

NASA Technical Memorandum 100146

# Pressure Effects on the Thermal Stability of SiC Fibers

{NASA-TM-100146) PRESSURE EFFECTS ON THE  
THERMAL STABILITY OF SiC FIBERS (NASA) 16  
p Avail: NTIS HC A03/MF A01 CSCL 11D

N88-10120

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Martha H. Jaskowiak and James A. DiCarlo  
*Lewis Research Center*  
*Cleveland, Ohio*

Prepared for the  
88th Annual Meeting of the American Ceramic Society  
Chicago, Illinois, April 27—May 1, 1986

**NASA**

## PRESSURE EFFECTS ON THE THERMAL STABILITY OF SiC FIBERS

Martha H. Jaskowiak and James A. DiCarlo  
National Aeronautics and Space Administration  
Lewis Research Center  
Cleveland, Ohio 44135

### SUMMARY

Commercially available polymer-derived SiC fibers were treated at temperatures from 1000 to 2200 °C in vacuum and argon gas pressures of 1 and 1360 atm. Effects of increasing inert gas pressure on the thermal stability of the fibers were determined through studies of the fiber microstructure, weight loss, grain growth, and tensile strength. The 1360 atm argon gas treatment was found to shift the onset of fiber weight loss from 1200 to above 1500 °C. Grain growth and tensile strength degradation were correlated with weight loss and were thus also inhibited by high pressure treatments. Retreatment in 1 atm argon of the fibers initially treated at 1360 atm argon caused further weight loss and tensile strength degradation, thus indicating that high pressure inert gas conditions were effective only in delaying fiber strength degradation and that no permanent microstructural changes were induced.

### INTRODUCTION

With the growing technical interest in fiber-reinforced ceramic matrix composites, the demand is continually increasing for ceramic fibers with the properties necessary for yielding composites with high strength, high toughness, and temperature capability to 1400 °C and above (ref. 1). Commercially available polymer-derived SiC fibers offer many of these properties but show significant degradation in tensile strength beyond 1200 °C (refs 2 to 4). The generally accepted mechanism for the thermally-induced degradation is an increase in porosity and flaw growth due to internal reactions near 1200 °C involving excess C and SiO<sub>2</sub> which are present throughout the as-produced fiber structure (ref. 5). Another suggested mechanism is grain growth of the microcrystalline β-SiC that exists in the as-produced fiber (refs. 4 and 5).

The objective of this work was to determine the feasibility of improving the thermal stability of polymer-derived SiC fibers by heat treatment in a high pressure inert gas. To accomplish this, measurements were made of the strength and physical properties of commercially available SiC fibers after heat treatment between 1000 and 2200 °C at high and low pressures of argon. It was anticipated that high pressure argon could be advantageous in either of two ways. First, the degrading reaction involving C and SiO<sub>2</sub> could be significantly reduced since the high gas pressure would serve to delay evolution of the reaction products, CO and SiO gas, from the fiber pores until temperatures above 1200 °C. Delaying the reaction to higher temperatures might allow internal compositional changes to occur which could be beneficial to maintaining fiber strength. Second, the high argon gas pressure could act to densify the fiber since creep has been observed for polymer-derived SiC fibers at temperatures below 1200 °C (ref. 5). Densification could either inhibit CO and SiO gas evolution by closing off open porosity to the fiber surface or reverse any pore growth that would occur as a result of the C and SiO<sub>2</sub> reaction. If indeed any of the above phenomena would occur, it

would suggest that a hot isostatic press (HIP) treatment in argon could be an effective post-fabrication method for improving the thermal stability of polymer-derived SiC fibers.

## PROCEDURES

### Materials

Two types of the commercially available Nicalon<sup>1</sup> SiC fibers were studied in this work: standard grade (SG), NLM-102, and ceramic grade (CG), NLM 202. Both grades are derived from polycarbosilane polymer precursors. Because the polymer precursor is cured in air and does not completely convert to stoichiometric SiC during pyrolysis, the fibers are not pure SiC. Instead, they are composed of a random network of SiC, SiO<sub>2</sub>, excess carbon, and some combined nitrogen, with a slightly higher O<sub>2</sub> content existing in the NLM 102 fiber. Previous elemental analysis of the fibers has shown them to be composed of approximately 35 to 40 at % Si, 50 to 55 at % C, 6 to 10 at % O, and 2 at % N (ref. 6). In the as-received state the fibers are covered with a nonuniform sizing coating which is polyvinyl acetate for the NLM-102 and 202 fibers. The fibers were received in tow form with approximately 500 fibers per tow and an average fiber diameter of 16 to 20  $\mu\text{m}$ .

### Heat-treatments

Fiber samples were treated under vacuum and at 1 and 1360 atm of argon at temperatures ranging from 1000 to 2200 °C (depending on equipment capabilities). For treatment under vacuum at approximately 10<sup>-8</sup> atm, a thermogravimetric analysis (TGA) unit was used so that weight loss could also be monitored in real time. For treatment at 1 and 1360 atm of argon, fiber samples were placed in Grafoil<sup>2</sup> boats and loaded in a graphite lined induction furnace and a graphite resistance heated hot isostatic press (HIP), respectively. Because graphite will oxidize more readily and at lower temperatures than SiC, it was assumed that the Grafoil boats and graphite lined furnaces significantly reduced any residual oxygen partial pressure in the argon, thereby preventing oxidation effects on the fibers themselves. For all treatments, the samples were held at temperature and pressure for a 1 hr period. In the case of the high pressure runs, pressure was increased as rapidly as possible while the temperature was still below 1000 °C. Similar heating rates of 15 to 20 °C/min were used for all runs.

### Property Measurement

All fiber bundles were weighed before and after heat treatment to obtain weight loss measurements. Weight loss data for the fibers treated in vacuum were in good agreement with the TGA results. Crystallite sizes and phases of the fibers were determined using x-ray diffraction analysis on crushed samples from the as-received and heat treated fibers. Tensile strength tests were conducted on single fibers at room temperature using a 25 mm (1 in.) gauge length and a constant crosshead speed of 1.3 mm/min (0.05 in./min). Approximately 10 fibers were tested to determine the average tensile strength. Fracture surfaces from the tensile test were retained for microstructural studies by scanning electron microscopy.

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<sup>1</sup>Nippon Carbon Co., Tokyo Japan

<sup>2</sup>Union Carbide Co., Danbury

In order to determine whether the high pressure treatments effected any permanent changes in fiber microstructure and thermal stability, certain fiber samples were treated again under low pressure conditions. For example, fibers initially treated in the HIP at 1200 and 1600 °C were subjected to an additional treatment in 1 atm argon at 1400 °C. After the additional treatment, fiber weight loss and tensile strength were again measured.

## RESULTS

Weight loss behavior is displayed in figure 1 for fibers heated at the three pressures: a vacuum of  $10^{-8}$  atm, 1 atm argon, and 1360 atm argon. It can be observed from this data that the samples treated at the two lower pressures showed similar weight loss trends. It appeared that the increase in pressure from vacuum to 1 atm argon was not great enough to suppress the abrupt weight loss which began at approximately 1250 °C for both fiber types. Weight loss of the fibers treated at 1 atm appeared to level off at a maximum of approximately 26 and 28 wt % for the standard grade (SG) and ceramic grade (CG), respectively. Although limitations in the TGA prevented heat treatment above 1400 °C, it is assumed that the fibers treated in vacuum would also reach the same maximum weight loss values. From figure 1, it is also apparent that the HIP treatment was effective in delaying the onset of weight loss to a significantly higher temperature of approximately 1550 °C. The fibers treated at this high pressure reached a maximum weight loss equivalent to that of the fibers treated at the lower pressures.

The effects of pressure and temperature on the grain growth of the heat treated fibers is seen in figure 2. The grain growth followed the same basic trends as the weight loss for the treated fibers. Initially, the fibers possessed a microcrystalline structure with a grain size of 20 to 30 Å. Beyond 1200 °C, grain growth began at approximately the same temperature for the fibers treated at the two lower pressures. Once growth had been initiated, the grain size rapidly increased resulting in a very coarse microstructure. Again grain growth was suppressed until approximately 1550 to 1600 °C for both the ceramic and standard grade fibers treated at 1360 atm argon.

Tensile strength data for the treated fibers are shown in figure 3. Average room temperature strengths for the as-received ceramic and standard grade fibers were 1724 and 1593 MPa (250 and 231 ksi), respectively. The average standard deviation in the tensile strength before and after treatment was approximately ±35 percent. For the fibers heated to 1000 °C in vacuum, a slight strength loss was observed. Substantial loss in strength occurred between 1000 and 1200 °C for the vacuum treated fibers, after which the individual fibers could no longer be tensile tested. Strength degradation began at approximately 1200 °C for the fibers treated at 1 atm argon and around 1400 °C for the fibers treated in the HIP. For the samples treated at high pressure at 1800 °C and above, no further strength measurements could be made on the fibers. Thus the tensile strength data revealed slightly different trends than those seen for either weight loss or grain growth. Here the onset of strength loss was shifted upward approximately 200 °C with each increasing pressure treatment.

Microstructural examination of the fracture surfaces of the fibers (figs. 4 to 7) revealed changes in morphology as the fibers underwent heat treatment at increasingly higher temperatures. As seen in previous studies (ref. 7 and 8), small internal or surface flaws were typically observed as the



cause of brittle failure in the as-received fibers. After treatment at 1000 °C (fig. 4), these small flaws, which are inherent to the polymer-derived fibers, still appear to be the source of failure in the fibers, regardless of the pressure which was applied at these temperatures. The weight loss, which was still very low at this point (~0.8 percent), can be accounted for mainly by the sizing removal. Strength loss at 1000 °C was minimal for the fibers treated at 1360 and 1 atm argon. However, the fibers treated in vacuum had already undergone a significant strength loss.

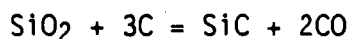
After treatment to 1400 °C (fig. 5), surface flaws were still responsible for the failure in the fibers treated at 1360 and 1 atm argon. The fibers treated in vacuum appear to have coarsened considerably, both along the surface of the fibers and within the cross-sectional area. For these fibers, the weight loss had reached its maximum level and tensile strength could no longer be measured. For the fibers treated at 1 atm, slight coarsening also occurred and again the weight loss was considerable. However, the fibers still maintained some of their strength. Only the fibers treated at 1400 °C in the HIP displayed a low weight loss, no grain growth, and good strength retention.

Fibers treated to 1600 °C (fig. 6) showed grain coarsening for all pressures. For the fibers treated at 1 atm, the increase in grain size and porosity greatly reduced fiber strength so that they could no longer be tensile tested. The fibers treated in the HIP, however, still displayed brittle fracture at this point with surface flaws as the primary cause of failure. The fiber strength dropped to approximately one third of its original value with still only a very slight weight loss. From figure 7 it is evident that further HIP treatment to 2200 °C led to a very coarse structure with significantly increased porosity and unmeasurable tensile strengths.

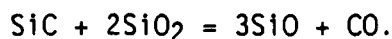
Results of additional heat treatment at 1 atm argon and 1400 °C are listed in table I for those fibers initially treated in the HIP at 1200 and 1600 °C. It was found that the subsequent heat treatment caused increased weight loss and tensile strength degradation. The weight loss reached approximately the maximum weight loss achieved with any of the as-received samples previously heat treated at 1 atm argon and 1400 °C, and the tensile strength once again became too low to measure.

## DISCUSSION

The HIP treatment in argon clearly delayed the onset of fiber weight loss to higher temperatures. The application of an external gas pressure of 1360 atm suppressed the evolution of gaseous products from the reactions involving excess C and SiO<sub>2</sub> which are found in the as-received fiber. These gaseous products are principally CO and SiO possibly according to the reactions (ref. 9):



and



By providing a method for removing excess  $\text{SiO}_2$  in the system these reactions could be beneficial for such properties as fiber creep resistance. However, they apparently also lead to fiber strength degradation by increasing fiber porosity and the size of strength limiting flaws.

Two possible mechanisms by which the high pressure gas can inhibit CO and SiO evolution are by either the formation of a CO and SiO-rich gaseous barrier near the fiber surface which could temporarily inhibit further evolution of these gases and thus delay the strength degrading reactions or by fiber densification leading to the closure of surface connected porosity. Whereas the former mechanism is temporary, the latter should lead to some permanent changes in fiber properties. The observation of an ultimate maximum weight loss value (approx 26 to 28 percent for all samples), which was independent of gas pressure both after initial and secondary treatments, indicates only temporary effects of high pressure. It therefore appears that high external gas pressure acts only to delay evolution of the gaseous reaction products from the fibers, thus significantly inhibiting the internal reactions leading to fiber degradation. This effect then delays the onset of fiber weight loss and porosity development to higher temperatures which are dependent on the magnitude of the applied pressure.

The suppression of grain growth by high pressure treatment can be explained through the dependence of grain growth on weight loss. In figure 8, the correlation between the fiber weight loss and grain growth can be seen. From this graph it appears that grain growth does not occur readily until approximately 15 to 20 percent weight loss is reached. It seems then that grain growth becomes favorable only with a substantial weight loss and the accompanying shift in composition. With the conversion of the microcrystalline structure to a perceptible grain structure and subsequent grain growth, it is expected that some local densification also occurred which resulted in shrinkage of the fibers. Although measurements to confirm this phenomena were not made in this study, there is evidence from length change measurements that such effects do occur at temperatures slightly higher than those corresponding to the onset of weight loss (ref. 5). Thus it is expected that fiber shrinkage and related physical effects were also delayed to higher temperature by the high pressure gas treatment.

By comparing figures 1 and 3, it can be observed that the initial and major loss in tensile strength occurred within the first few percent weight loss after sizing removal. From figure 8 it is evident that no noticeable grain growth occurred within the first few percent weight loss. Therefore, it appears that the major strength loss is related to the initial weight loss rather than the grain growth. This correlation can be seen in figure 9 where tensile strength is plotted against weight loss. Again it is evident that the strength loss occurs within the first 1 to 2 percent weight loss following the weight loss due to sizing removal. It is thought that this initial strength loss is due to reactions at the surface connected flaws and pores. At these surface connected sites the evolution of SiO and CO gases allows the pre-existing strength controlling flaws to rapidly grow and reduce fiber strength.

The microstructural study supports this theory of rapid growth of existing surface flaws. The micrographs of the fracture surfaces show very little visible change in fiber microstructure before the onset of strength degradation occurs. Most micrographs still displayed surface flaws as the cause of brittle fracture in the fibers where the strength had already fallen to half its original value. There appeared to be little or no grain coarsening and only a slight weight loss for these fibers.

The figure 9 correlation between strength degradation and weight loss for a range of pressures suggests that an approximate strength loss can be associated with each weight loss value, regardless of the pressure treatment used to arrive at the weight loss. This correlation implies that the high pressure argon gas treatments had little if any permanent effect either on improving fiber tensile strength or on affecting the mechanisms responsible for strength degradation. This conclusion was confirmed by the results of additional treatments shown in table I which indicate that the high pressure treatments were only temporary as measured by weight loss and tensile strength. Thus it would appear that inhibiting internal reactions to higher temperature does not allow compositional changes to occur which could be beneficial to fiber strength behavior. Nor does it appear that at 1600 °C and below, high pressure gas treatment results in bulk fiber densification effects which are measurable within the properties examined in this study. The initial porosity of the fibers may have been too high to effectively HIP the fibers without encapsulation.

#### CONCLUDING REMARKS

It has been demonstrated that application of high external gas pressure can temporarily inhibit the reactions which lead to degradation of the polymer-derived SiC fibers. High pressure treatment delayed fiber weight loss to temperatures above 1500 °C. Strength degradation and grain growth were correlated with weight loss and were thus also inhibited by high pressure treatment. Major strength losses were observed at very low weight loss levels; whereas the grain growth did not proceed until greater weight loss had occurred. Because the substantial strength losses occurred at such low weight losses, the major strength degradation mechanism is thought to be due to the reactions at the surface connected flaw sites. The reactions at these flaw sites involve the evolution of gaseous products which lead to the rapid growth of the pre-existing flaws. While the high gas pressure was effective in suppressing the fiber weight loss, grain growth, and strength loss, the effects disappeared after pressure removal. Thus hot isostatic pressing under inert gas does not appear to be an effective post-fabrication method for permanently improving the fiber thermal stability.

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TABLE 1. - RESULTS OF ADDITIONAL HEAT TREATMENT TESTS  
ON HIGH PRESSURE TREATED SiC FIBERS

Heat treatment conditions	Weight loss, percent		Tensile strength, MPa (ksi)	
	CG	SG	CG	SG
1200 °C - 1 hr 1360 atm Ar	0.8	0.7	1627 (236)	1869 (271)
1200 °C - 1 hr 1360 atm Ar + 1400 °C - 1 hr 1 atm Ar	28	24	---	---
1600 °C - 1 hr 1360 atm Ar	0.8	2.0	621 (90)	538 (78)
1600 °C - 1 hr 1360 atm Ar + 1400 °C - 1 hr 1 atm Ar	23	26	---	---

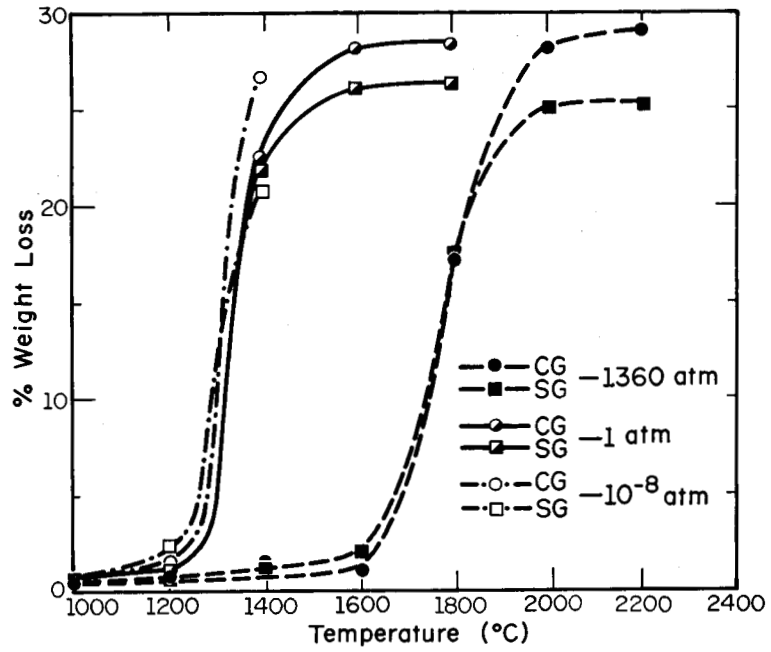


FIGURE 1. - EFFECTS OF TEMPERATURE AND ARGON GAS PRESSURE ON THE WEIGHT LOSS OF CERAMIC GRADE (CG) AND STANDARD GRADE (SG) SiC FIBERS.

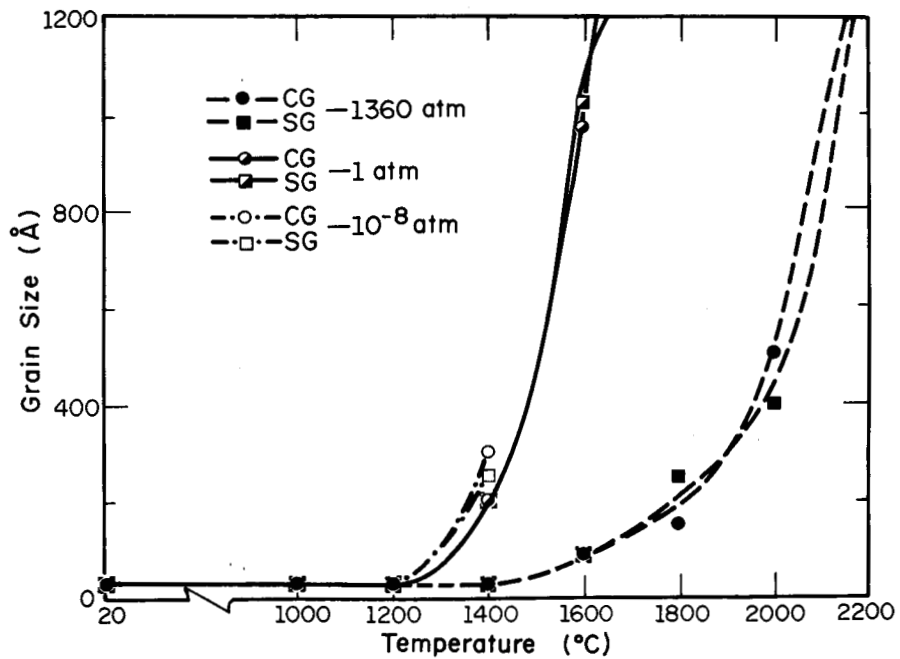


FIGURE 2. - EFFECTS OF TEMPERATURE AND ARGON GAS PRESSURE ON THE GRAIN GROWTH OF CG AND SG SiC FIBERS.

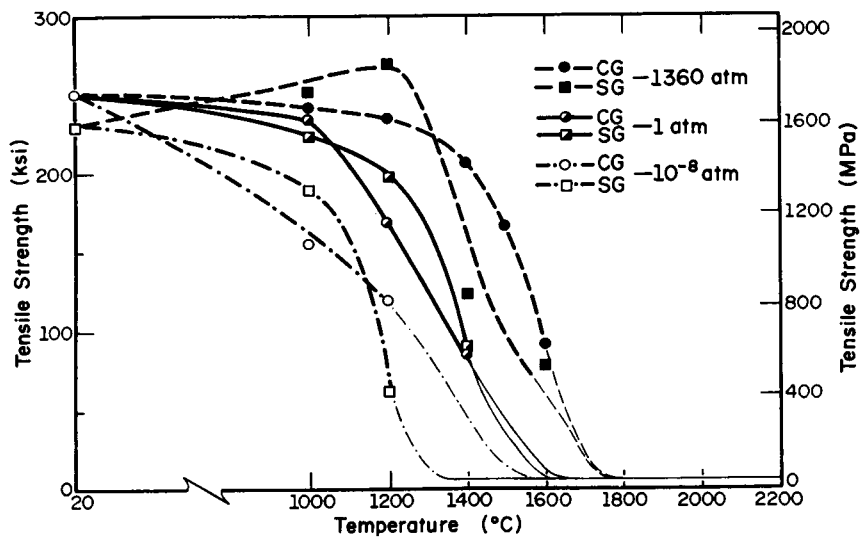
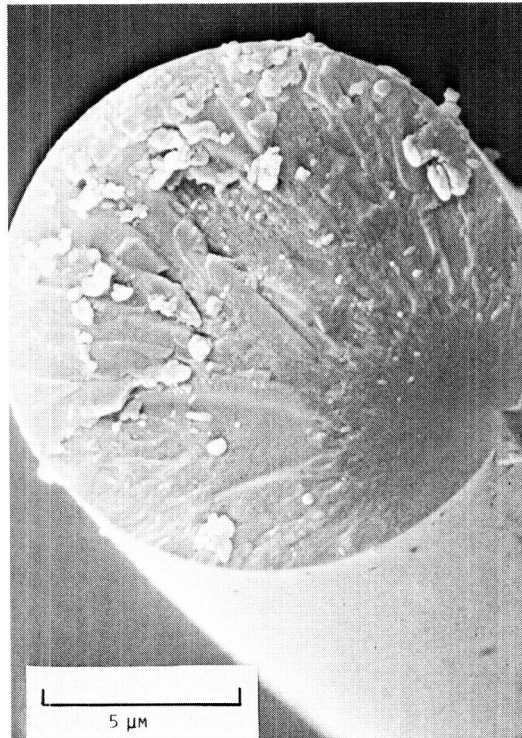


FIGURE 3. - EFFECTS OF TEMPERATURE AND ARGON GAS PRESSURE ON THE AVERAGE ROOM TEMPERATURE TENSILE STRENGTH OF SINGLE CG AND SG SiC FIBERS.

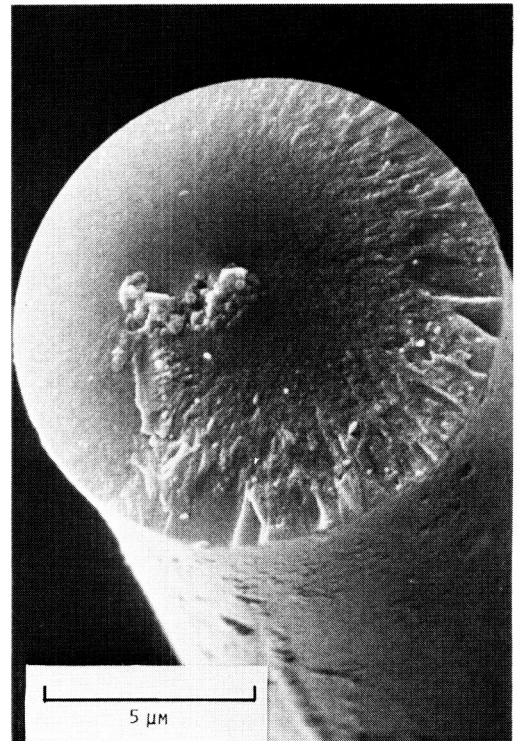
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(A) FIBERS TREATED AT 1360 ATM AR, WITH WT LOSS OF 0.6% AND TENSILE STRENGTH OF 240 KSI (1655 MPa).



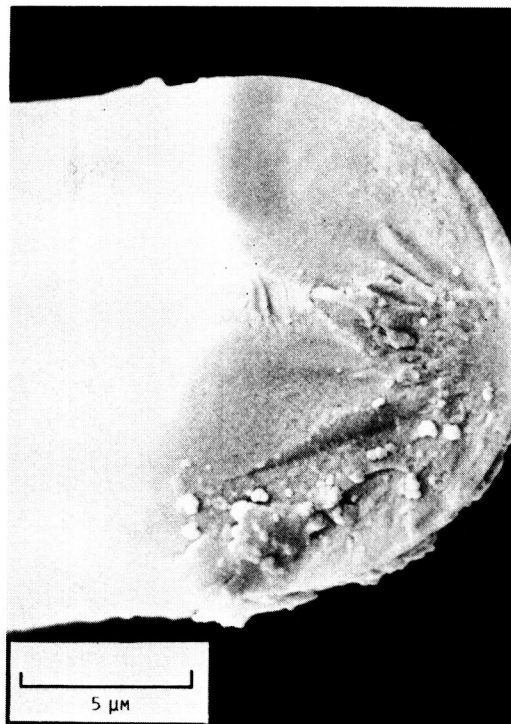
(B) FIBERS TREATED AT 1 ATM AR, WITH WT LOSS OF 0.8% AND TENSILE STRENGTH OF 235 KSI (1620 MPa).



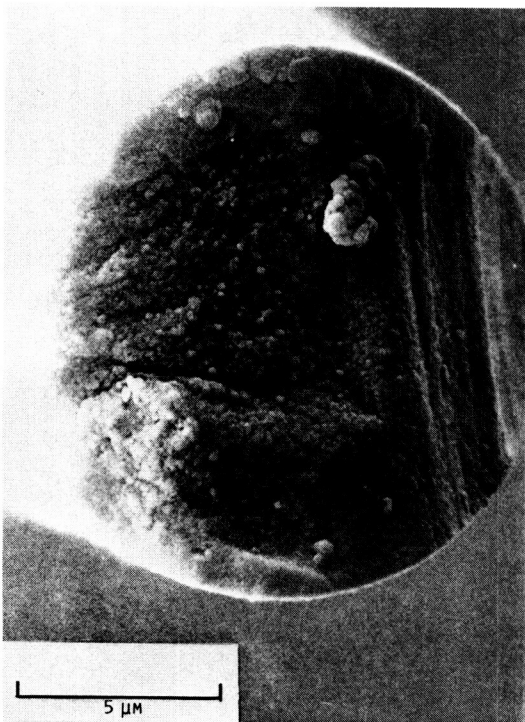
(C) FIBERS TREATED AT  $10^{-8}$  ATM, WITH WT LOSS OF 0.8% AND TENSILE STRENGTH OF 154 KSI (1062 MPa).

FIGURE 4. - SEM MICROGRAPHS OF CERAMIC GRADE SiC FIBERS TREATED AT 1000 °C IN VARIOUS ARGON PRESSURES. FOR COMPARISON, FIBER WEIGHT LOSS AND ROOM TEMPERATURE TENSILE STRENGTH RESULTS ARE INDICATED.

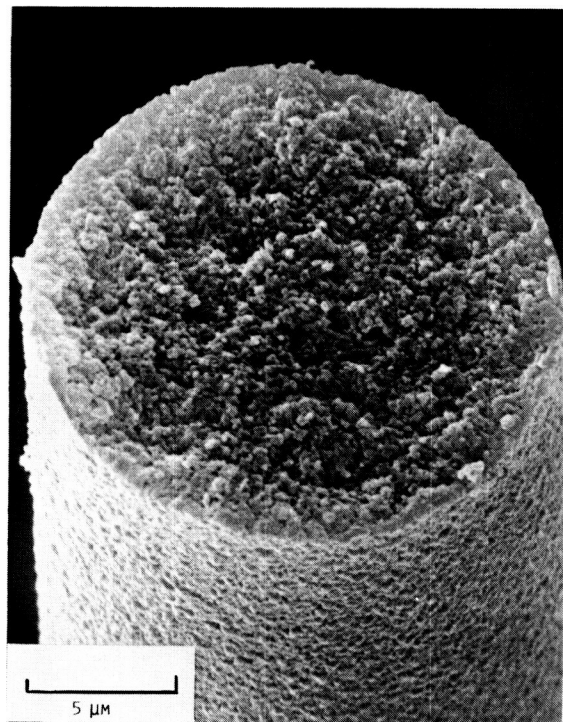
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(A) FIBERS TREATED AT 1360 ATM AR, WITH WEIGHT LOSS OF 1.0% AND TENSILE STRENGTH OF 208 KSI (1434 MPa).



(B) FIBERS TREATED AT 1 ATM AR, WITH WEIGHT LOSS OF 22% AND TENSILE STRENGTH OF 85 KSI (586 MPa).

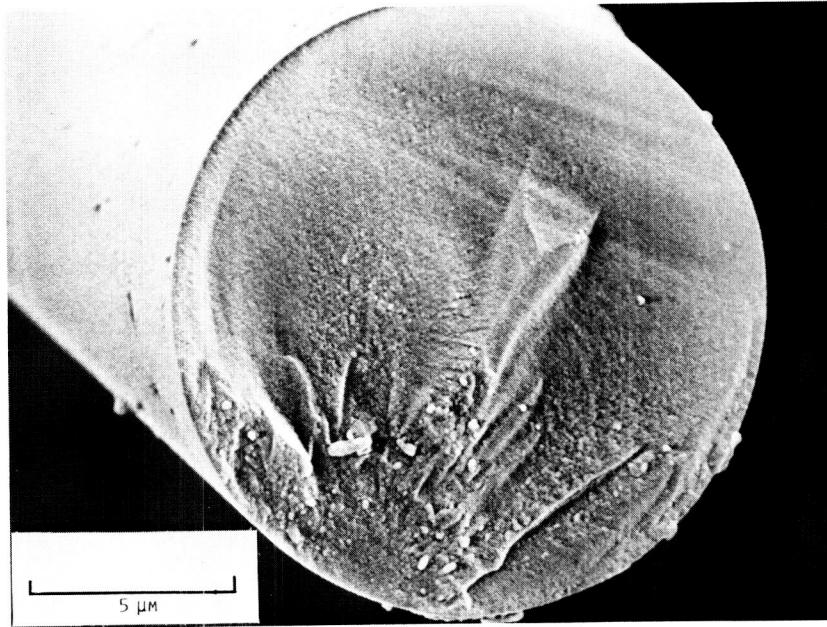


(C) FIBERS TREATED AT  $10^{-8}$  ATM, WITH WEIGHT LOSS OF 27% AND TENSILE STRENGTH TOO LOW TO MEASURE.

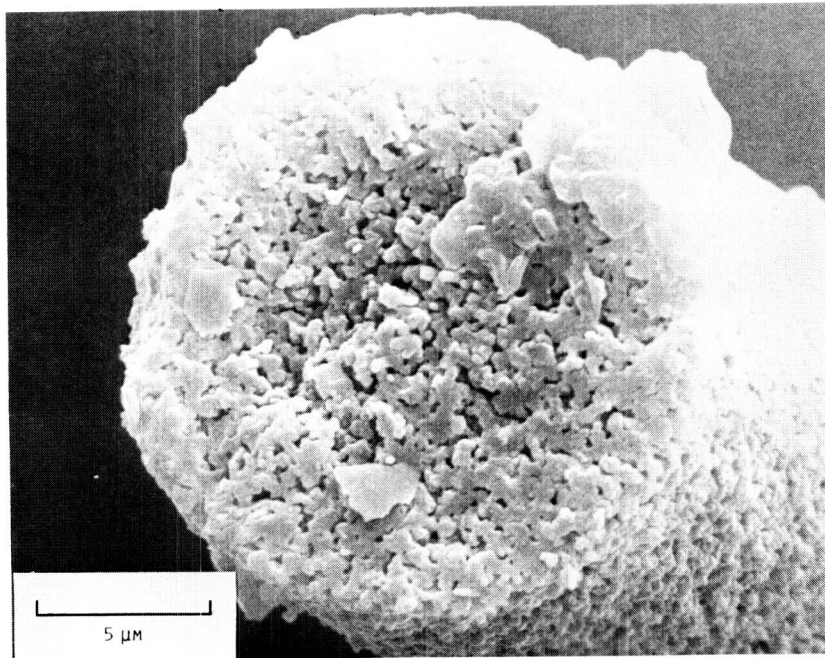
FIGURE 5. - SEM MICROGRAPHS OF CERAMIC GRADE SiC FIBERS TREATED AT 1400 °C IN VARIOUS ARGON PRESSURES. FOR COMPARISON, FIBER WEIGHT LOSS AND ROOM TEMPERATURE TENSILE STRENGTH RESULTS, WHEN MEASUREABLE, ARE INDICATED.



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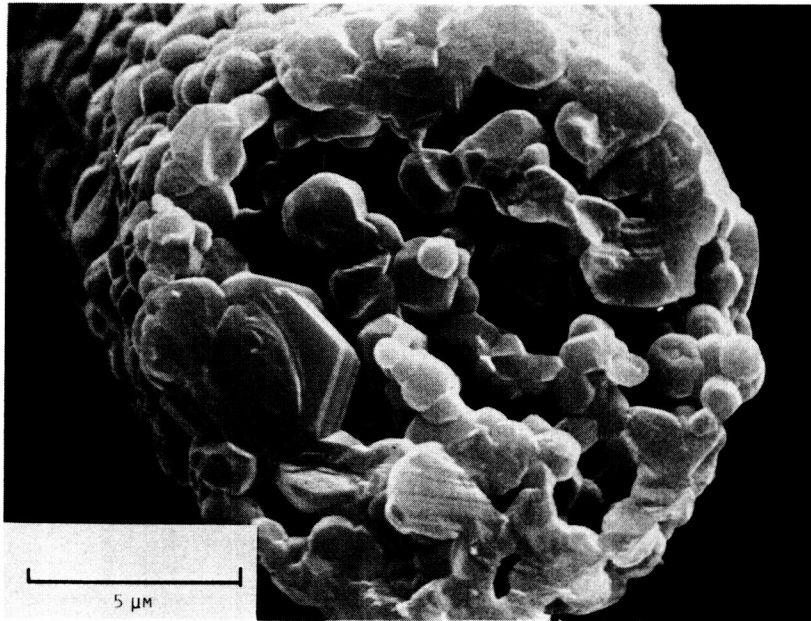
(A) FIBERS TREATED AT 1360 ATM AR, WITH WEIGHT LOSS OF 0.8% AND TENSILE STRENGTH OF 90 KSI (621 MPa).



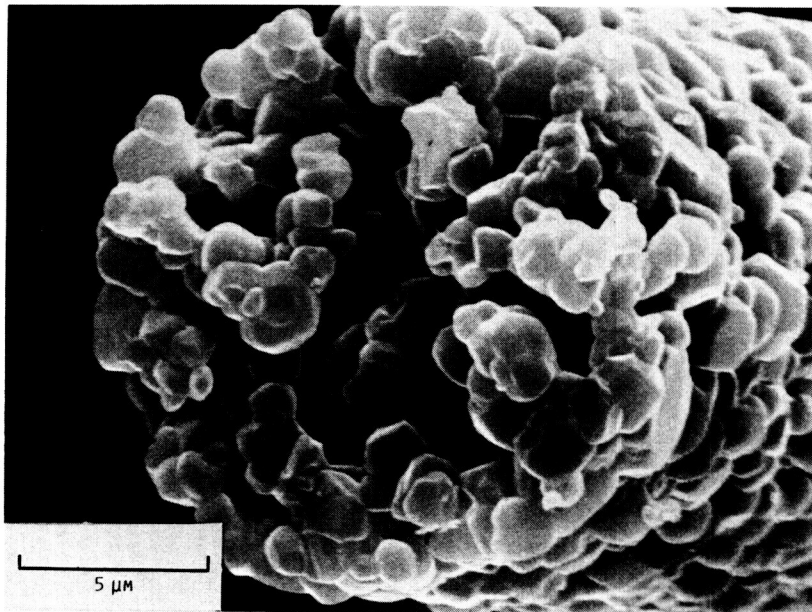
(B) FIBERS TREATED AT 1 ATM AR, WITH WEIGHT LOSS OF 28% AND TENSILE STRENGTH TOO LOW TO MEASURE.

FIGURE 6. - SEM MICROGRAPHS OF CERAMIC GRADE SiC FIBERS TREATED AT 1600 °C IN VARIOUS ARGON PRESSURES. FOR COMPARISON, FIBER WEIGHT LOSS AND ROOM TEMPERATURE TENSILE STRENGTH RESULTS ARE INDICATED.

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(A) STANDARD GRADE FIBERS WITH 25% WT LOSS.



(B) CERAMIC GRADE FIBERS WITH 28% WT LOSS.

FIGURE 7. - SEM MICROGRAPHS OF STANDARD AND CERAMIC GRADE SIC FIBERS TREATED AT 2200 °C IN 1360 ATM ARGON. AVERAGE FIBER WEIGHT LOSS PERCENT RESULTS ARE INDICATED. RESIDUAL TENSILE STRENGTHS WERE UNMEASURABLE.

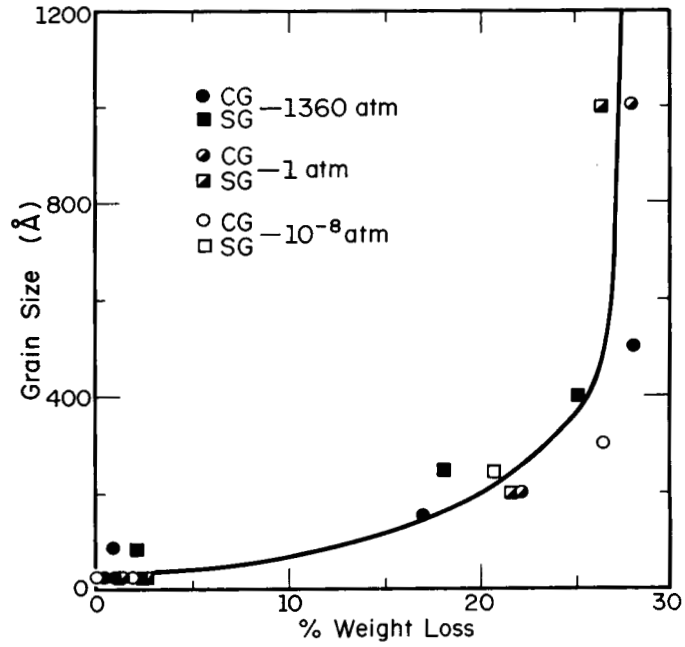


FIGURE 8. - CORRELATION BETWEEN GRAIN GROWTH AND FIBER WEIGHT LOSS FOR HEAT AND PRESSURE TREATED SiC FIBERS.

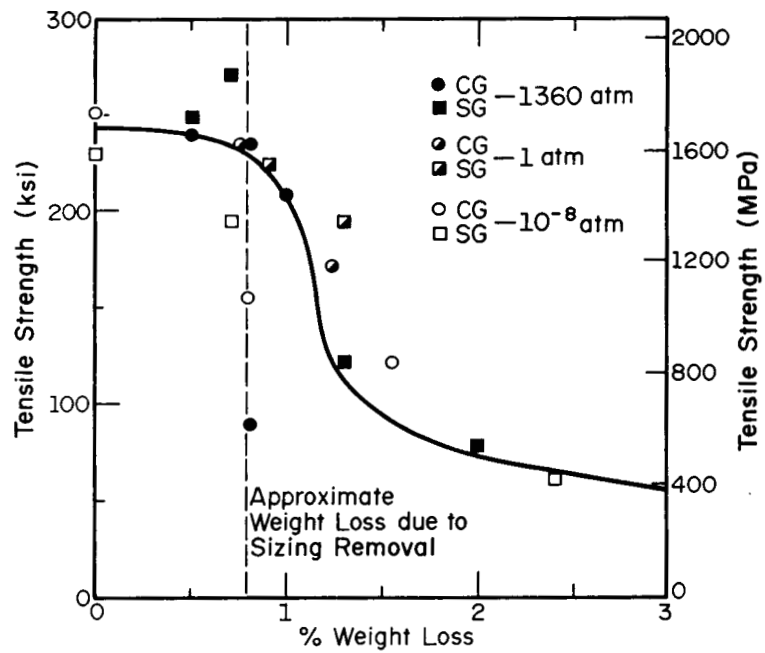


FIGURE 9. - CORRELATION BETWEEN RESIDUAL FIBER TENSILE STRENGTH AND WEIGHT LOSS FOR HEAT AND PRESSURE TREATED SiC FIBERS.

1. Report No. <b>NASA TM-100146</b>		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle <b>Pressure Effects on the Thermal Stability of SiC Fibers</b>				5. Report Date	
				6. Performing Organization Code <b>505-63-81</b>	
7. Author(s) <b>Martha H. Jaskowiak and James A. DiCarlo</b>				8. Performing Organization Report No. <b>E-3704</b>	
				10. Work Unit No.	
9. Performing Organization Name and Address <b>National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135</b>				11. Contract or Grant No.	
				13. Type of Report and Period Covered <b>Technical Memorandum</b>	
12. Sponsoring Agency Name and Address <b>National Aeronautics and Space Administration Washington, D.C. 20546</b>				14. Sponsoring Agency Code	
15. Supplementary Notes <b>88th Annual Meeting of the American Ceramic Society, Chicago, Illinois, April 27 - May 1, 1986.</b>					
16. Abstract <p>Commercially available polymer-derived SiC fibers were treated at temperatures from 1000 to 2200 °C in vacuum and argon gas pressure of 1 and 1360 atm. Effects of gas pressure on the thermal stability of the fibers were determined through property comparison between the pressure treated fibers and vacuum treated fibers. Investigation of the thermal stability included studies of the fiber microstructure, weight loss, grain growth, and tensile strength. The 1360 atm argon gas treatment was found to shift the onset of fiber weight loss from 1200 to above 1500 °C. Grain growth and tensile strength degradation were correlated with weight loss and were thus also inhibited by high pressure treatments. Additional heat treatment in 1 atm argon of the fibers initially treated at 1360 atm argon caused further weight loss and tensile strength degradation, thus indicating that high pressure inert gas conditions would be effective only in delaying fiber strength degradation. However, if the high gas pressure could be maintained throughout composite fabrication, then the composites could be processed at higher temperatures.</p>					
17. Key Words (Suggested by Author(s)) <b>Polymer-derived; Fiber thermal stability; Pressure treatment; Physical properties; Mechanical properties</b>			18. Distribution Statement <b>Unclassified - unlimited STAR Category 24</b>		
19. Security Classif. (of this report) <b>Unclassified</b>		20. Security Classif. (of this page) <b>Unclassified</b>		21. No of pages <b>15</b>	22. Price* <b>A02</b>