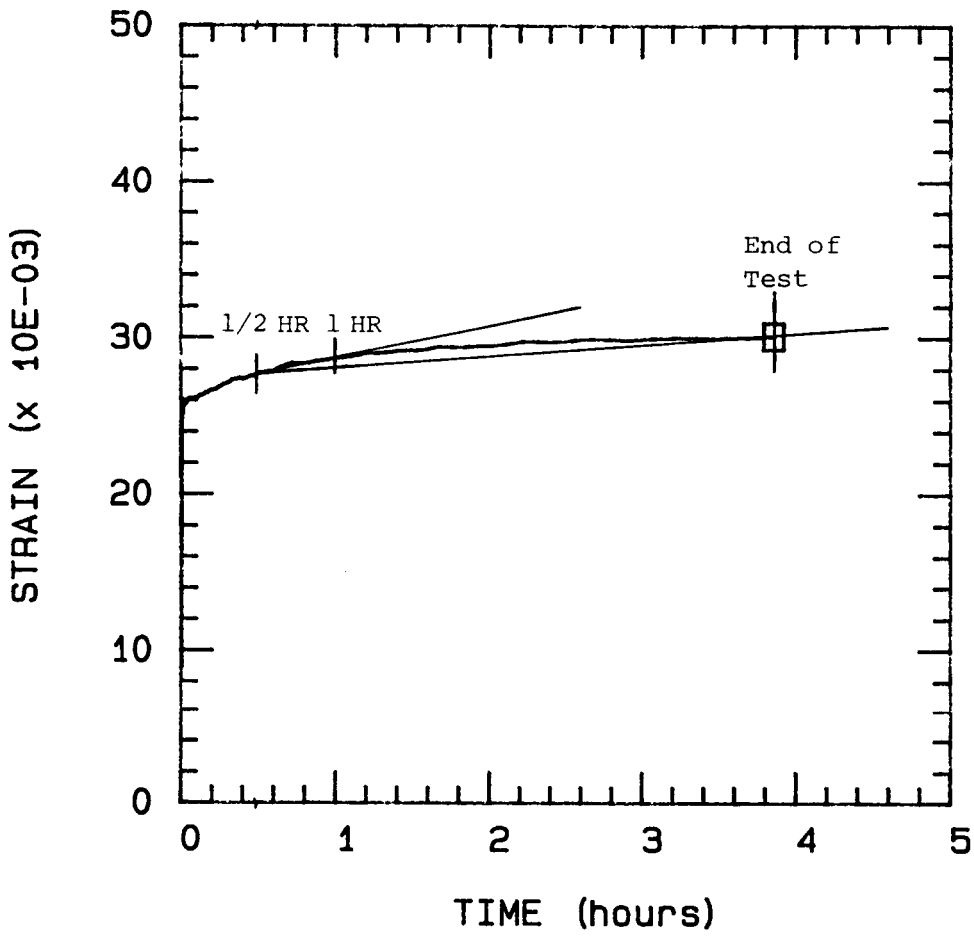


Figure 15. Typical Strain vs. Time Creep Plot Showing Two Creep Strain Rates.

## SECTION 5

### CONCLUSIONS

The five types of ceramic fibers tested exhibited reasonable static mechanical properties over a wide temperature range, each fiber being tested up to a maximum temperature consistent with its particular capabilities. Time spent at the higher elevated temperatures proved to be a parameter to consider due to some fiber degradation occurring during the creep testing. Creep testing was thus performed at slightly lower test temperatures than static tensile testing temperatures. The Nicalon NLM-102 silicon carbide fibers were tested over the highest temperature range, while the Nextel 312 glass fibers were tested over the lowest temperature range of all types of fibers. The Astroquartz 9288 glass fibers and Nextel 380 and 480 alumina/silica/boria fibers were tested over intermediate temperature ranges between the Nextel 312 and Nicalon NLM-102 fibers.

All five fiber types tested exhibited mechanical properties comparable with published literature values, as noted in the Summary of this report. These ceramic fibers are extremely brittle, which contributed to the scatter seen in some of the reported data. Strength properties were most affected, with high standard deviations being obtained. Variations in cross-sectional area of the fibers also contributed to the scatter seen in the data.

The high temperature test chambers designed and built for this program worked well, as did the temperature controllers. However, at least two more permanently mounted thermocouples will be added to the chambers in the heated zone, one near the top and one near the bottom,

to allow better monitoring of temperature gradients. Also, better materials will be sought for the chamber heating elements, which periodically failed due to the high temperatures used in this program.

The creep apparatus designed and built for this program worked very well. No major modifications are envisioned for this piece of equipment at the present time.

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Appendix A

Individual Single Fiber Static Tensile Test Data

STATIC TENSILE SPECIMEN NUMBER CODE

Eight digits total : " UNAVWXYZ "

U - Test Temperature

- R - 25°C
- 4 - 400°C
- 5 - 500°C
- 6 - 600°C
- 7 - 700°C
- 8 - 800°C
- 9 - 900°C
- 0 - 1000°C
- 1 - 1100°C
- 2 - 1200°C
- 3 - 1300°C

NA - Signifies NASA-Ames Program

V - Fiber Type

- 1 - Astroquartz 9288
- 2 - Nicalon NLM-102
- 6 - Nextel 312
- 8 - Nextel 380
- 9 - Nextel 480

WX - Gage Length

- 01 - 1"
- 02 - 2"
- 04 - 4"
- 08 - 8"
- 10 - 10"

YZ - Specimen Number

01 through 07

Example: "2NA20803"

V = 2 -- 1200°C Test Temperature  
NA = NA -- NASA-Ames Program  
U = 2 -- Nicalon NLM-102 Fiber  
WX = 08 -- 8" Gage Length  
YZ = 03 -- Specimen No. 3

TABLE A2

## INDIVIDUAL SINGLE FIBER ROOM TEMPERATURE

## STATIC TEST DATA FOR NICALON NLM-102

Specimen No.	Fiber Cross-Sectional Area ( $\mu\text{m}^2$ ) (10 <sup>-7</sup> in <sup>2</sup> )	Tensile Strength (GPa) (ksi)	Tensile Modulus (GPa) (Msi)	Ultimate Strain (percent)	
<u>1" Gage Length</u>					
RNA20101	150	2.32	2.37 344.0	212 30.7	1.07
2			3.16 458.0	240 34.8	1.33
3			0.00* 000.0*	000* 00.0*	0.00*
4			0.00* 000.0*	000* 00.0*	0.00*
5			2.32 335.8	214 31.0	1.09
6			0.00* 000.0*	000* 00.0*	0.00*
7			2.17 314.5	203 29.5	1.06
8			0.00* 000.0*	000* 00.0*	0.00*
9			1.99 289.2	253 36.7	1.07
10			<u>2.41 349.4</u>	<u>227 32.9</u>	<u>1.14</u>
Average			2.40 348.5	225 32.6	1.13
Std. Dev.			0.40 58.0	19 2.7	0.10
<u>2" Gage Length</u>					
RNA20201	150	2.32	0.00* 000.0*	000* 00.0*	0.00*
2			2.42 351.2	174 25.3	1.38
3			3.13 454.1	325 47.1	0.97
4			2.97 430.9	302 43.9	0.98
5			2.52 365.2	190 27.6	1.32
6			2.80 405.8	205 29.7	1.35
7			2.96 430.0	212 30.7	1.39
8			2.78 402.4	236 34.2	1.19
9			0.00* 000.0*	000* 00.0*	0.00*
10			<u>0.00* 000.0*</u>	<u>000* 00.0*</u>	<u>0.00*</u>
Average			2.80 405.7	235 34.1	1.23
Std. Dev.			0.25 36.9	57 8.3	0.18
<u>4" Gage Length</u>					
RNA20401	150	2.32	2.06 299.0	202 29.3	0.98
2			2.24 324.6	224 32.4	1.00
3			2.59 375.4	250 36.2	1.04
4			2.07 300.5	264 38.4	0.78
5			3.13 454.6	315 45.7	0.99
6			3.36 487.9	336 48.7	1.00
7			0.00* 000.0*	000* 00.0*	0.00*
8			1.64 238.2	250 36.3	0.65
9			3.07 445.9	308 44.7	1.00
10			<u>2.25 326.6</u>	<u>285 41.3</u>	<u>0.78</u>
Average			2.49 361.4	270 39.2	0.91
Std. Dev.			0.58 84.7	44 6.4	0.14

\* Numbers not used in calculating the Average or Standard Deviation



TABLE A4

## INDIVIDUAL SINGLE FIBER ROOM TEMPERATURE

## STATIC TEST DATA FOR NEXTEL 380

Specimen No.	Fiber Cross-Sectional Area ( $\mu\text{m}^2$ ) (10 <sup>-7</sup> in <sup>2</sup> )	Tensile Strength (GPa) (ksi)	Tensile Modulus (GPa) (Msi)	Ultimate Strain (percent)	
<u>1" Gage Length</u>					
RNA80101	85	1.32	1.92 278.4	129 18.7	1.49
2			1.12 162.2	116 16.8	0.98
3			1.00 144.6	79 11.5	1.26
4			1.38 199.5	111 16.1	1.22
5			1.80 261.8	130 18.8	1.38
6			1.28 185.0	122 17.7	1.05
7			0.00* 000.0*	000* 00.0*	0.00*
8			1.01 146.4	117 17.0	0.86
9			1.48 214.0	128 18.6	1.13
10			<u>1.52</u> <u>220.4</u>	<u>115</u> <u>16.6</u>	<u>1.31</u>
Average			1.39 201.4	116 16.9	1.19
Std. Dev.			0.33 47.6	16 2.2	0.20
<u>2" Gage Length</u>					
RNA80201	85	1.32	0.93 134.4	115 16.7	0.81
2			1.44 208.1	139 20.1	1.03
3			1.17 169.6	117 17.0	1.00
4			1.33 193.0	147 21.4	0.91
5			1.46 212.4	132 19.2	1.10
6			0.82 118.7	137 19.9	0.60
7			1.57 227.2	135 19.6	1.16
8			0.90 131.0	137 19.8	0.62
9			1.21 175.1	138 20.0	0.87
10			<u>1.13</u> <u>164.3</u>	<u>122</u> <u>17.6</u>	<u>0.93</u>
Average			1.20 173.4	132 19.1	0.90
Std. Dev.			0.26 37.1	10 1.5	0.19
<u>4" Gage Length</u>					
RNA80401	85	1.32	0.85 123.3	124 17.9	0.68
2			1.02 148.0	115 16.7	0.88
3			0.71 102.3	120 17.3	0.60
4			0.99 143.7	122 17.7	0.80
5			1.40 203.5	133 19.3	1.05
6			1.03 149.5	120 17.4	0.85
7			1.33 192.4	139 20.2	0.95
8			1.10 158.8	128 18.6	0.85
9			0.95 137.5	118 17.1	0.80
10			<u>0.00*</u> <u>000.0*</u>	<u>000*</u> <u>00.0*</u>	<u>0.00*</u>
Average			1.04 151.0	124 18.0	0.83
Std. Dev.			0.22 31.5	8 1.1	0.13

\* Numbers not used in calculating the Average or Standard Deviation

TABLE A6  
 INDIVIDUAL SINGLE FIBER HIGH TEMPERATURE  
 STATIC TEST DATA FOR ASTROQUARTZ 9288

Specimen No.	Fiber Cross- Sectional Area		Tensile Strength		Tensile Modulus		Ultimate Strain
	( $\mu\text{m}^2$ )	( $10^{-7}\text{in}^2$ )	(GPa)	(ksi)	(GPa)	(Msi)	(percent)
<u>500°C, 8" Gage Length</u>							
5NA10801	60	0.93	1.36	197	84.5	12.3	1.61
2			1.76	255	84.9	12.3	2.06
3			0.77	112	72.9	10.6	1.09
4			0.89	128	77.8	11.3	1.13
5			<u>1.13</u>	<u>163</u>	<u>80.5</u>	<u>11.7</u>	<u>1.61</u>
Average	60	0.93	1.18	171	80.1	11.5	1.50
Std. Dev.	0	0	0.39	57	5.0	0.7	0.40
<u>500°C, 10" Gage Length</u>							
5NA11001	60	0.93	1.64	237	84.2	12.2	1.93
2			1.33	193	90.0	13.0	1.45
3			1.27	184	84.4	12.2	1.50
4			1.58	229	82.6	12.0	1.83
5			<u>1.57</u>	<u>227</u>	<u>89.4</u>	<u>13.0</u>	<u>1.74</u>
Average	60	0.93	1.48	214	86.1	12.5	1.69
Std. Dev.	0	0	0.17	24	3.3	0.5	0.21
<u>600°C, 8" Gage Length</u>							
6NA10801	60	0.93	1.54	223	77.6	11.3	1.96
2			1.13	163	68.8	10.0	1.60
3			1.08	157	70.5	10.2	1.49
4			1.18	170	74.0	10.7	1.57
5			<u>0.76</u>	<u>110</u>	<u>70.4</u>	<u>10.2</u>	<u>1.07</u>
Average	60	0.93	1.14	165	72.3	10.5	1.54
Std. Dev.	0	0	0.28	40	3.5	0.5	0.32

TABLE A7

## INDIVIDUAL SINGLE FIBER HIGH TEMPERATURE

## STATIC TEST DATA FOR NICALON NLM-102

Specimen No.	Fiber Cross- Sectional Area		Tensile Strength		Tensile Modulus		Ultimate Strain
	( $\mu\text{m}^2$ )	( $10^{-7}\text{in}^2$ )	(GPa)	(ksi)	(GPa)	(Msi)	(percent)
<u>1000°C, 8" Gage Length</u>							
ONA20801	150	2.32	1.87	271	277	40.2	0.67
2			1.76	254	247	35.9	0.70
3			1.88	273	228	33.1	0.79
4			2.08	301	272	39.4	0.77
5			1.42	206	253	36.7	0.56
6			<u>1.80</u>	<u>261</u>	<u>223</u>	<u>32.3</u>	<u>0.80</u>
Average	150	2.32	1.80	261	250	36.3	0.72
Std. Dev	0	0	0.22	31	22	3.2	0.92
<u>1000°C, 10" Gage Length</u>							
ONA21001	150	2.32	1.51	219	201	29.2	0.74
2			1.42	206	221	32.1	0.63
3			0.99	144	207	30.0	0.48
4			1.84	267	263	38.1	0.70
5			1.76	254	235	34.1	0.73
6			<u>1.37</u>	<u>198</u>	<u>198</u>	<u>28.6</u>	<u>0.68</u>
Average	150	2.32	1.48	215	221	32.1	0.66
Std. Dev.	0	0	0.30	44	25	3.6	0.10
<u>1100°C, 8" Gage Length</u>							
1NA20801	150	2.32	1.76	255	214	31.0	0.82
2			1.34	194	172	24.9	0.79
3			2.42	351	256	37.1	0.95
4			1.54	223	212	30.8	0.73
5			<u>2.61</u>	<u>379</u>	<u>271</u>	<u>39.2</u>	<u>0.96</u>
Average	150	2.32	1.93	280	225	32.6	0.85
Std. Dev.	0	0	0.55	81	39	5.7	0.10

TABLE A8  
INDIVIDUAL SINGLE FIBER HIGH TEMPERATURE  
STATIC TEST DATA FOR NEXTEL 312

Specimen No.	Fiber Cross-Sectional Area		Tensile Strength		Tensile Modulus		Ultimate Strain (percent)
	( $\mu\text{m}^2$ )	( $10^{-7}\text{in}^2$ )	(GPa)	(ksi)	(GPa)	(Msi)	
<u>400°C, 8" Gage Length</u>							
4NA60801	77	1.19	1.00	145	139	20.2	0.72
2			1.00	146	137	19.9	0.73
3			0.96	139	136	19.7	0.70
4			0.98	142	149	21.6	0.65
5			<u>0.86</u>	<u>124</u>	<u>144</u>	<u>20.9</u>	<u>0.59</u>
Average	77	1.19	0.96	139	141	20.5	0.68
Std. Dev.	0	0	0.06	9	5	0.8	0.06
<u>500°C, 8" Gage Length</u>							
5NA60801	77	1.19	0.77	112	130	18.8	0.59
2			0.81	117	138	20.1	0.58
3			0.86	124	129	18.6	0.70
4			0.84	122	122	17.7	0.70
5			0.88	128	126	18.2	0.69
6			<u>1.15</u>	<u>167</u>	<u>138</u>	<u>20.0</u>	<u>0.84</u>
Average	77	1.19	0.88	128	130	18.9	0.69
Std. Dev.	0	0	0.14	20	6	1.0	0.09
<u>500°C, 10" Gage Length</u>							
5NA61001	77	1.19	1.16	168	183	26.5	0.62
2			0.63	92	124	18.0	0.50
3			0.88	128	133	19.4	0.66
4			0.98	141	132	19.1	0.72
5			<u>0.75</u>	<u>109</u>	<u>118</u>	<u>17.1</u>	<u>0.63</u>
Average	77	1.19	0.88	128	138	20.0	0.63
Std. Dev.	0	0	0.20	29	26	3.7	0.08

TABLE A9  
 INDIVIDUAL SINGLE FIBER HIGH TEMPERATURE  
 STATIC TEST DATA FOR NEXTEL 380

Specimen No.	Fiber Cross- Sectional Area		Tensile Strength		Tensile Modulus		Ultimate Strain
	( $\mu\text{m}^2$ )	( $10^{-7}\text{in}^2$ )	(GPa)	(ksi)	(GPa)	(Msi)	(percent)
<u>600°C, 8" Gage Length</u>							
6NA80801	85	1.32	1.28	186	147	21.4	0.87
2			1.41	205	167	24.2	0.85
3			1.39	201	148	21.5	0.94
4			0.80	116	141	20.5	0.58
5			<u>0.99</u>	<u>143</u>	<u>140</u>	<u>20.4</u>	<u>0.71</u>
Average	85	1.32	1.17	170	149	21.6	0.79
Std. Dev.	0	0	0.27	39	11	1.5	0.14
<u>600°C, 10" Gage Length</u>							
6NA81001	85	1.32	1.17	170	137	19.9	0.85
2			1.32	192	147	21.3	0.93
3			1.27	184	149	21.6	0.85
4			1.24	180	143	20.7	0.87
5			<u>1.18</u>	<u>171</u>	<u>130</u>	<u>18.8</u>	<u>0.88</u>
Average	85	1.32	1.24	179	141	20.5	0.88
Std. Dev.	0	0	0.06	9	8	1.1	0.03
<u>700°C, 8" Gage Length</u>							
7NA80801	85	1.32	1.55	225	163	23.6	0.96
2			1.02	148	131	19.0	0.79
3			1.38	200	134	19.4	1.01
4			1.22	177	125	18.1	0.95
5			<u>0.92</u>	<u>134</u>	<u>120</u>	<u>17.4</u>	<u>0.75</u>
Average	85	1.32	1.22	177	135	19.5	0.89
Std. Dev.	0	0	0.26	37	17	2.4	0.11

TABLE A10  
INDIVIDUAL SINGLE FIBER HIGH TEMPERATURE  
STATIC TEST DATA FOR NEXTEL 480

Specimen No.	Fiber Cross-Sectional Area		Tensile Strength		Tensile Modulus		Ultimate Strain
	( $\mu\text{m}^2$ )	( $10^{-7}\text{in}^2$ )	(GPa)	(ksi)	(GPa)	(Msi)	(percent)
<u>900°C, 8" Gage Length</u>							
9NA90801	65	1.01	1.56	227	203	29.4	0.78
2			1.45	211	251	36.4	0.57
3			1.72	249	237	34.4	0.72
4			1.36	198	196	28.4	0.68
5			<u>1.67</u>	<u>242</u>	<u>204</u>	<u>29.6</u>	<u>0.81</u>
Average	65	1.01	1.55	225	218	31.6	0.71
Std. Dev.	0	0	0.15	21	24	3.5	0.09
<u>1000°C, 8" Gage Length</u>							
ONA90801	65	1.01	0.68	98	170	24.6	0.40
2			1.68	244	273	39.5	0.62
3			1.72	250	263	38.1	0.65
4			1.41	204	193	28.0	0.73
5			<u>1.46</u>	<u>212</u>	<u>195</u>	<u>28.3</u>	<u>0.75</u>
Average	65	1.01	1.39	202	219	31.7	0.63
Std. Dev.	0	0	0.42	61	46	6.7	0.14
<u>1000°C, 10" Gage Length</u>							
ONA91001	65	1.01	1.09	158	214	31.0	0.50
2			0.77	112	241	34.9	0.32
3			1.37	199	229	33.2	0.60
4			1.05	152	210	30.5	0.50
5			1.14	165	182	26.5	0.62
6			<u>0.76</u>	<u>110</u>	<u>200</u>	<u>29.0</u>	<u>0.38</u>
Average	65	1.01	1.03	149	213	30.9	0.49
Std. Dev.	0	0	0.23	61	21	3.0	0.12

Appendix B

Individual Single Fiber Tensile Creep Test Data

## TENSILE CREEP SPECIMEN NUMBER CODE

All creep specimens begin with a "DT".

Four digits follow which identify the fiber type, test temperature, and specimen number, viz., "DTWXYZ" where:

### W - Fiber Type

- 1 - Astroquartz 9288
- 2 - Nicalon NLM-102
- 6 - Nextel 312
- 8 - Nextel 380
- 9 - Nextel 480

### X - Temperature Code

- 4 - 400°C
- 5 - 500°C
- 6 - 600°C
- 7 - 700°C
- 8 - 800°C
- 9 - 900°C
- 0 - 1000°C
- 1 - 1100°C

### YZ - Specimen Number

01 through 18

---

Example: DT2912

Explanation:

- "DT" - Creep Specimen
- "2" - Fiber Type: Nicalon NLM-102
- "9" - Test Temperature: 900°C
- "12" - Specimen No.: 12



Table B2

## INDIVIDUAL SINGLE FIBER HIGH TEMPERATURE

## TENSILE CREEP DATA FOR NICALON NLM-102

Specimen No.	Test Temperature  (°C)	Creep Rate From $\frac{1}{2}$ Hour to 1 Hour	Creep Rate From $\frac{1}{2}$ Hour to End of Test
		( $10^{-6}$ /Hour)	( $10^{-6}$ /Hour)
DT2806	800	-432*	-91*
DT2809		10	30
DT2811		<u>177</u>	<u>27</u>
Average		94	29
Std. Dev.		118	2
DT2911	900	601	320
DT2912		537	86
DT2913		<u>243</u>	<u>12</u>
Average		461	139
Std. Dev.		191	160
DT2013	1000	498	230
DT2015		708	247
DT2016		<u>437</u>	<u>287</u>
Average		548	255
Std. Dev.		142	29
DT2108	1100	1090	500
DT2113		357	121
DT2116		<u>1220</u>	<u>736</u>
Average		890	452
Std. Dev.		466	310

\* Numbers not used in calculating the Average or Standard Deviation.

Table B4

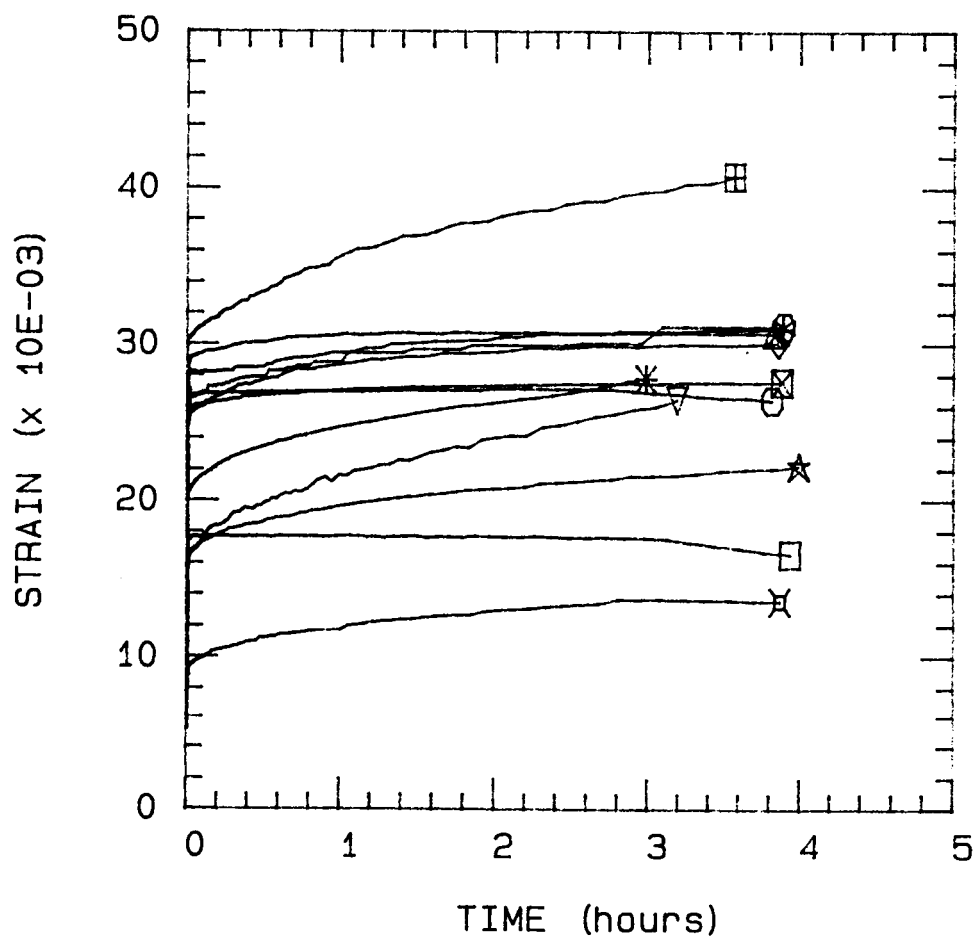
## INDIVIDUAL SINGLE FIBER HIGH TEMPERATURE

## TENSILE CREEP DATA FOR NEXTEL 380

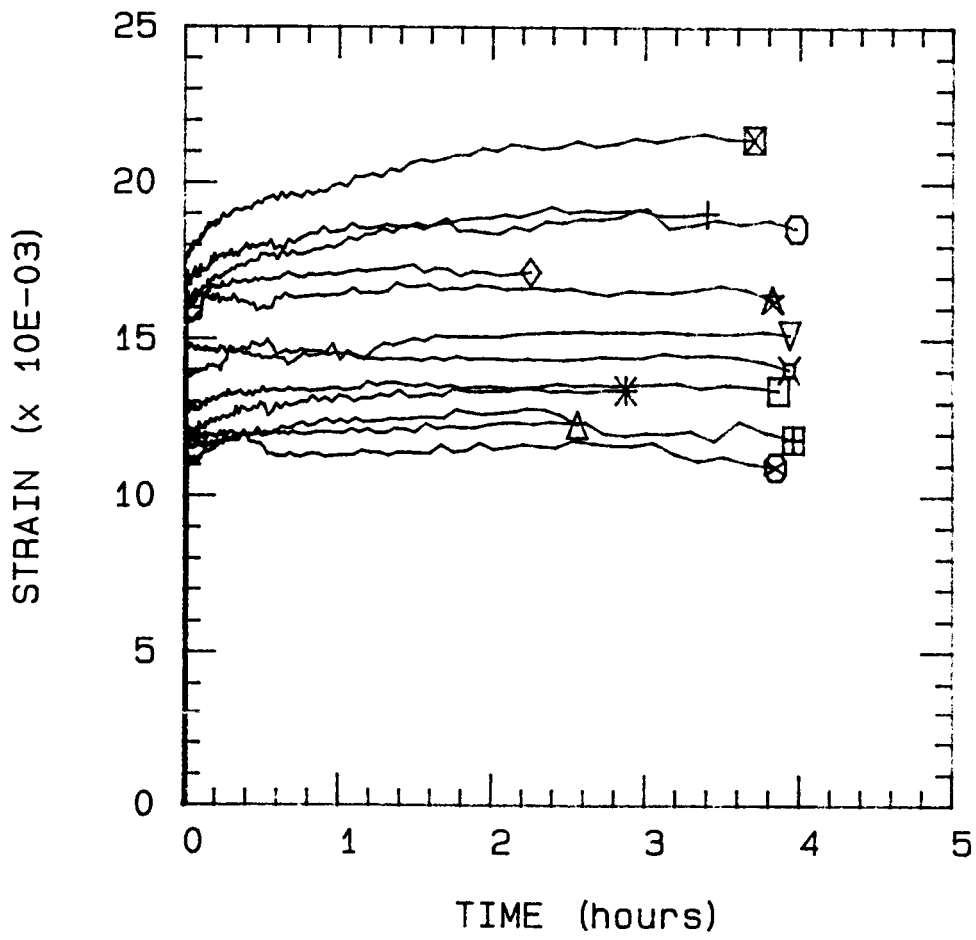
Specimen No.	Test Temperature	Creep Rate From $\frac{1}{2}$ Hour to 1 Hour	Creep Rate From $\frac{1}{2}$ Hour to End of Test
	(°C)	( $10^{-6}$ /Hour)	( $10^{-6}$ /Hour)
DT8501	500	196	9
DT8502		295	-34*
DT8503		416	-35*
Average		302	9
Std. Dev.		110	0
DT8601	600	276	222
DT8602		436	196
DT8606		294	112
Average		335	177
Std. Dev		88	58
DT8701	700	732	392
DT8702		760	343
DT8703		517	300
Average		669	345
Std. Dev.		133	46
DT8803	800	1840	1350
DT8805		2160	2220
DT8809		1460	887
Average		1820	1490
Std. Dev.		350	678

\* Numbers not used in calculating the Average or Standard Deviation.

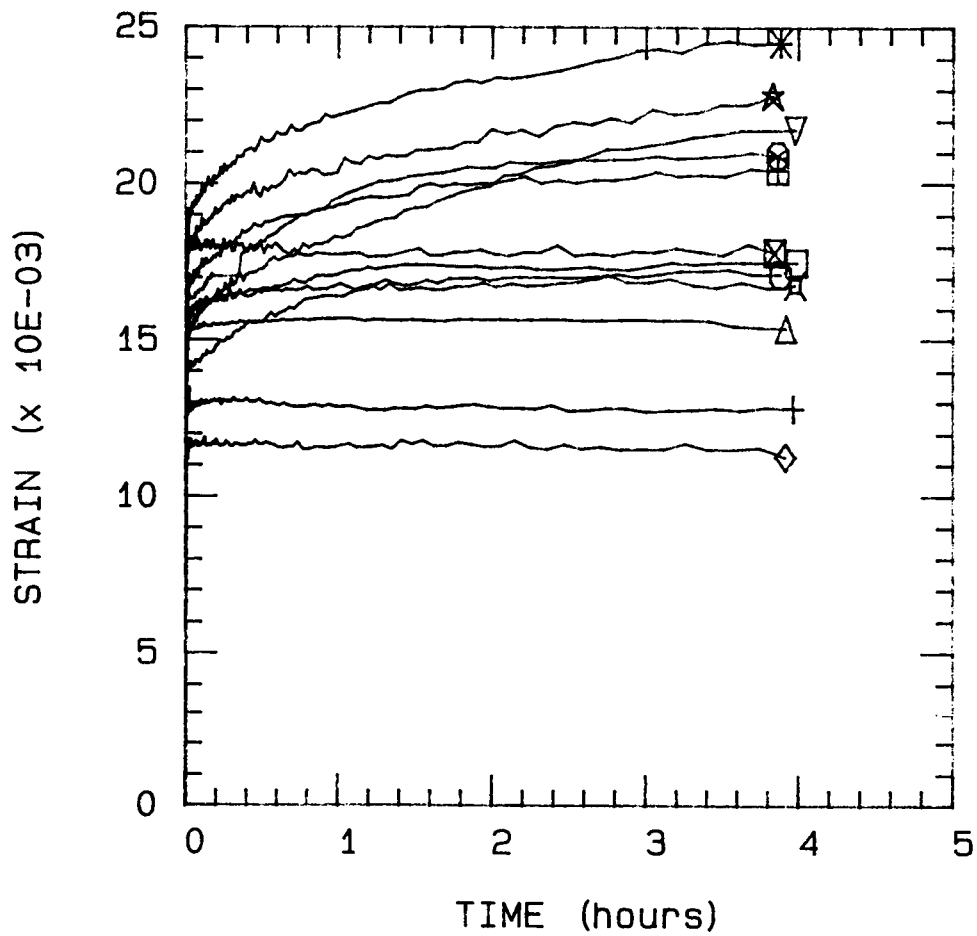
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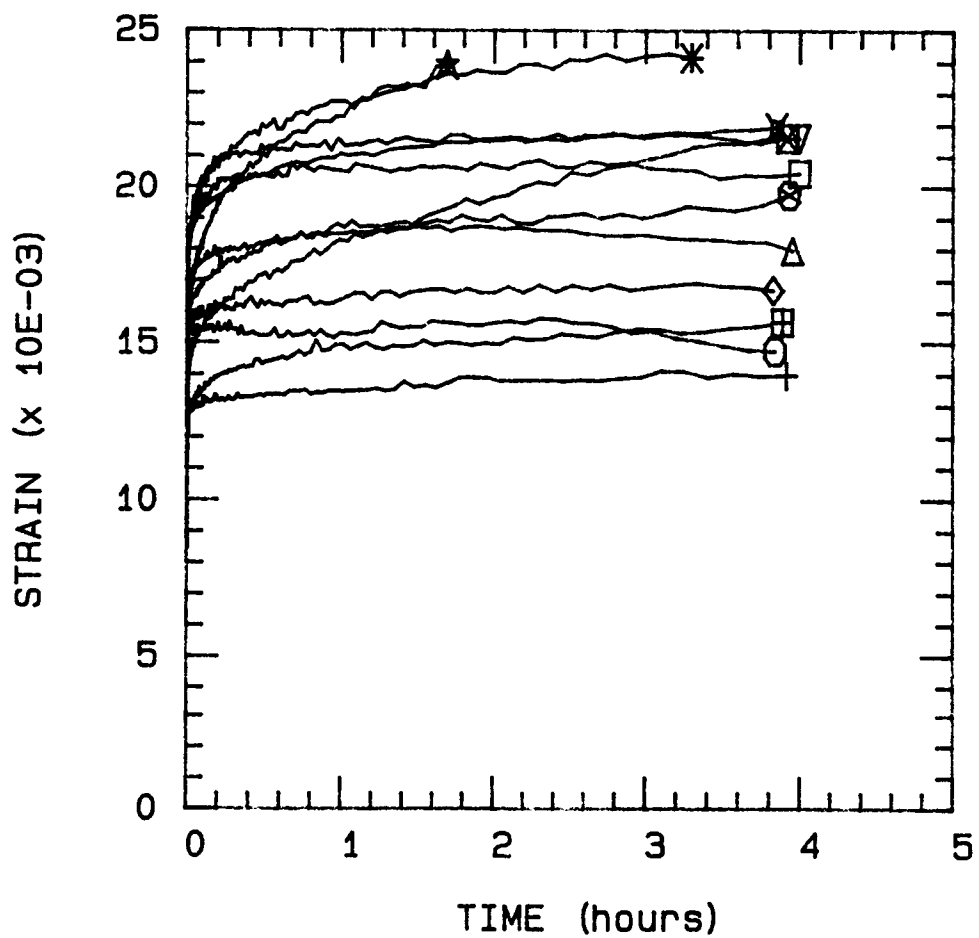
# NICALON NLM-102



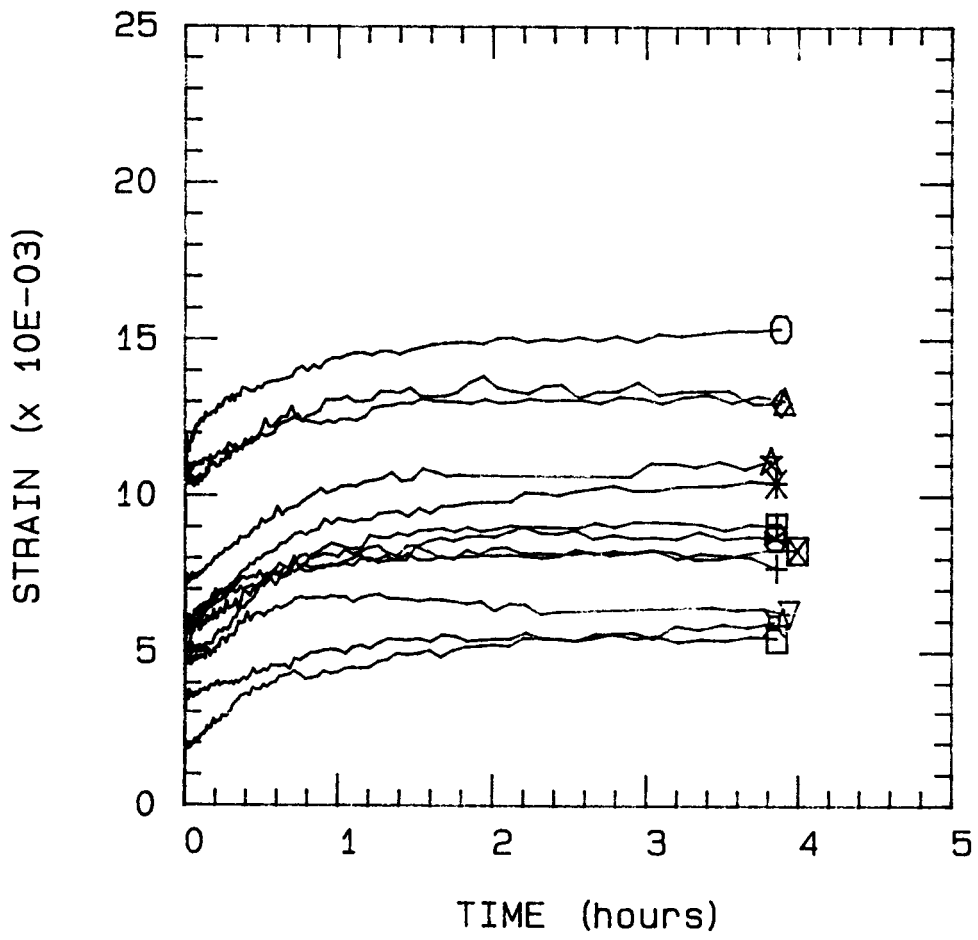
# NEXTEL 312



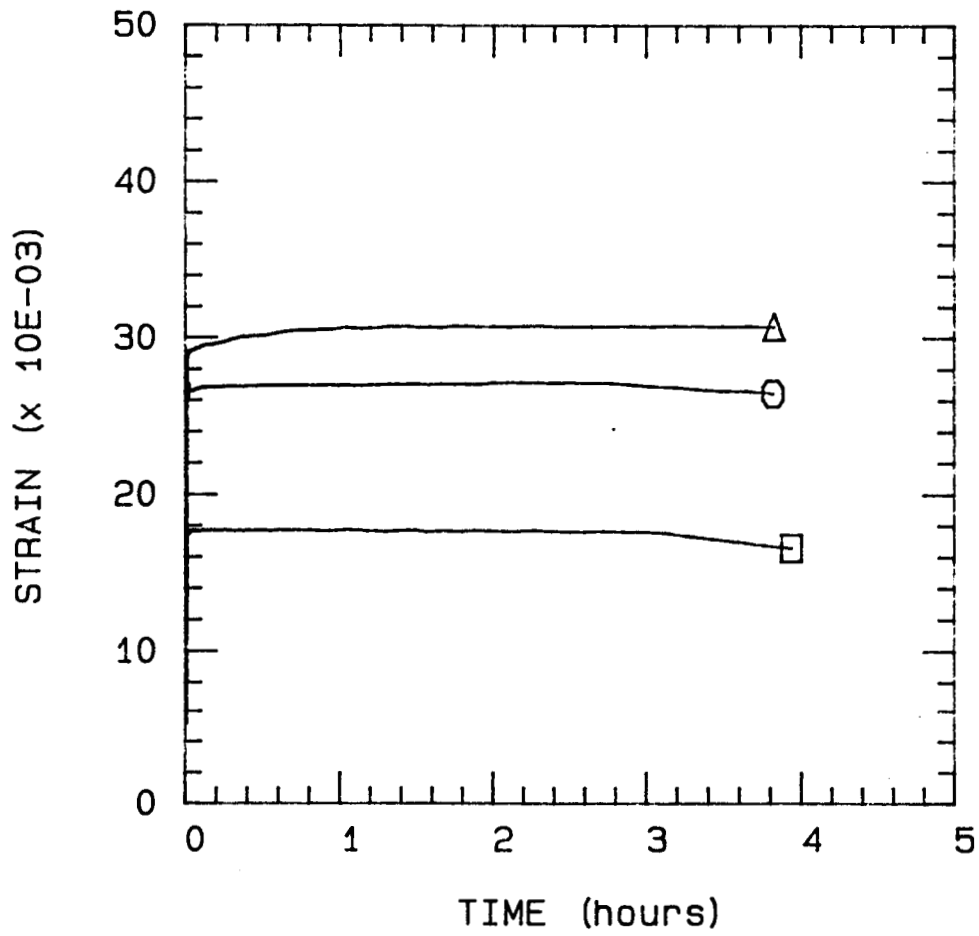
# NEXTEL 380



# NEXTEL 480

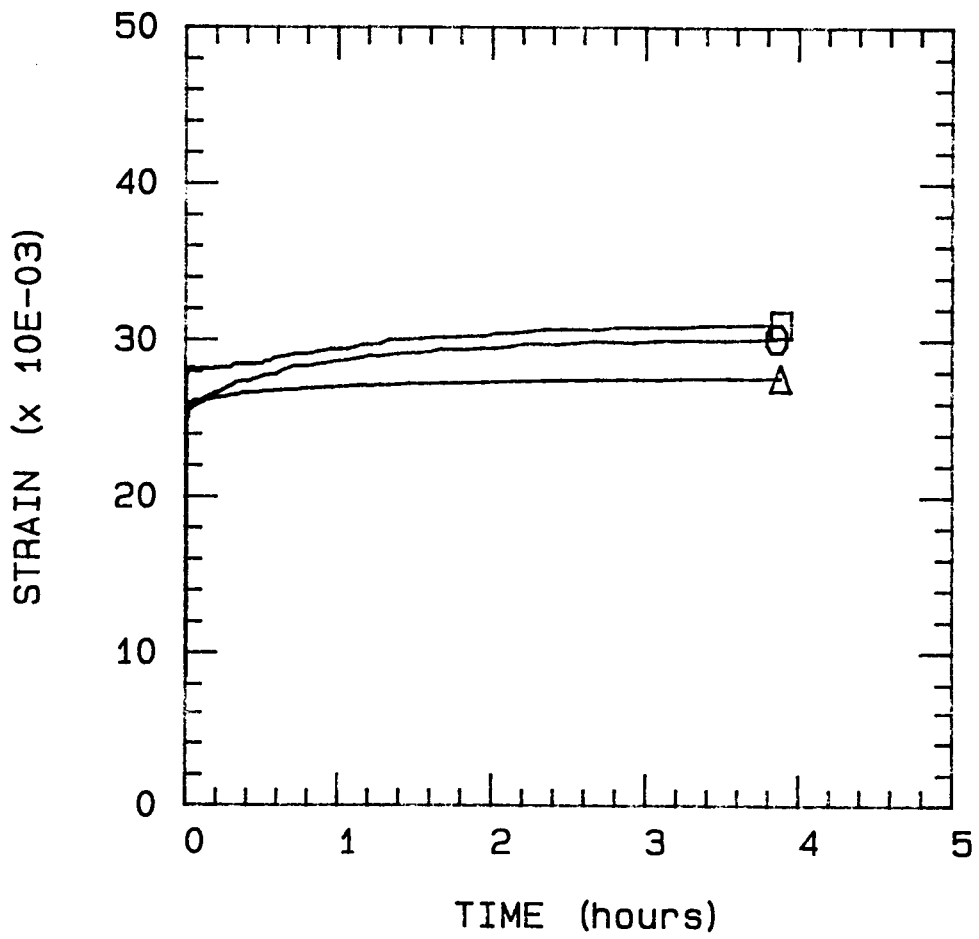


ASTROQUARTZ 9288 400 DEG C

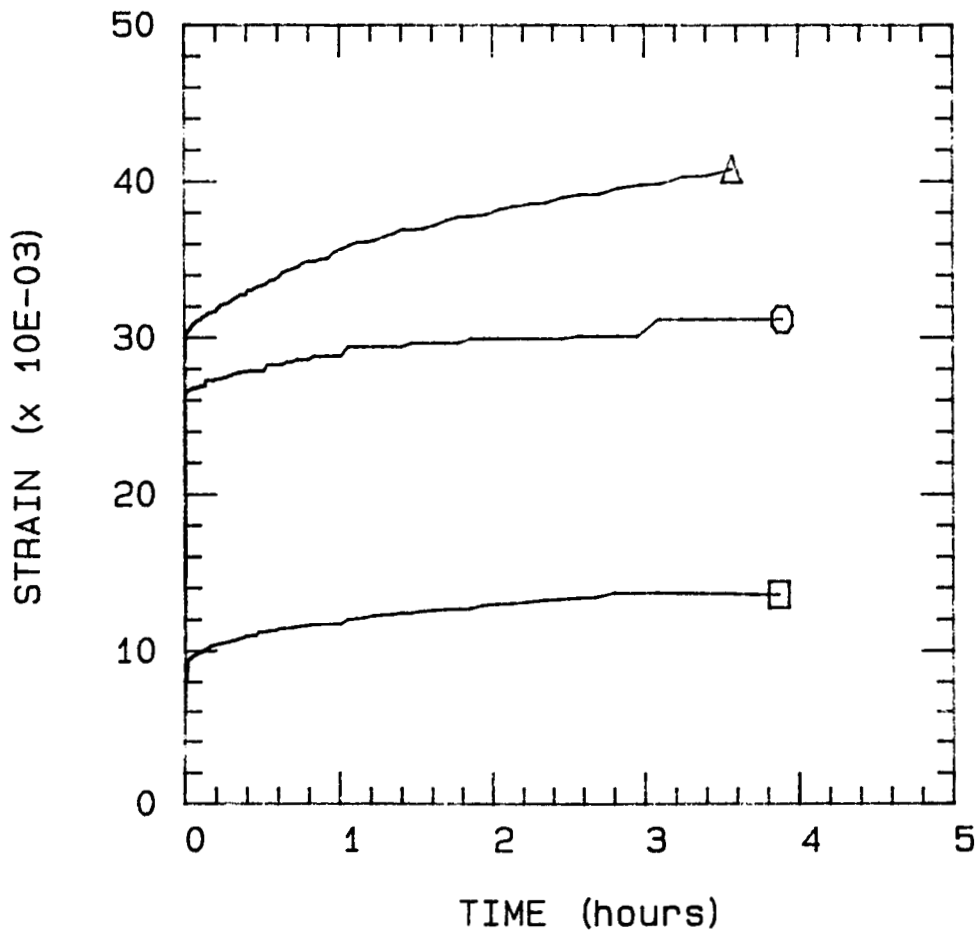




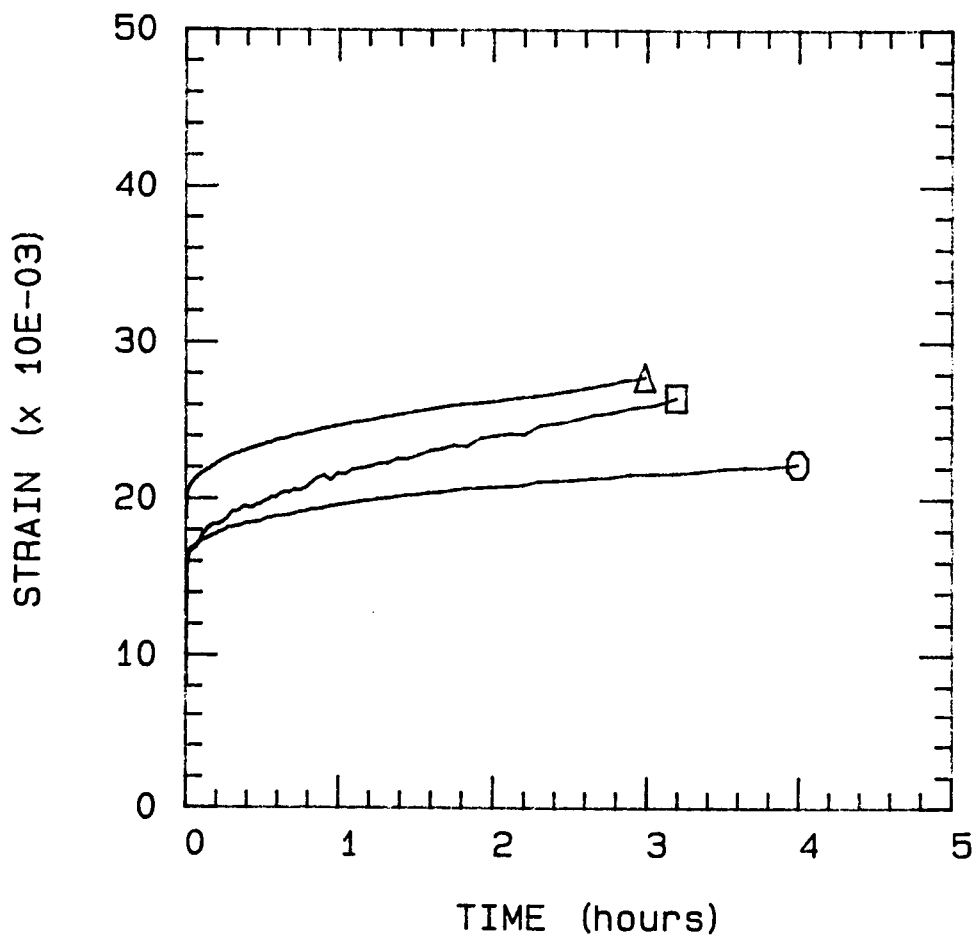
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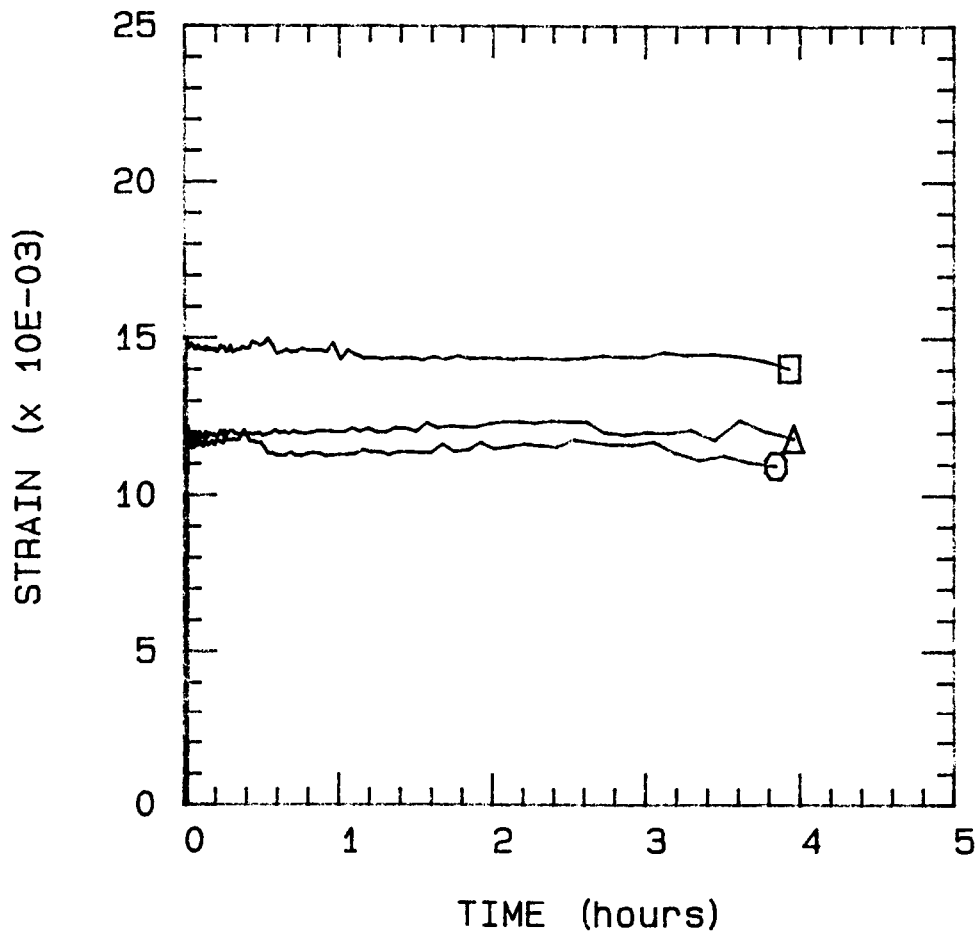
ASTROQUARTZ 9288 600 DEG C



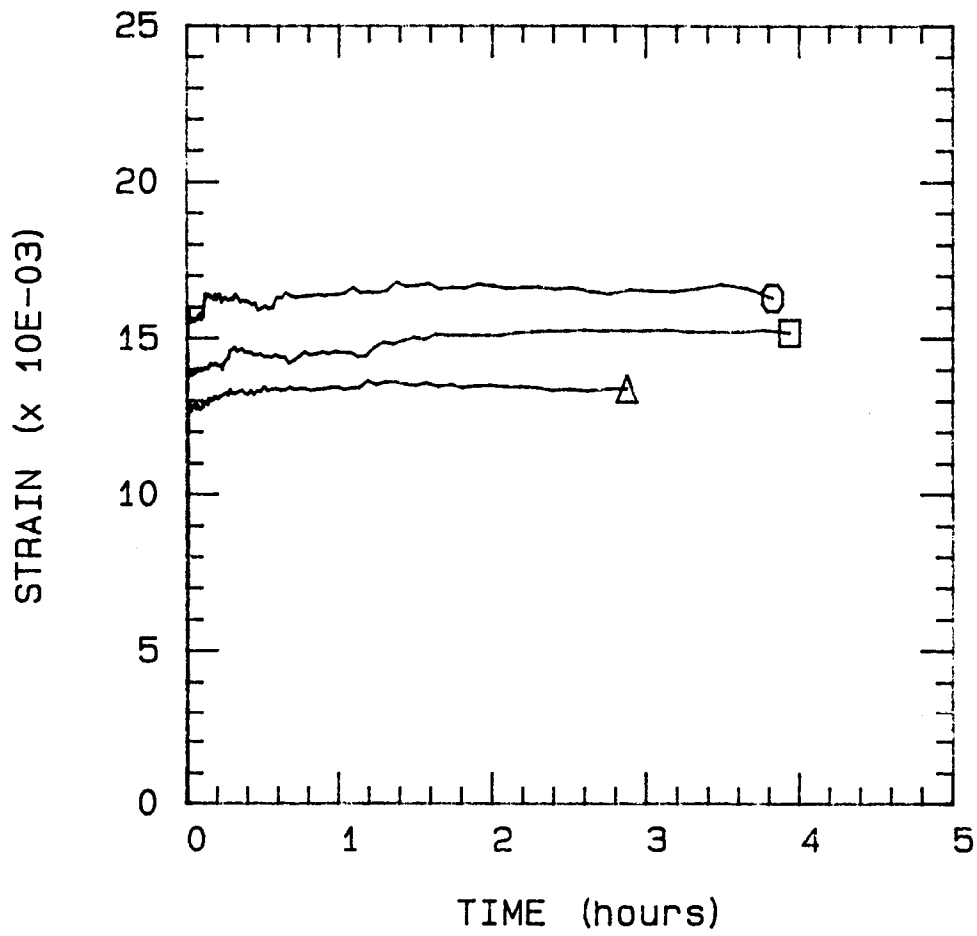
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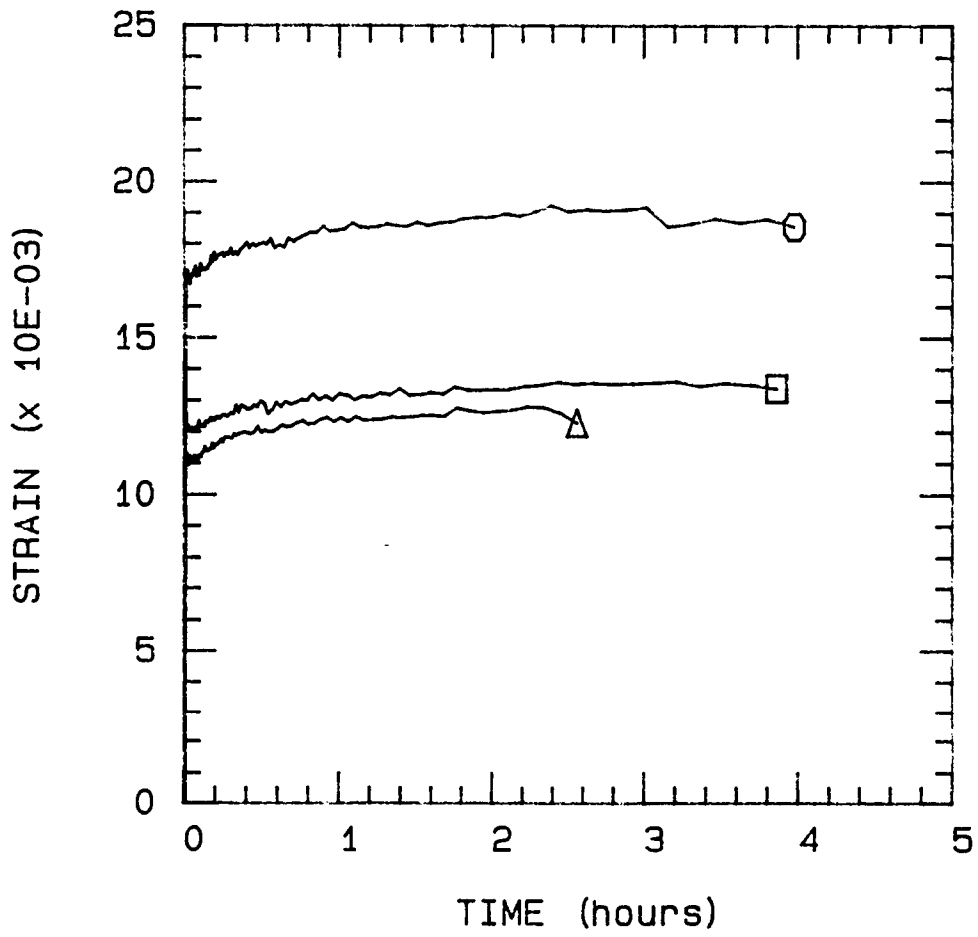
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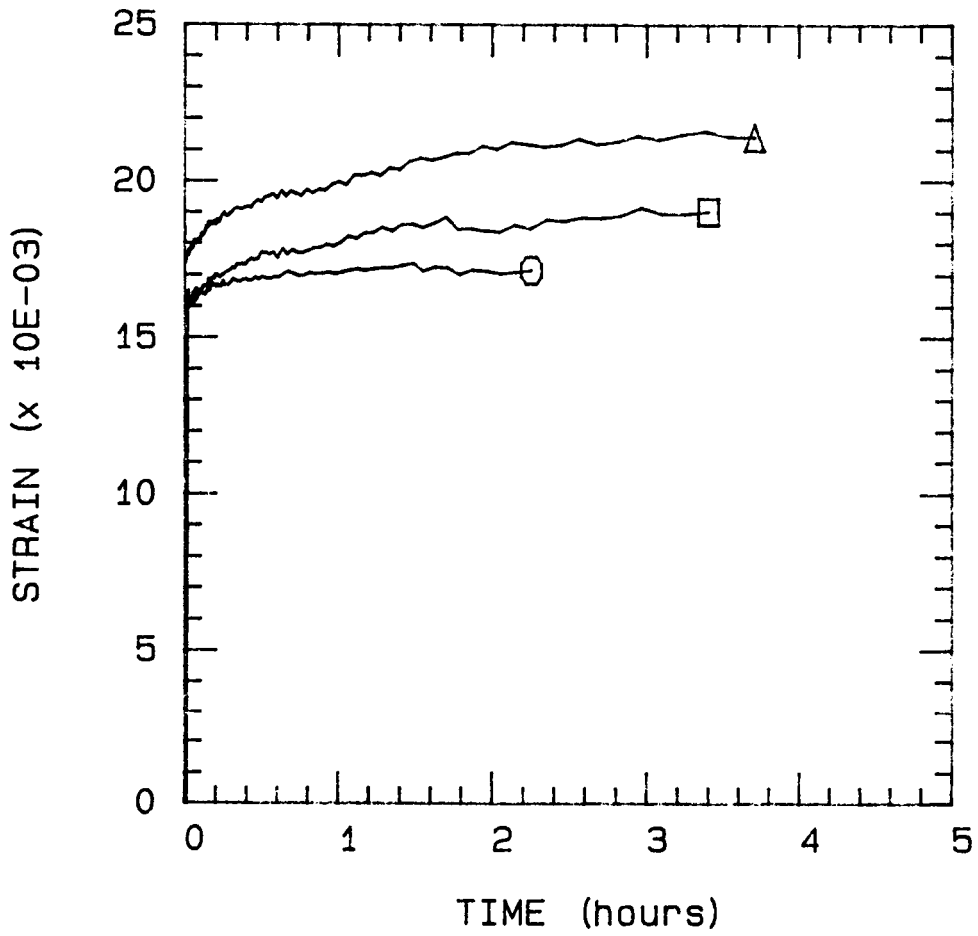
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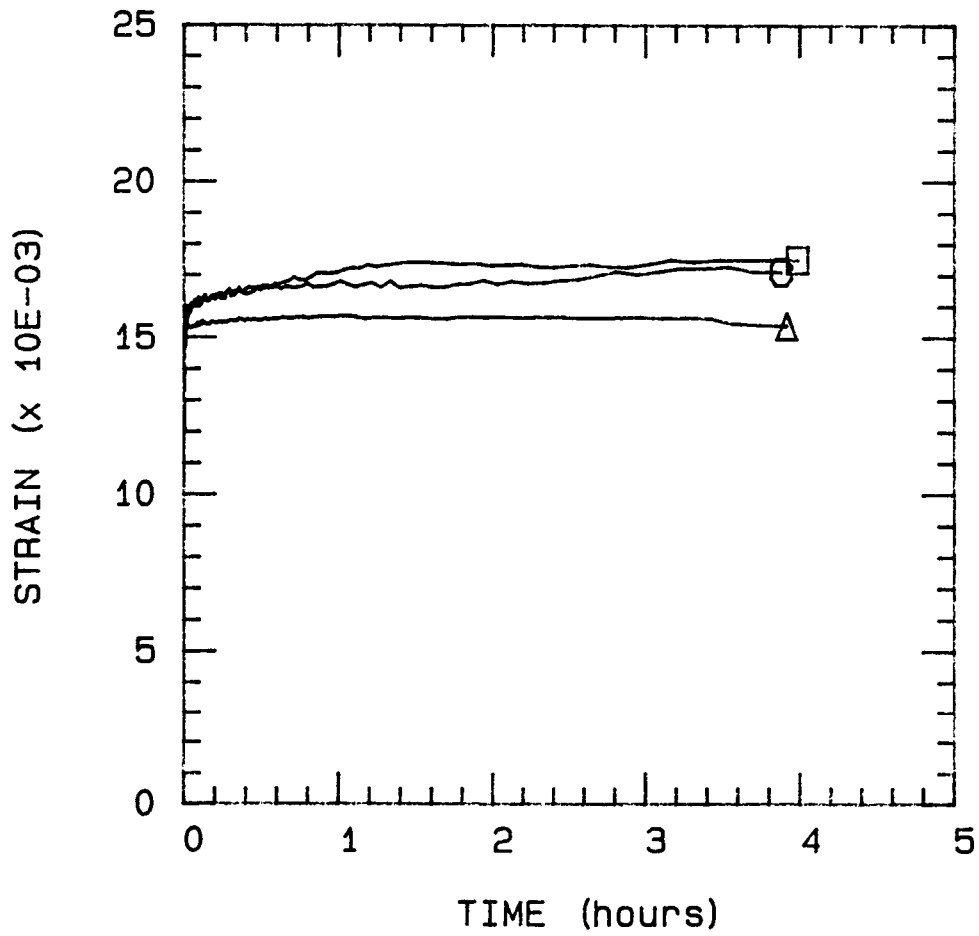
NICALON NLM-102 900 DEG C



NICALON NLM-102 1000 DEG C

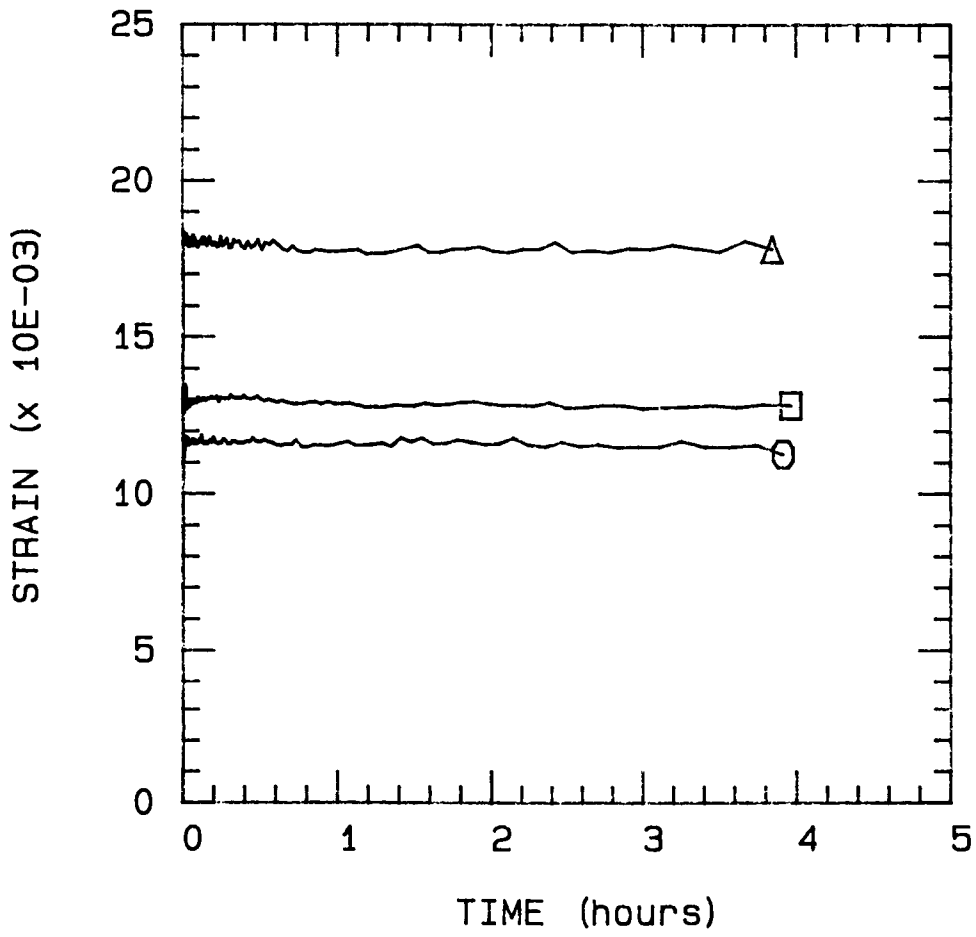


NEXTEL 312 400 DEG C



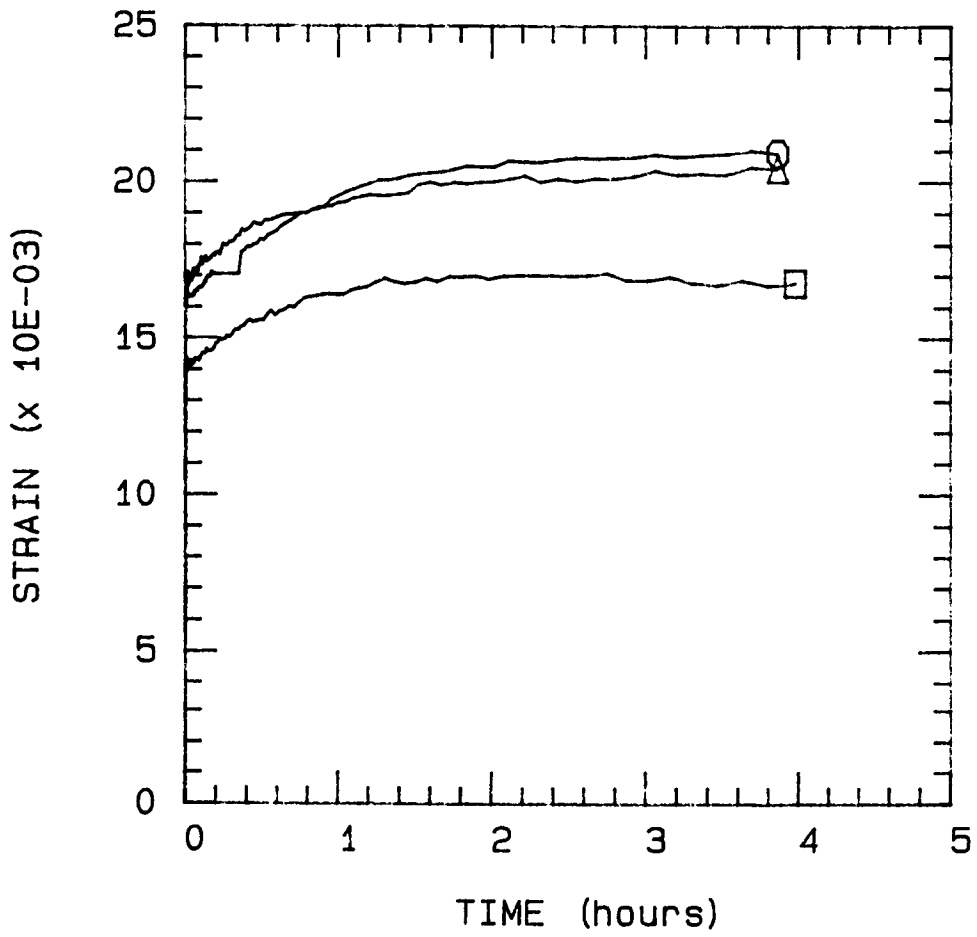


NEXTEL 312 500 DEG C

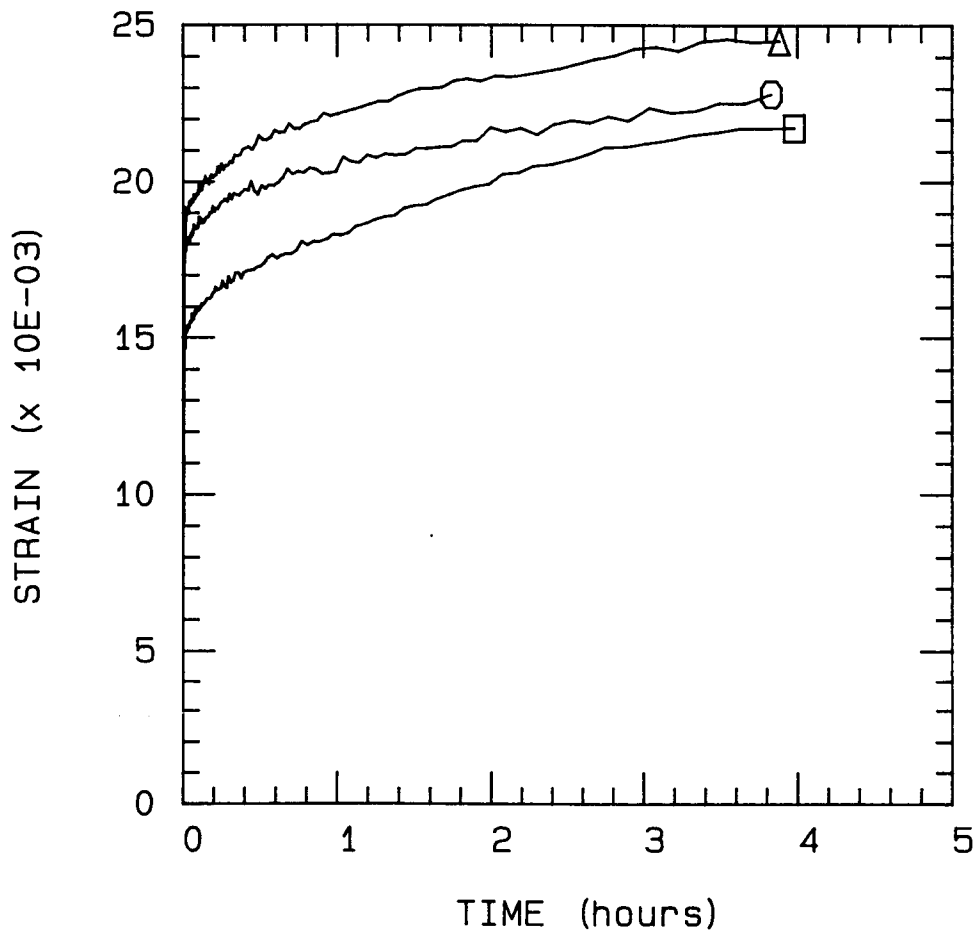


C-2

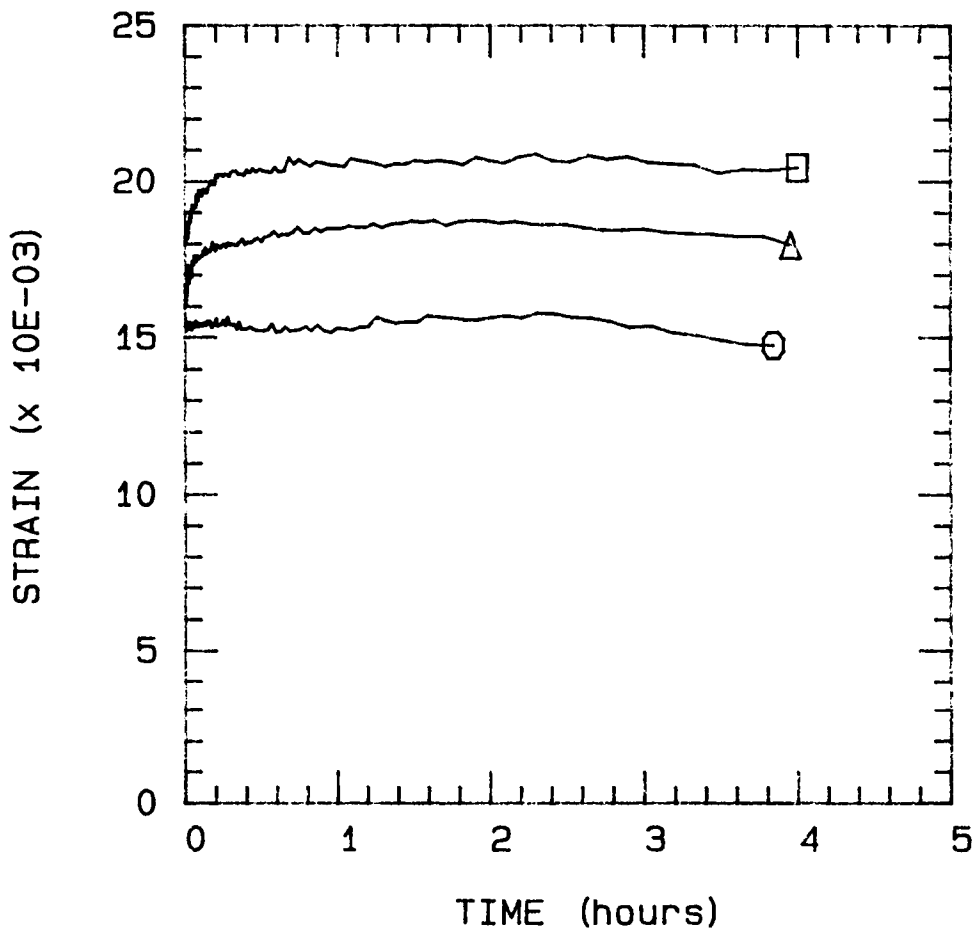
NEXTEL 312 600 DEG C



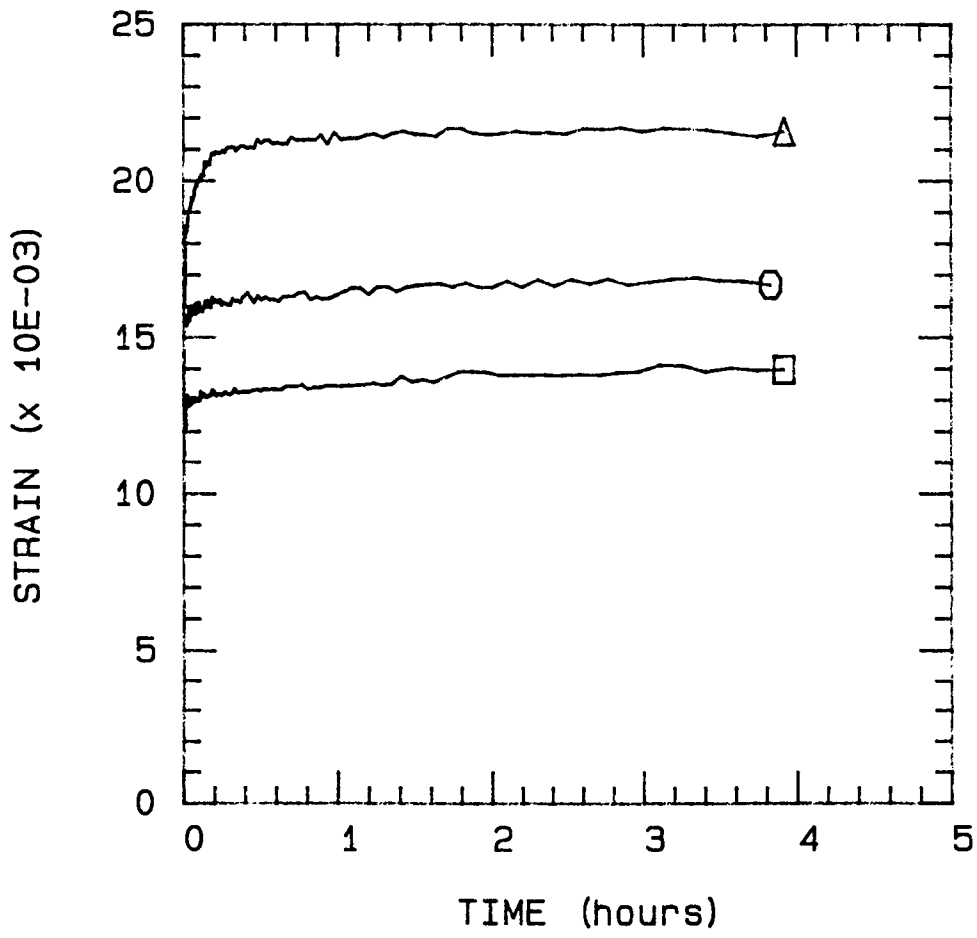
NEXTEL 312 700 DEG C



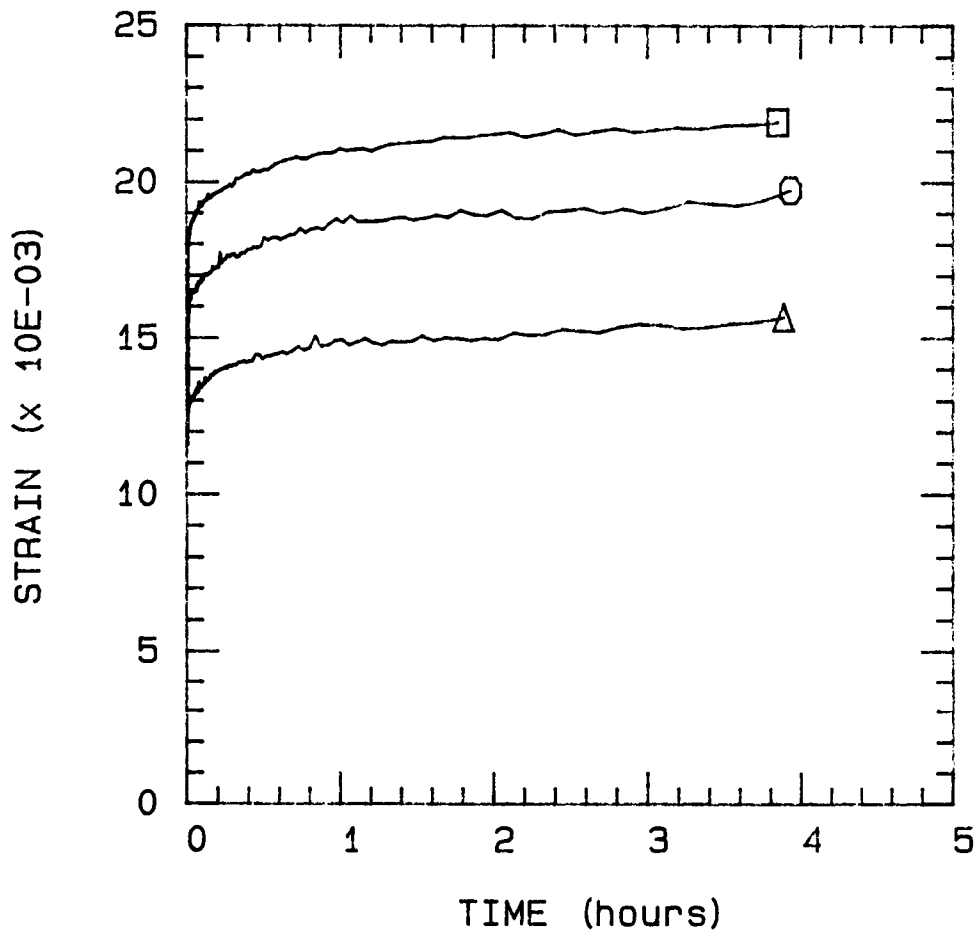
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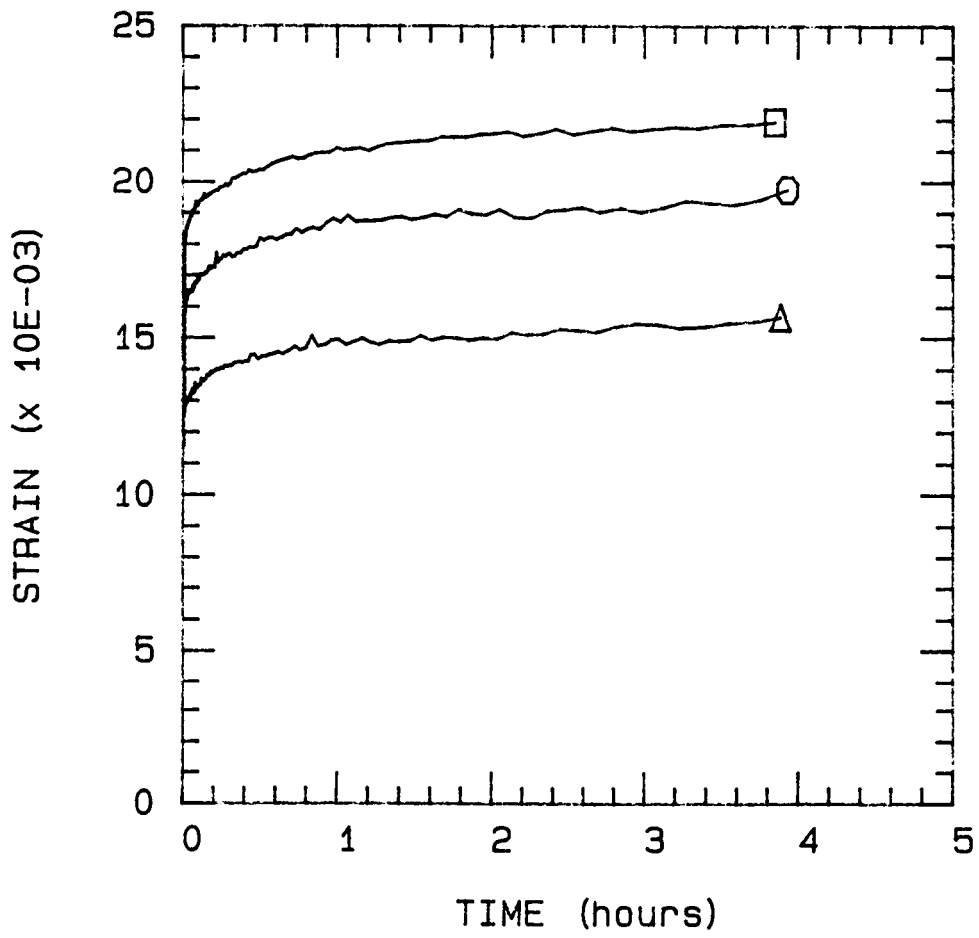
NEXTEL 380 600 DEG C



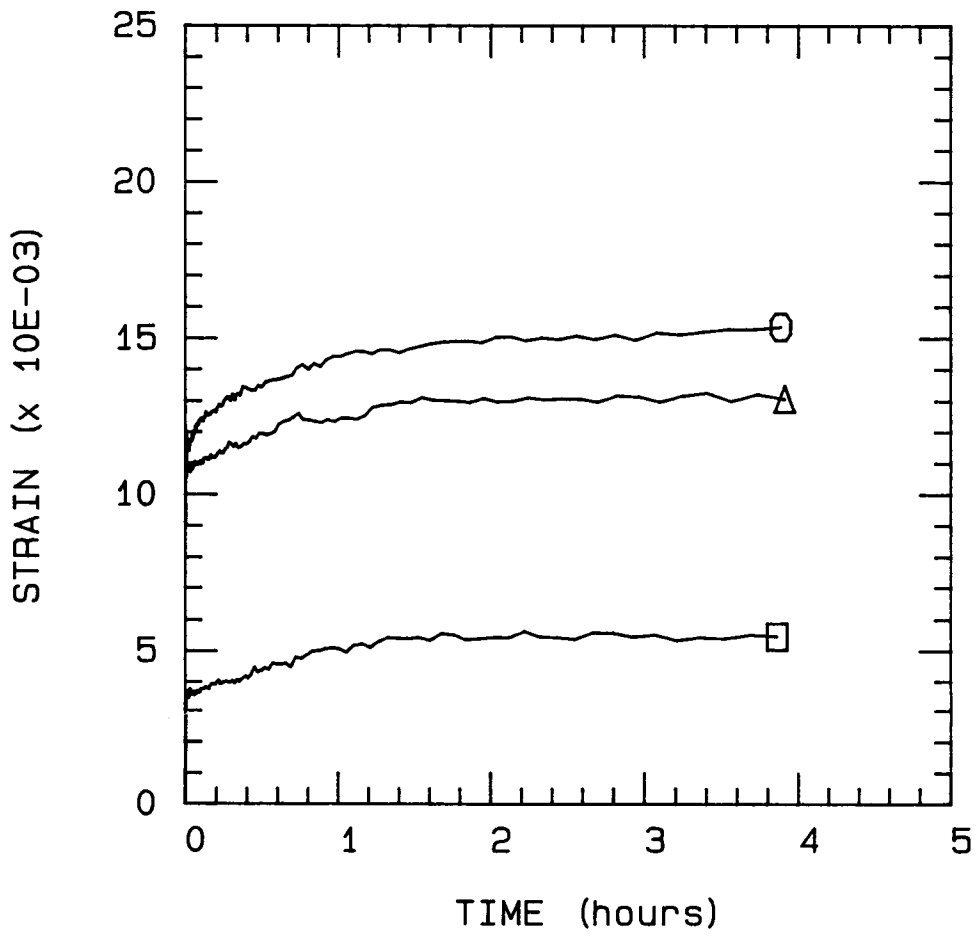
NEXTEL 380 700 DEG C



NEXTEL 380 800 DEG C

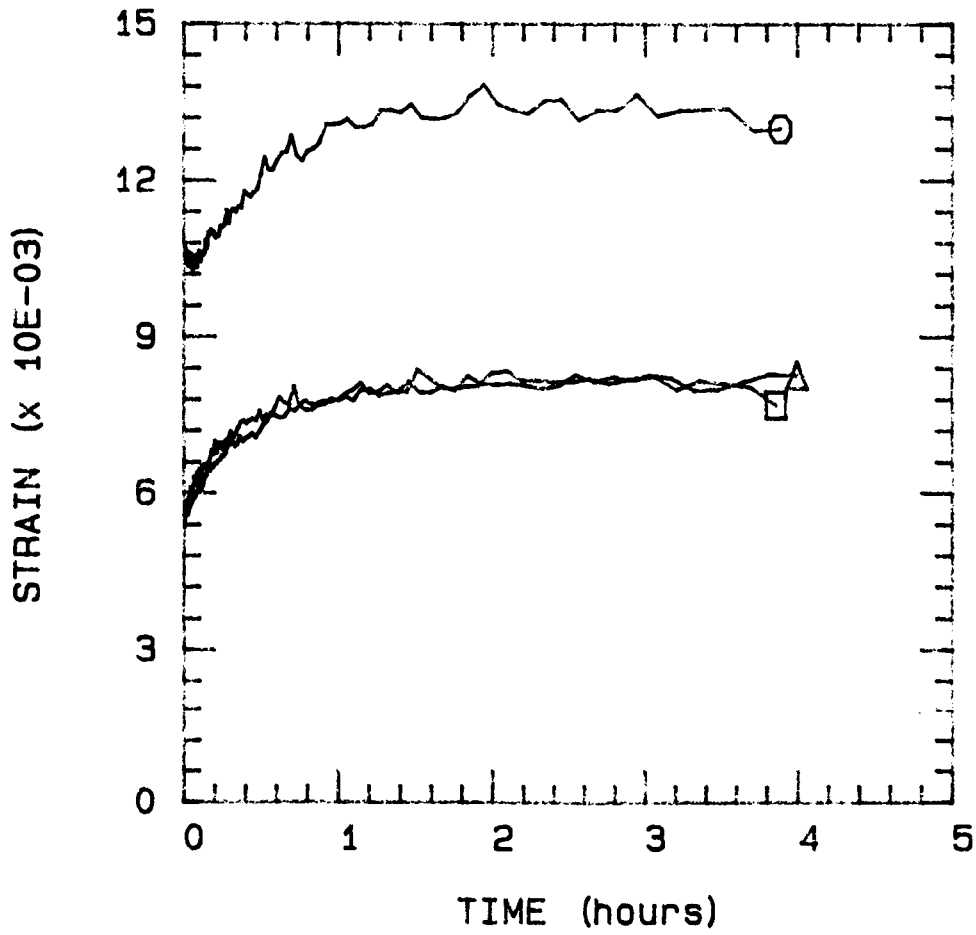


NEXTEL 480 700 DEG C

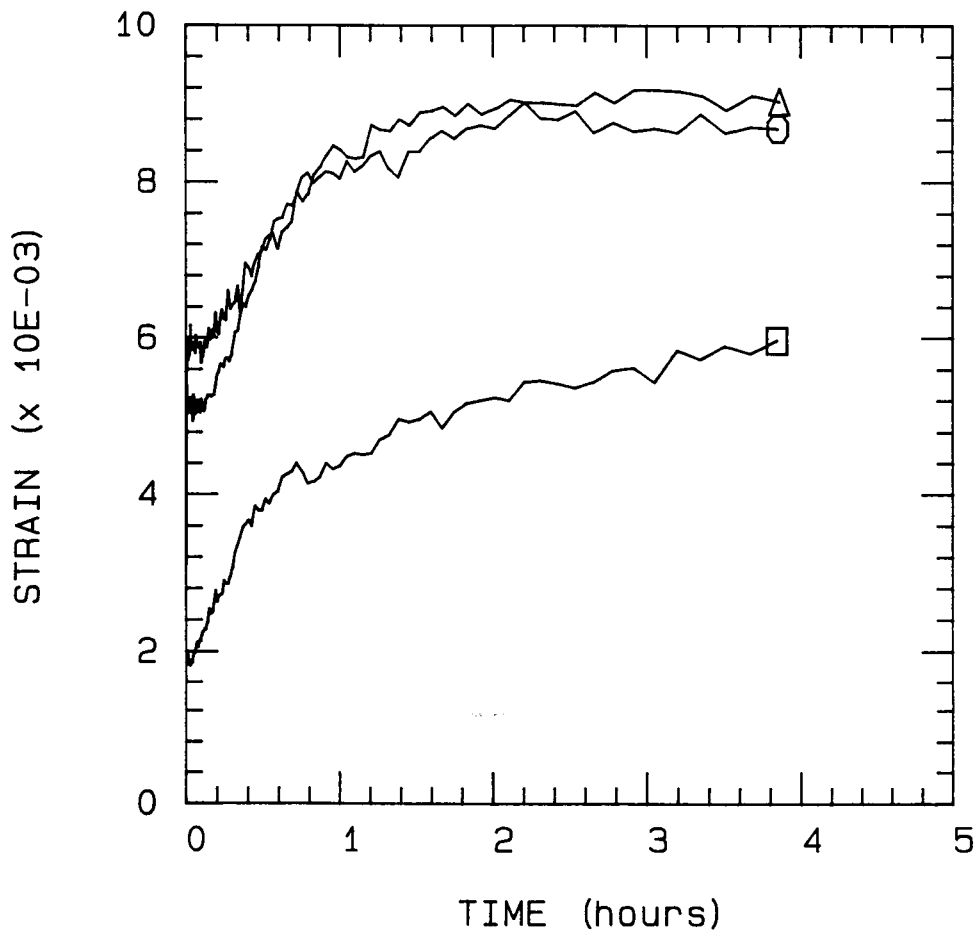




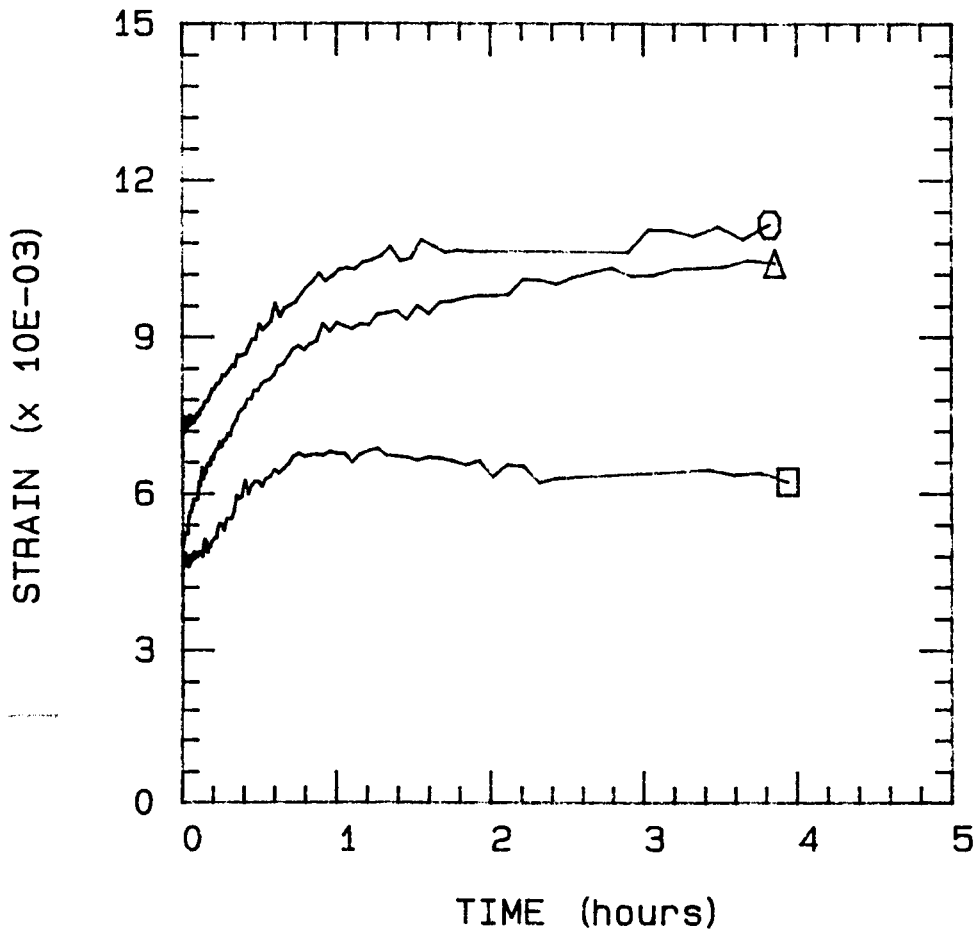
NEXTEL 480 800 DEG C



NEXTEL 480 900 DEG C



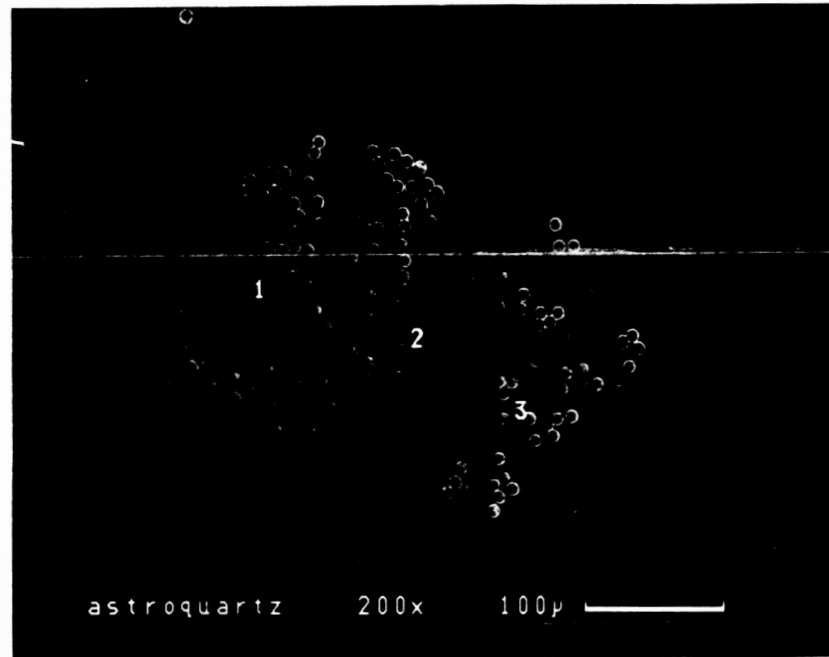
NEXTEL 480 1000 DEG C



Appendix C

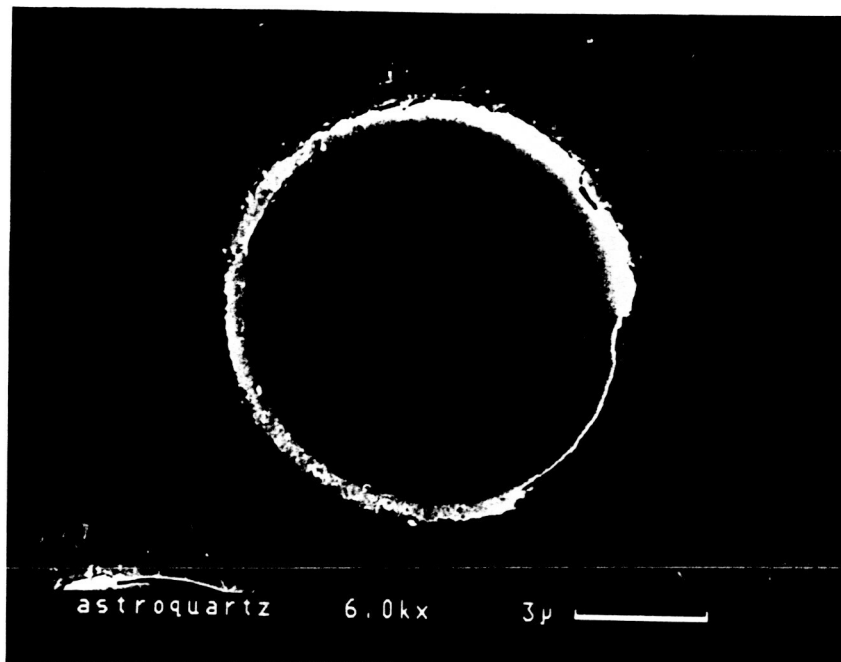
Additional Scanning Electron Microscope Photographs

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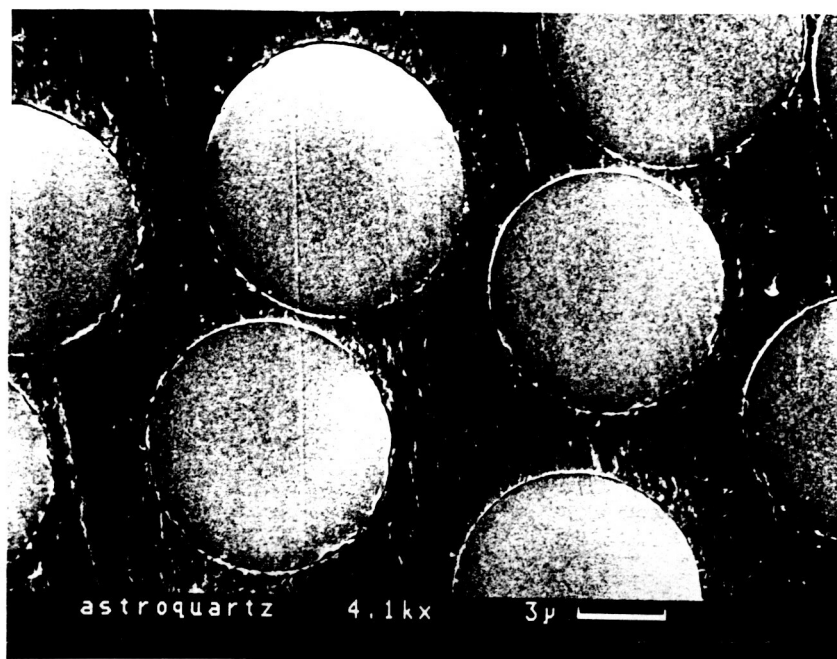


Astroquartz. Overall view of whole strand cross sectioned, areas 1-3.  
(200X)

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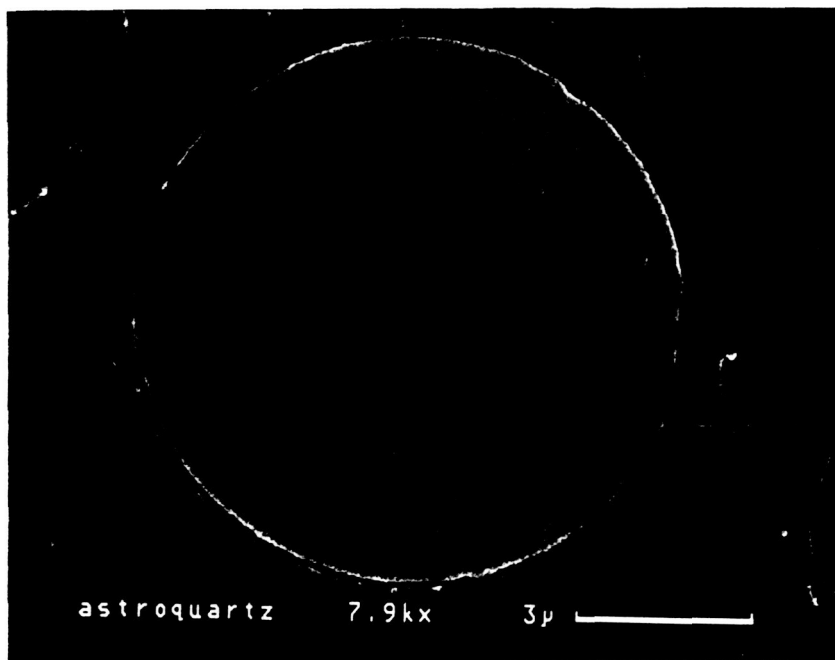


Astroquartz. General view of fiber from area 1. (6000X)



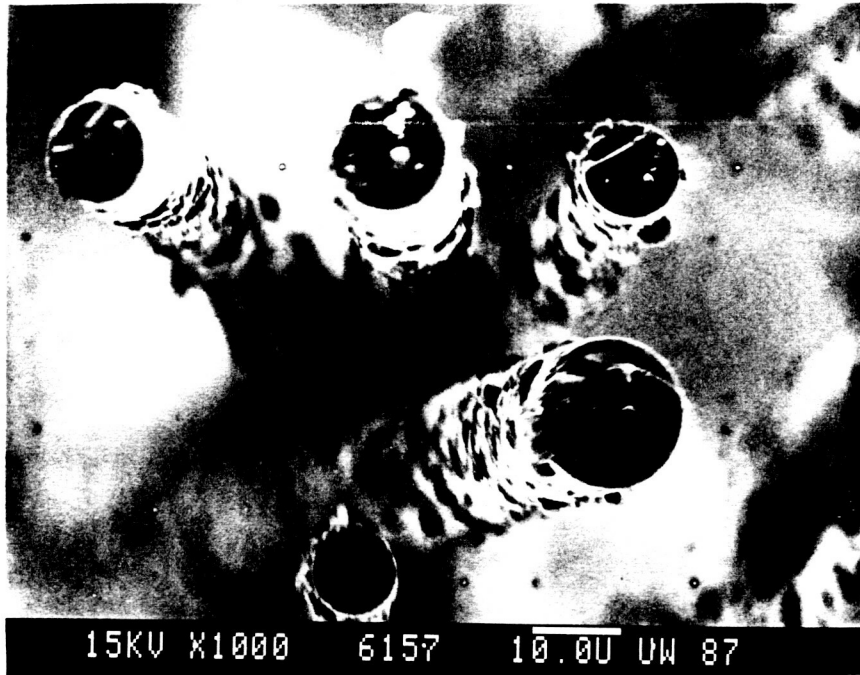
Astroquartz. General view of fibers from area 2. (4100X)

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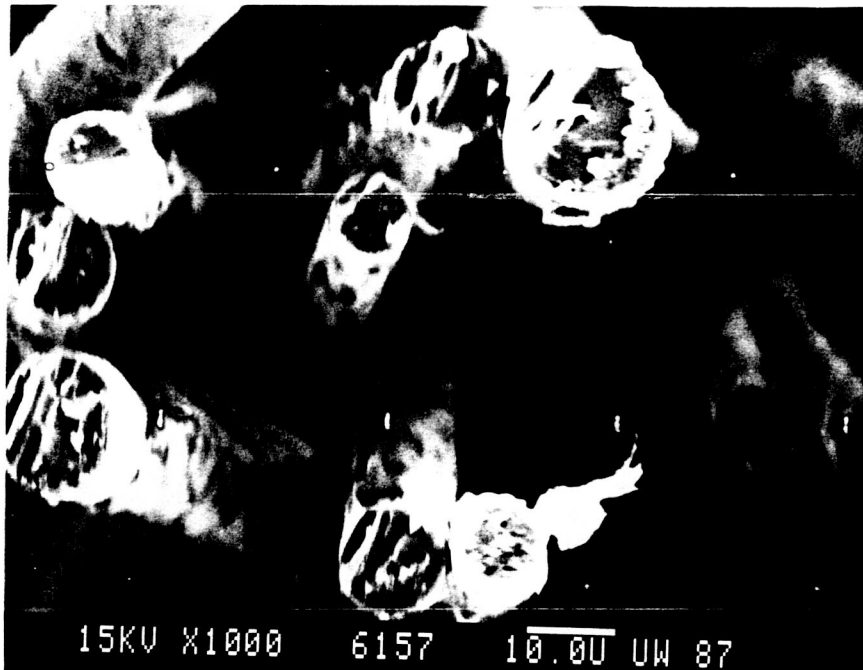


Astroquartz. Detailed view of fiber from area 2. (7900X)

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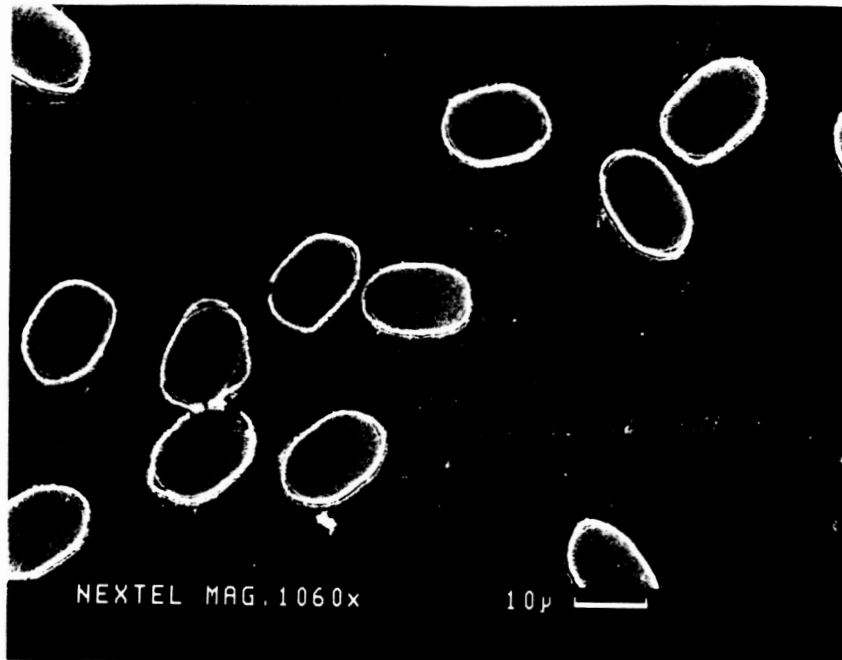
Group of Nicalon NLM-102 fibers. (1000X)



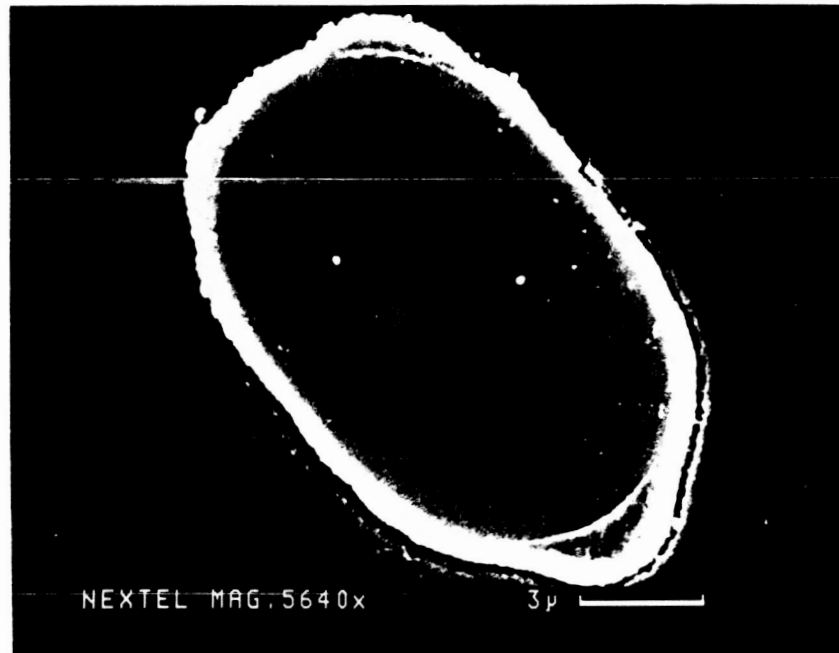
Group of Nicalon NLM-102 fibers. (1000X)



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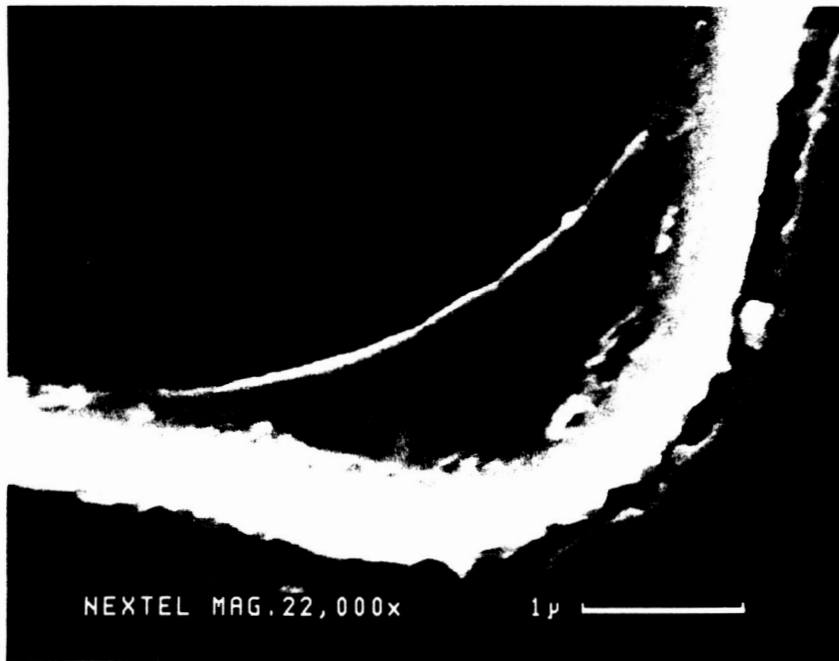


General view of Nextel 312 fibers. (1060X)

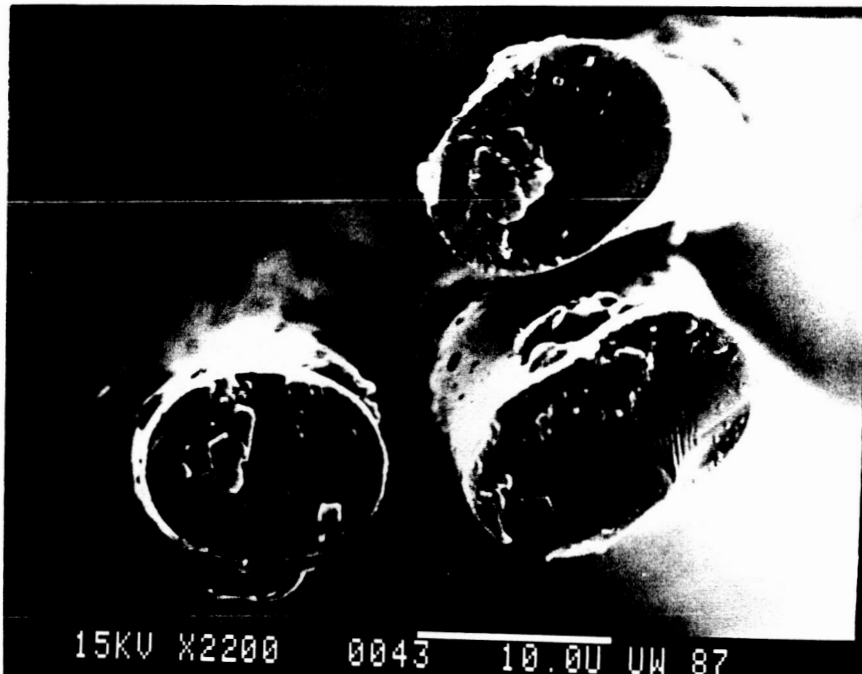


Detailed view of Nextel 312 fiber. (5640X)

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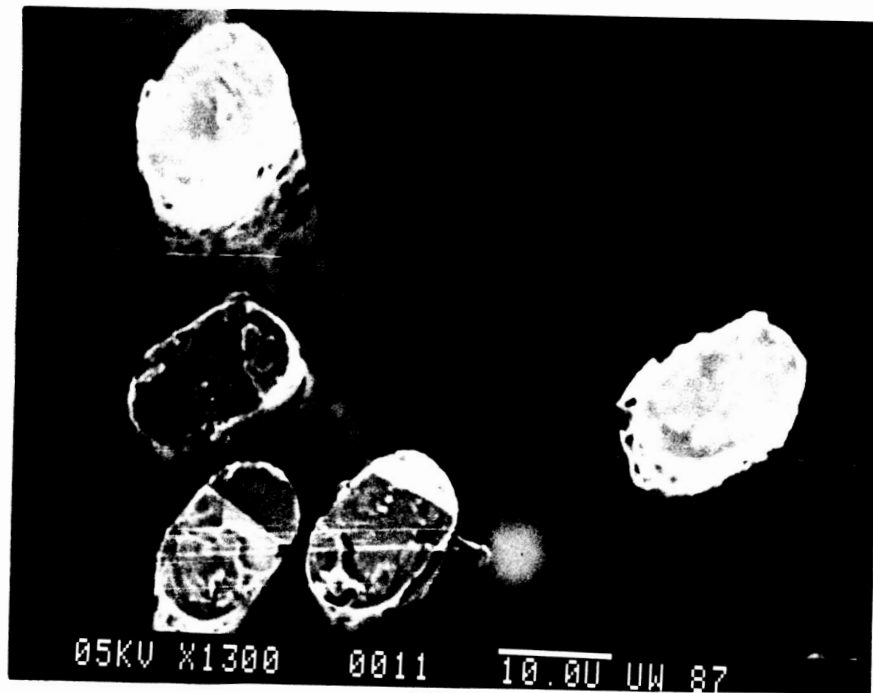


Detailed view of protrusion on Nextel 312 fiber. (22,000X)



Group of Nextel 312 fibers. (2200X)

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Group of Nextel 380 fibers. (1300X)

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Group of Nextel 480 fibers. (1000x)

1. Report No.		2. Government Accession No.		3. Recipient's Catalog No.	
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16. Abstract  <p>Static tensile and tensile creep testing of five ceramic fibers at elevated temperature was performed. J.P. Stevens, Co., Astroquartz 9288 glass fiber, Nippon Carbon, Ltd., (Dow Corning) Nicalon NLM-102 silicon carbide fiber, and 3M Company Nextel 312, 380, and 480 alumina/silica/boria fibers were supplied in unsized tows. Single fibers were separated from the tows and tested in static tension and tensile creep. Elevated test temperatures ranged from 400°C to 1300°C and varied for each fiber. Room temperature static tension was also performed. Computer software was written to reduce all single fiber test data into engineering constants using ASTM Standard Test Method D3379-75 as a reference.</p> <p>A high temperature furnace was designed and built to perform the single fiber elevated temperature testing up to 1300°C.</p> <p>A computerized single fiber creep apparatus was designed and constructed to perform four fiber creep tests simultaneously at temperatures up to 1300°C. Computer software was written to acquire and reduce all creep data.</p>					
17. Key Words (Suggested by Author(s)) ceramic fibers high temperature single fiber static tensile testing high temperature single fiber tensile creep testing			18. Distribution Statement  Unclassified, Unlimited		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of Pages 124	22. Price*

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