



Ultra High Temperature (UHT) SiC Fiber (Phase II)

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This report contains preliminary findings, subject to revision as analysis proceeds.

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Background

Silicon-carbide fiber-reinforced silicon-carbide ceramic matrix composites (SiC/SiC CMC) are emerging lightweight structural materials not only for hot section components in gas turbine engines, but also for control surfaces and leading edges of reusable hypersonic vehicles as well as for nuclear propulsion and reactor components. It has been shown that when these CMC are employed in engine hot-section components, the higher the upper use temperature of the SiC fiber and matrix constituents, the more performance benefits are accrued, such as higher operating temperatures up to 2700 °F, reduced component cooling air, reduced fuel consumption, and reduced emissions. The first generation of SiC/SiC CMC with a temperature capability of 2200 to 2400 °F are on the verge of being introduced into the hot-section components of commercial and military gas turbine engines.

Today the SiC fiber type currently recognized as the world's best in terms of thermo-mechanical performance is the "Sylramic-iBN" fiber. This fiber was previously developed by the Principal Investigator at the NASA Glenn Research Center using patented processes (US-7687016) to improve the durability of the high-cost commercial "Sylramic" fiber, which in turn was derived from the low-cost low-performance commercial Lox-M fiber. Although the Sylramic-iBN fiber shows state-of-the art (SOA) creep and rupture resistance for use temperatures up to ~2550 °F and is coated with a thin in-situ grown boron-nitride (iBN) protective layer, NASA has shown by fundamental creep studies and model development that its microstructure and creep resistance could be significantly improved to produce an Ultra High Temperature (UHT) SiC fiber. In addition, because the high stiffness of the Sylramic-iBN fiber limits its formability into the fiber architectures needed for complex-shaped CMC components, the advanced UHT SiC fiber should also seek to reduce this issue.

Purpose of Project

This Phase II Seedling Fund effort continues Phase I efforts (Ref. 1) focused on the key objective of developing a UHT SiC fiber by effectively repeating similar processes used for producing the Sylramic-iBN fiber and by employing a design of experiments approach to first understand the cause of the less than optimum Sylramic-iBN microstructure and then attempting to develop process conditions that eliminate or minimize these key microstructural issues. In so doing, it is predicted that that these advanced processes could result in an UHT SiC fiber with >20 times more creep resistance than the Sylramic-iBN fiber, which in turn would allow SiC/SiC CMC to operate up to 2700 °F and above, further enhancing the performance benefits of SiC/SiC components in aero-propulsion engines. It is also envisioned that the fiber processes developed during Phase II efforts would not only reduce production costs for the UHT fiber by combining processes and using low-cost precursor fibers, but also would allow the UHT fibers to be directly produced within complex-shaped architectural preforms of the precursor fibers, which, because of their lower stiffness, are more amenable to typical textile-forming processes.

If successful, the UHT SiC fiber production approach selected for this project is expected to be innovative in multiple ways in that

- It begins with a low-cost, low-grade precursor fiber which is converted by judiciously selected high-temperature chemical processes into a state-of-the-art high-performance UHT SiC fiber. The fiber temperature capability is projected to be at least 300 °F higher than the Sylramic-iBN fiber.
- It can be applied to precursor fibers within a variety of textile-formed architectures, which can range from continuous lengths of multifiber tows to the complex-shaped architectural preforms needed for reinforcement of multidirectionally stressed CMC components.
- It can be used for a wide range of commercial precursor fiber types with different additives that may provide extra beneficial properties to the final UHT fiber.
- It can be stream-lined with less process steps than currently employed for the best SiC fibers, and thus be more cost-effective.

The UHT fiber approach is also expected to produce high performance fibers with useful properties other than greater temperature capability, such as, high thermal conductivity, and with surface coatings that are not only environmentally protective, but also compliant enough to provide the weak matrix bonding needed for tough CMC.

Approach

For providing SiC/SiC CMC with structural reliability at high temperatures, polycrystalline SiC fibers must meet a variety of property requirements (Ref. 2), the most important of which are high rupture strength and high creep resistance. These fibers are thermally stable to well over 3000 °F, but under stress will rupture with time at much lower temperatures due to creep and creation of flaws as grains slide over one another. Fiber creep and rupture resistance can be improved by increasing grain size, grain size uniformity, and viscosity of the grain boundary phases. Currently the state-of-the-art Sylramic-iBN SiC fiber is limited in temperature capability to ~2500 °F due to a variety of microstructural issues. Key amongst these is the fact that the iBN fiber and its precursor Sylramic fiber typically display a center core region where the SiC grains are smaller in size than the grains near the fiber surface, thereby creating a shell and core morphology within the fiber cross-section, as shown in Figure 1. Associated with this center core is excess carbon and a high density of small voids that remain after final processing. Since creep-resistance (and temperature capability) increases with grain size, the shell region provides the primary fiber structural capability at high temperatures. Thus as one aspect of the UHT fiber development, process conditions are being sought that result in little if any excess creep-prone carbon or

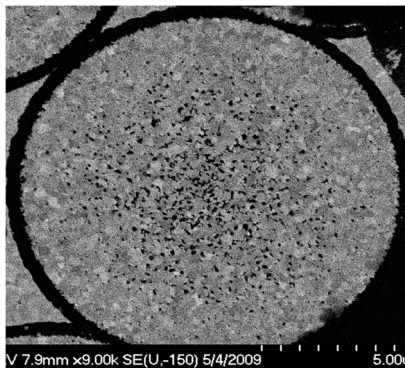


Figure 1.—Sylramic-iBN SiC fiber core-shell morphology with small voids (and excess carbon) in the core.

volume fraction of voids in the fiber core and a nearly uniform grain size across the fiber diameter so that the whole cross-section will be load bearing at high temperatures. Another objective will be to develop process conditions that increase the average grain size without significantly debiting fiber strength since the grains are effectively flaws in the fiber. Increased grain size will not only increase fiber creep resistance, but also increase fiber thermal conductivity, which is important in reducing thermal stresses in SiC/SiC CMC components.

The technical approach selected for this project is to follow process steps similar to those of Sylramic-iBN fiber, but apply innovative thermo-chemical treatments at various process stages that result in a UHT fiber with larger grain sizes more uniformly distributed in the cross-section, with reduced porosity, and with higher viscosity phases in the grain boundaries. The general processing approach selected for the UHT fiber is shown in Figure 2.

First, amorphous commercial precursor SiCO fibers, which contain a high fraction of silicon oxycarbide impurity phases, are heat-treated in high-purity argon within Stage 1 furnaces to allow gaseous decomposition of the oxide phases leaving pores behind within the fibers. In Phase I, the precursor fibers were in straight multifiber tow form; while in Phase II the goal was to look at both straight tows and tows woven into complex architectural preforms. Just as in the production of the commercial Sylramic fiber (Ref. 3), the porous fibers are then infiltrated with a boron-containing gas to form solid boron-containing phases within the fiber pores that act as sintering aids. The boron-doped fiber is then exposed to a high sintering temperature where, after the properly selected decomposition and infiltration processes, the infused phases are intended to facilitate growth of the fiber grains uniformly across the fiber diameter, resulting in the dense and nearly-stoichiometric SiC fiber. Then, as in the development of the Sylramic-iBN fiber, since the infused boron-containing phases inhibit fiber creep resistance, the sintered fiber is exposed in the Stage 2 furnaces to nitrogen-containing gases that remove and alter the creep-prone phases so that the final fiber will not only display increased creep resistance and temperature capability, but also a thin protective crystalline BN coating on all fiber surfaces (Ref. 4). A key metric for the UHT fiber, shown in Figure 3, is a fiber with higher rupture strength and better retention of this strength than the iBN fiber as measured under constant stress in air at 2550 °F. Actual upper use temperature for the UHT fiber would depend on the life requirements and stresses within a UHT fiber-reinforced CMC component.

Once UHT fibers are satisfactorily developed by experimental tests on single fibers removed from multifiber tows, technical efforts were planned aimed at developing and demonstrating the UHT SiC fiber in multiple 2D and 3D architectural preforms that should display thermo-structural capability similar to that of the fiber. These preforms will be needed as reinforcement for multidirectionally stressed components, such as cooled CMC vanes and blades, but are not easily obtainable with current high-performance high-modulus SiC fibers due to their tendency to fracture when sharply bent to form the complex architecture. A final scale-up objective was to seek ways for streamlining the UHT processes and for making them as cost-effective as possible.

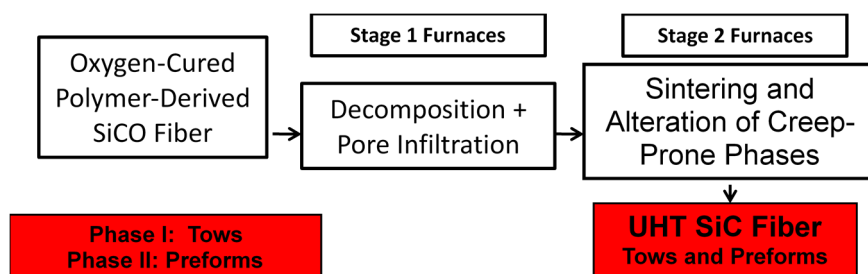


Figure 2.—The general process stages selected for producing the UHT SiC fiber.

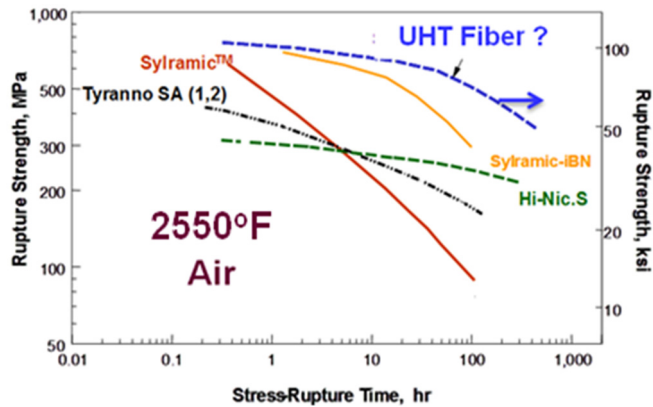


Figure 3.—Metric goal for the UHT SiC fiber: better rupture strength and strength retention in air at 2550 °F than the state-of-the-art Sylramic-iBN fiber.

Beyond the technical approaches described above, efforts were planned to demonstrate industry-adaptable production methods for the UHT SiC fiber both as continuous multifilament tows and as final preforms of CMC components. These efforts will involve collaboration and transfer of the developed UHT fiber technologies to select fiber vendors and to CMC end-users to verify enhanced fiber performance in commercially-produced SiC/SiC components. For transfer of production methods for UHT fiber tows, the infusion process will be faster since we are improving the properties of an existing precursor fiber and thus the implementation will not require establishment of a new fiber production line. However, it may be more significant and more cost-effective to transfer the processes for UHT fiber preforms directly to select CMC vendors, where they perform the UHT preform conversion before infiltrating the preforms with matrix material to form the final CMC component.

Phase II Research

The primary objective of the Phase II efforts was to continue the fiber development efforts initiated in Phase I and described in the Final Phase I report (Ref. 1). During both phases, two types of oxidation-cured commercial SiC fibers, the Japanese-produced “Lox-M” and “Nicalon” SiC fibers, were selected as the UHT precursor fibers. These fibers are not only low cost and commercially available in large volume, but also display low moduli that allows them to be easily woven into complex-shaped fiber preforms without fracturing prior to their conversion.

Experimental Procedure

As in Phase I, the experimental procedure was to place straight multifiber tows of the precursor fibers over a BN boat containing boron-containing powder, and the to process the fibers in a Stage 1 furnace under various time-temperature conditions through the steps of (1) gaseous decomposition of their silicon-oxycarbide impurity phases, (2) doping with boron-containing gas, and (3) pre-sintering at an upper temperature limited by furnace capability. Processed tows were then removed from the Stage 1 furnace and subjected to final sintering in the Stage 2 one-atmosphere argon furnace at 1800 °C for 1 hr. After each process step, microstructures were then characterized for single fibers taken from the tows to monitor and understand the physical-chemical changes occurring in the microstructures. If the sintered microstructures appeared to show no visible pores in the fiber core, the bend creep, tensile strength, and high-temperature rupture-strength of single fibers were then measured using GRC-developed procedures and facilities (Ref. 5). If the results of these tests compared well against our creep resistance and tensile strength requirements, the sintered fibers would then be subject to high-temperature nitrogen treatments to further improve the creep resistance and form an in-situ grown BN coating on the fibers.

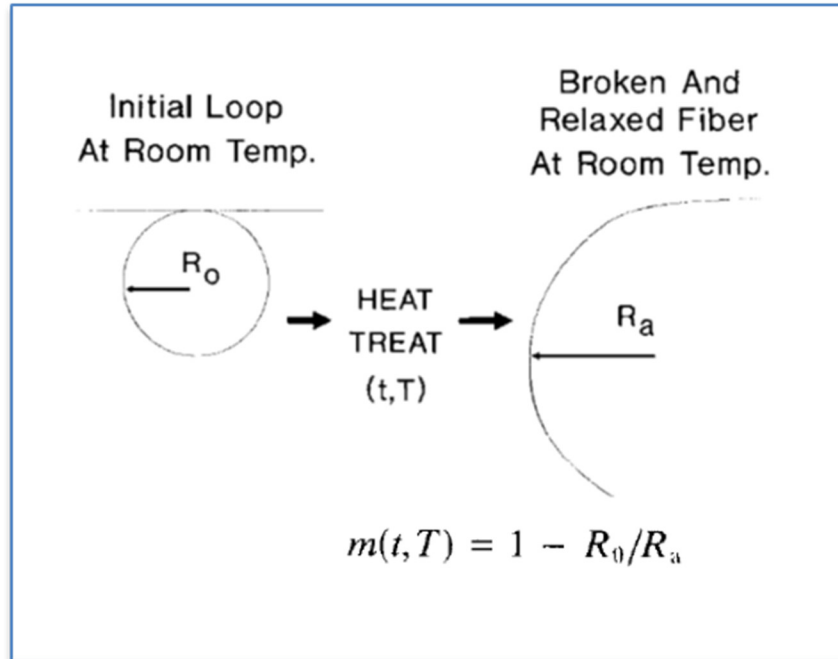


Figure 4.—Experimental details of the fiber Bend Stress Relaxation (BSR) test.

For meeting the UHT improved creep-resistance and temperature capability requirements, single processed fibers were subjected to the NASA-developed Bend Stress Relaxation (BSR) test to evaluate their performance against other SiC fibers (Ref. 6). Figure 4 shows the key details of this test in which the processed fiber is bent around a circular graphite mandrel and then heat treated for 1 hr in argon at high temperatures. The stress relaxation ratio, m , is defined as the ratio of final to initial stress:

$$m = \sigma(t, T, \epsilon_0) / \sigma_0(t = 0, T, \epsilon_0) = 1 - R_0/R_a. \quad (1)$$

As shown in Equation (1), the m value can simply be determined by the radius the fiber is subjected to on the mandrel, R_0 , and the remaining radius after removal from the mandrel, R_a . If $m = 1$ (complete spring back, $R_a = \infty$), then the fiber behaves perfectly elastically, i.e., no time-dependent creep deformation. If $m = 0$ ($R_0 = R_a$), then the fibers fully relaxed. Therefore, the higher the value of m for the same time/temperature conditions for a given fiber, the less stress relaxation (more creep resistance) occurred for that fiber when compared to another fiber-type.

The m -ratio results for the processed and sintered fibers were then compared against previous results for the Sylramic and Sylramic-iBN fibers to show after each process step whether the converted precursor fibers have improved in creep resistance over these two high-performance SiC fibers. For example, Figure 5 shows the m -ratio behavior for the Lox-M and Nicalon precursor fibers and how one might expect their creep resistance to improve, first to be equivalent to the Sylramic fiber, then the Sylramic-iBN fiber, and then to be further improved as a UHT fiber.

It should be mentioned that at the initiation of Phase II, in order to separate and better understand process effects during the decomposition and doping steps for both precursor types, a manual linear feed-through device shown in Figure 6 was added to the Stage 1 furnace. This allowed movement of the boron-containing boat in and out of the furnace hot zone for better control of the boron doping process. Also, it was discovered that when only the decomposition step was performed in the original Stage 1 furnace, boron was deposited on the fibers indicating that boron contamination from previous doping runs occurred on the furnace walls in the hot zone area. This issue required the development of a second Stage 1 process furnace dedicated to only the decomposition step without the presence of boron.

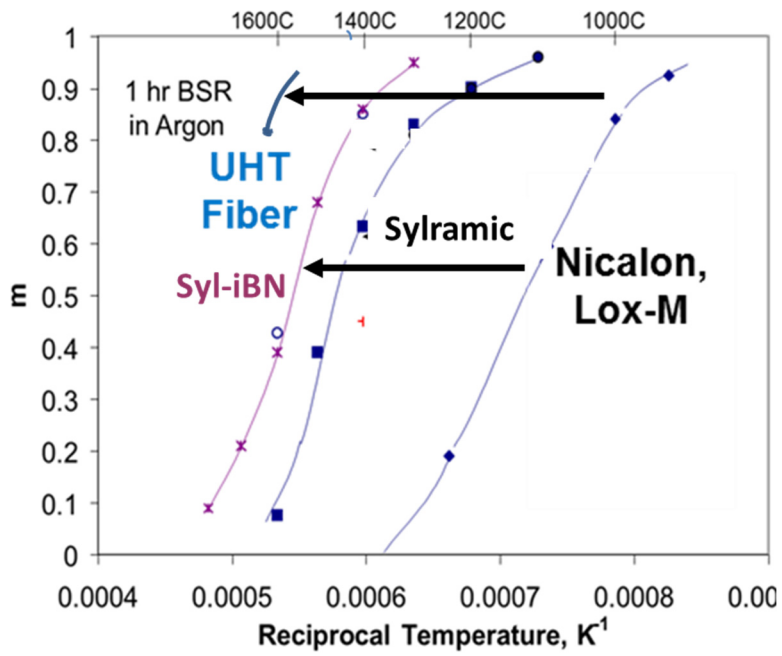


Figure 5.—BSR m-ratio data and goals for the various SiC fiber types of this study.



Figure 6.—New linear feed-through device for better control of doping process in the Stage 1 Furnace.

Results for Lox-M Precursor

During Phase I, primary emphasis was placed on conversion of the Lox-M precursor fiber since it is also the source of the Sylramic and SOA Sylramic-iBN SiC fibers. As shown in Figure 7, multiple trial runs at selected times and temperatures showed that core pores in sintered Lox-M precursor fiber originated during decomposition and remained after doping and sintering. Possible mechanisms include a high oxycarbide core content leaving pores too large to be sintered and/or residual carbon in the core, which is known as a sintering inhibitor for SiC materials. Thus Phase II efforts centered on a more detailed study of physical and chemical effects occurring during Lox-M decomposition process.

To determine the origin of the Lox-M core pores, the new decomposition furnace was used for detailed weight loss studies and physical property measurements on the Lox-M fibers after decomposition at 1360 °C. This low temperature was chosen to minimize the agglomeration of any excess carbon in the fiber core that occurs during higher temperature decomposition conditions (Ref. 7). Fiber physical measurements after decomposition of the Lox-M precursor fiber at 1360 °C are shown in Table 1. The length and diameter shrinkage as well as loss in tensile strength are to be expected due to the void creation by decomposition of the silicon-oxycarbide impurity phases in the original Lox-M fiber. However, the weight loss of ~25 percent for complete decomposition of our current Lox-M fibers indicates an impurity oxygen content of ~13 percent, which is larger than that indicated in the Dow Corning patent of 11 percent for their highest quality Sylramic fibers (Ref. 3).

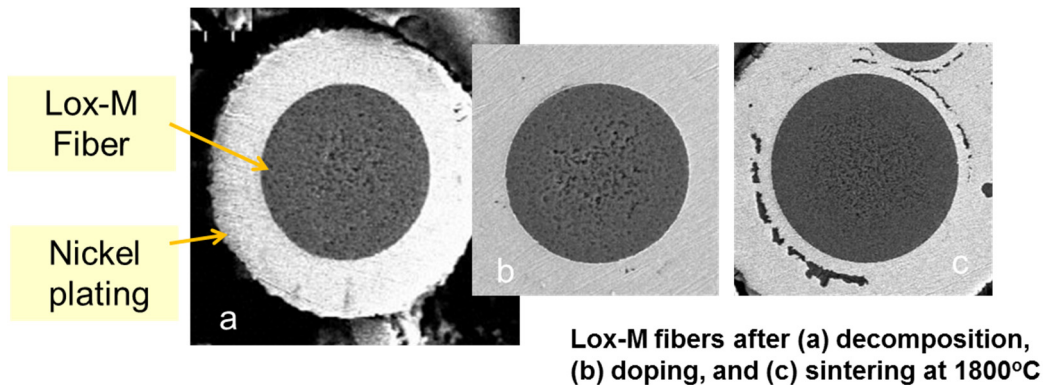


Figure 7.—Microstructures of treated Lox-M fibers showing retention of decomposition pores.

TABLE 1.—PHYSICAL MEASUREMENTS AFTER DECOMPOSITION OF THE LOX-M PRECURSOR FIBER AT 1360 °C

Decomp Hours	Weight Change	Length Change	Diam. Change	Tensile Strength
5	-21.7 %			
12 to 16	-24.7 %	- 3.2%	- ~2 %	~1.1 GPa

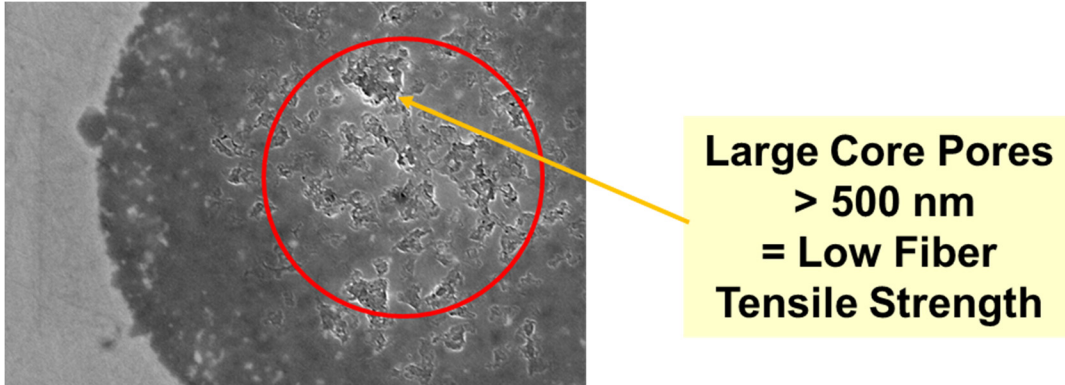


Figure 8.—Microstructure of fully decomposed Lox-M fibers showing large core pores that not only result in low fiber strength, but also are difficult to remove during the subsequent sintering process.

Also, after complete decomposition of our Lox-M fibers for 12 or greater hours at 1360 °C, microstructures of the decomposed fibers showed exceptionally large pores in the fiber cores as indicated in Figure 8. From these observations, it is currently concluded at the end of Phase II that: (1) the pores left by decomposition were probably too large to be eliminated during final sintering, an issue that goes back to amount of oxycarbide impurity introduced in original processing of our Lox-M fibers, and (2) for any future Lox-M studies, we need to use Lox-M fibers with lower oxygen and weight loss, which then should be expected to yield smaller and hopefully more sinterable core pores.

Results for Nicalon Precursor

In continued studies between Phase I and II, it was determined that under optimized process conditions, uniform microstructures with no obvious pores were observed in the treated Nicalon precursor fiber after sintering. These encouraging results, which are shown in Figure 9, resulted in a Phase II proposal and award. Thus in Phase II, those sintered Nicalon fibers with uniform microstructures were subjected to creep, tensile strength, and chemical analyses to check their quality for further processing into a UHT SiC fiber.

For creep analysis, the Bend Stress Relaxation (BSR) test was applied to single sintered Nicalon fibers with uniform microstructures. The m-ratio results shown in Figure 10 show that sintered Nicalon fibers with uniform microstructures display creep behavior equal to that of the Sylramic fibers, indicating in-house processes can successfully produce fibers with improved grain structure, which in turn should then be convertible to iBN-type fibers.

For tensile strength analysis, single sintered Nicalon fibers with uniform microstructures were then subject to room-temperature tests at 1-in. gauge length. Average strengths were found to be lower than those of the commercial Sylramic fibers (~1.5 vs ~3 GPa). As shown in Figure 11, reduced strength for the sintered Nicalon fiber is currently attributed to tiny kinks along its length as well as surface cracks, which have not been observed in the sintered Lox-M fibers. One possible mechanism for the kinks and cracks is that as the fiber sintered and contracted in volume, this volume change was non-uniform within the fiber causing local kinking and associated residual stresses that resulted in surface cracking. Trapped oxy-carbide phase in the fiber core may also have resulted in residual stresses. This kinking issue has also been noted in the literature for sintered SiC fibers and has been solved by applying a slight tension to the fibers during any shrinkage process (Ref. 8), a solution yet to be attempted.

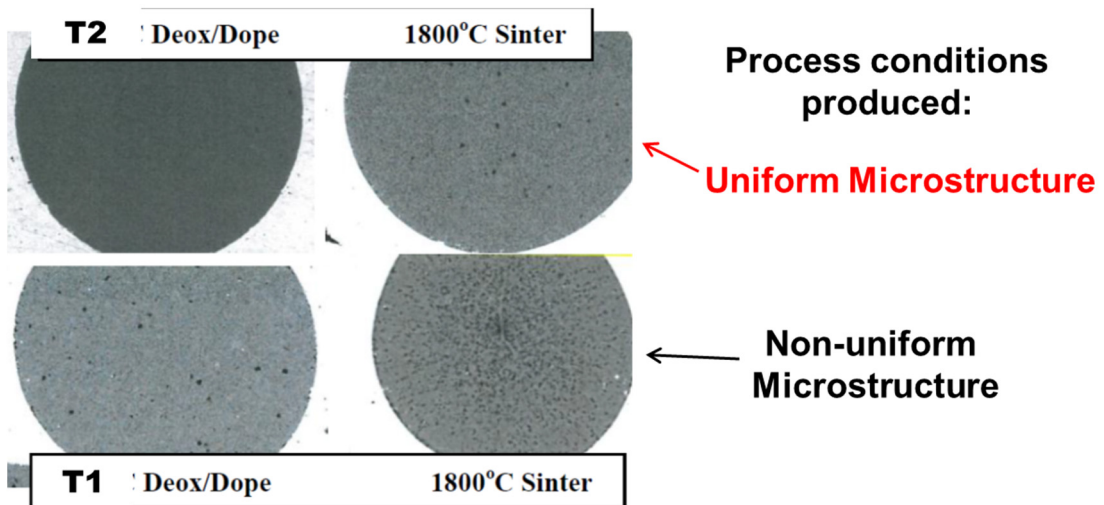


Figure 9.—Microstructure of treated and sintered Nicalon fibers showing that certain process conditions can produce near-uniformity and reduced void content across the fiber cross-section.

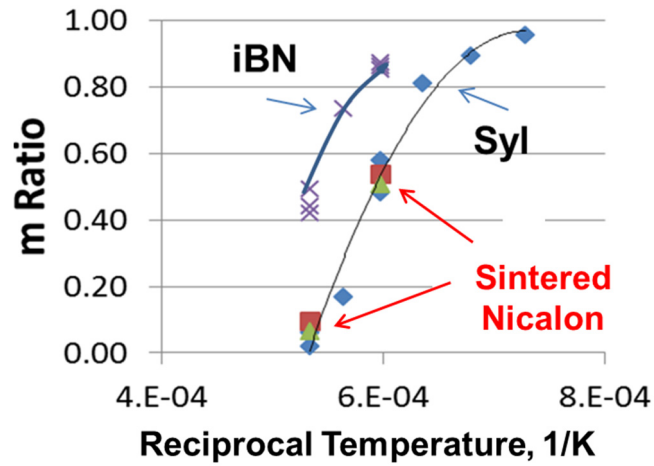


Figure 10.—BSR m-ratio data for treated and sintered Nicalon fibers with near-uniform microstructures showing creep resistance equivalent to the commercial Sylramic fiber.

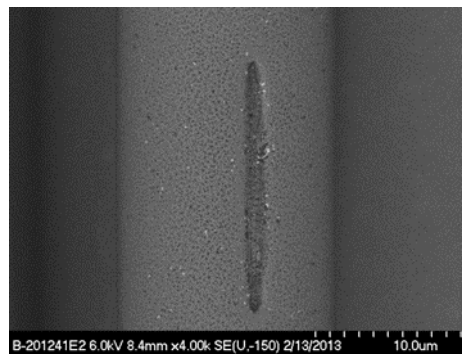


Figure 11.—Strength-limiting surface crack for treated and sintered Nicalon fiber with near-uniform microstructure.

To better understand any chemical sources for the fiber surface cracks, microprobe measurements were made at the end of Phase II in order to identify the chemical elements across the sintered Nicalon fiber diameter. The results are shown in Figure 12. From these observations, it is currently concluded that (1) the deox time-temperature conditions for the converted Nicalon fibers, even with apparently uniform microstructures, were not sufficient to completely remove the oxy-carbide phase in the fiber core, and (2) for any continued Nicalon precursor studies, we should be seeking improved process conditions that result in full decomposition and improved removal of carbon within the fiber core, as well as providing tension on the Nicalon fibers during all process stages during which fiber shrinkage occurs.

For future studies, in order to address the need for tension on the fibers during shrinkage, two new holders for precursor fiber tows have been developed (Fig. 13) where the tows are clamped or wound in grooves around graphite blocks, which because of their stable size, serve to apply tension to the fibers as they sinter and contract during the high-temperature process steps.

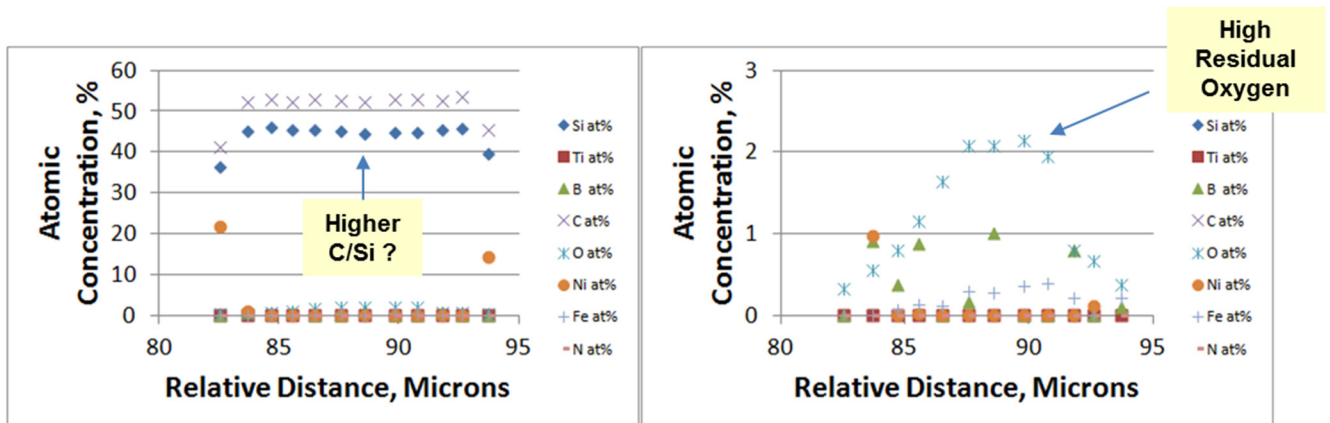


Figure 12.—Treated and sintered Nicalon fibers with near-uniform microstructures still retain the silicon-oxycarbide impurity in their core region.

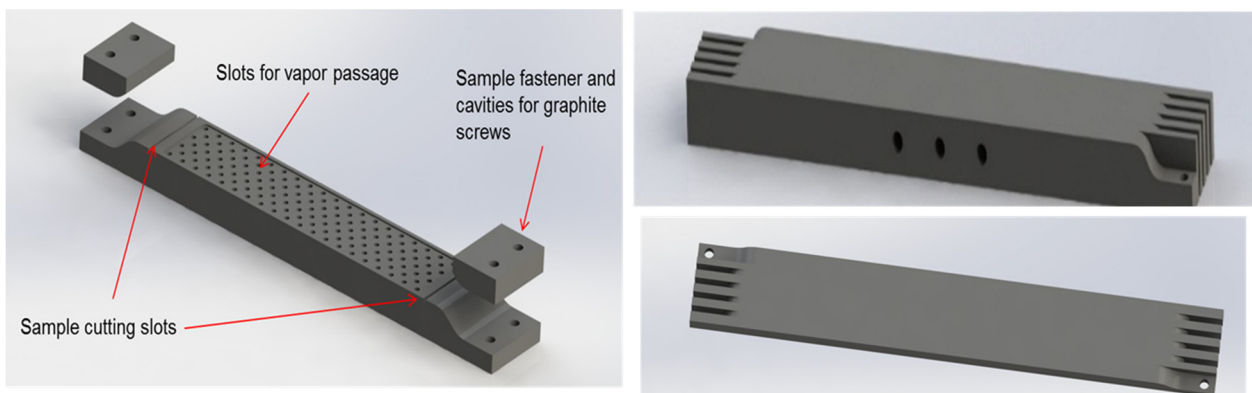


Figure 13.—New specimen holders for precursor fiber tows designed to apply tension to Nicalon precursors during fiber shrinkage stages.

Summary of Phase II Accomplishments

- Although time consuming, significant progress was made at NASA Glenn in developing the proper process equipment, safety permits, specimen preparation methods, characterization techniques, and property tests for producing and validating a UHT SiC fiber for SiC/SiC CMC.
- Task efforts verified that the Glenn UHT fiber process methods and facilities can indeed convert the impure microstructures of low-cost highly-available SiC fibers into microstructures equivalent to or better than those of the high-cost low-availability Sylramic SiC fiber. Using NASA's nitrogen processes, these fibers should be directly convertible into fibers with creep behavior similar or better than that of the current SOA Sylramic-iBN fiber.
- This result implies that as further studies increase the strength of the converted fibers, processes will be available within NASA and industry for producing fibers similar to the Sylramic-iBN fibers not only within tows, but more importantly within 2D and 3D complex-shaped preforms of CMC components, an important technical result not available today.
- Although complete success has not yet been achieved in completely eliminating issues in the converted fiber cores, lessons were learned, and feasible approaches for eliminating these issues will be studied under the NASA Transformational Tools and Technologies Project in order to meet one of its key technical challenges of a 2700 °F CMC turbine engine component.

Current TRL

Innovation has moved from basic principles (TRL1) to formulated concept (TRL2).

Applicable NASA Programs/Projects

In terms of NASA significance, the UHT fiber addresses aeronautics challenges within the NASA Transformational Tools and Technologies Project, such as minimally and un-cooled SiC/SiC propulsion components that will require temperatures on the order of 2700 °F. Similar goals also exist for the new Air Force multimillion dollar program aimed at developing 2700 °F SiC/SiC engine materials.

Publications and Patent Applications

When project is fully successful, patent applications are expected concerning the processing of SiC fibers with state-of-the-art thermal-structural capability and also concerning the low-cost processing of these fibers in the complex architectural preforms needed for aerospace components.

Awards and Honors Related to Seedling Research

To be determined

Seedling Related NASA Publication

J.A. DiCarlo: Modeling Creep of SiC Fibers and Its Effects on High Temperature SiC/SiC CMC. Proceedings of 38th Annual Conference on Composites, Materials, and Structures, January 2014, Cape Canaveral, Florida.

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