



**CREEP OF HI-NICALON™ S CERAMIC FIBER TOWS AT 900°C IN AIR AND IN
SILICIC ACID-SATURATED STEAM**

THESIS

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AFIT-ENY-MS-17-M-277

**DEPARTMENT OF THE AIR FORCE
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THESIS

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Master Sergeant, USAF

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Abstract

Advanced SiC/SiC ceramic matrix composites (CMCs) are being considered for demanding aerospace applications such as aircraft engine hot-section components. In these applications the composites will be subjected to cyclic and sustained loadings at elevated temperature in aggressive combustion environments. Current aircraft engines employ Nickel-based superalloys in applications such turbine blades, where the metallic alloys must perform at or near their operating temperature limits in highly corrosive environments. The SiC/SiC composites, which offer low density, high strength and fracture toughness at elevated temperatures could potentially replace Nickel-based superalloy in aircraft engine applications. However, before the SiC/SiC composites can be safely used in advanced aerospace applications their durability at elevated temperatures in service harsh environments must be assured. Therefore a thorough understanding of mechanical performance of SiC/SiC composites and their constituents in service environments is critical to design and life prediction of these materials. When composite is subjected to mechanical loading in combustion environment, surface matrix cracks form. Then steam (one of the main component of the service environment) enters the composite through matrix crack and reacts with the SiC matrix to leach Si and become saturated with Si(OH)_4 . The silicic acid-saturated steam travels into the composite interior and attacks the oxidation prone reinforcing SiC fibers. Hence thorough understanding of performance and durability of advanced SiC fibers at elevated temperatures in silicic acid-saturated steam is of paramount importance. This effort investigates creep of Hi-Nicalon™ S SiC fibers at 900°C in air and in Si(OH)_4 saturated steam. The fiber tows consisting of approximately 500 filaments with an average

diameter of 12 μm were subjected to creep tests at 900°C using a unique testing facility developed at AFIT, Creep stresses ranged from 3.5 to 1180 MPa in air and from 3.5 to 800 MPa in Si(OH)_4 saturated steam. Primary and secondary creep regimes were observed in all tests. Creep run-out defined as 100 h at creep stress was achieved at 736 MPa in air, but only at 3.5 MPa in Si(OH)_4 saturated steam. Creep rates in Si(OH)_4 saturated steam were approximately an order of magnitude higher than those in air. Post-test microstructural examination revealed passive oxidation of fibers tested in air or in steam, and showed no evidence of active oxidation.

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Ronald K. Mitchell 

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I. Introduction

Aerospace and thermal energy systems demand an ever increasing material capability to operate and sustain at higher temperatures and load levels. As aircraft are being designed to travel higher, faster, farther, it has become clear that structural materials must compensate for increasing requirements. Advanced ceramic matrix composites (CMCs) are key to fulfill this need. Adversity is the father of all ingenuity, a universal truth in scientific advancement. Composite materials are no different, the rapid development and use of composites began in the 1940s mainly due to military necessity, the expansion of polymer markets, and high strength applications needing a suitable replacement.

The first iteration of new materials came in the form of glass fiber reinforced polymers (GFRPs) in the 1940s. GFRPs presented a high strength material for possible military considerations. However, replacing a nosecone or helicopter rotor with these new materials posed a significant problem, brittle behavior. Also GFRPs suffered significantly in the area of quality control. Part to part, the material failed at different cycles/loads. This is attributed to flaws in the materials, such as a micro cracks on the surface/internal flaws, a problem not previously seen with metallic alloys to the extent displayed with this new material. The stress-to-failure varied significantly between identical components. The number of flaws and their sizes were different for each manufactured piece. The realm of fracture mechanics was forced to re-evaluate how this class of materials was treated and implemented. A significant milestone in the development of composite materials was the realization of scientists and engineers that if fibers could be placed in a matrix of a lower fracture strength material, they could arrest crack propagation (see **Figure 1**). The fiber added strength to the fragile polymer through their strong interfacial bonds.

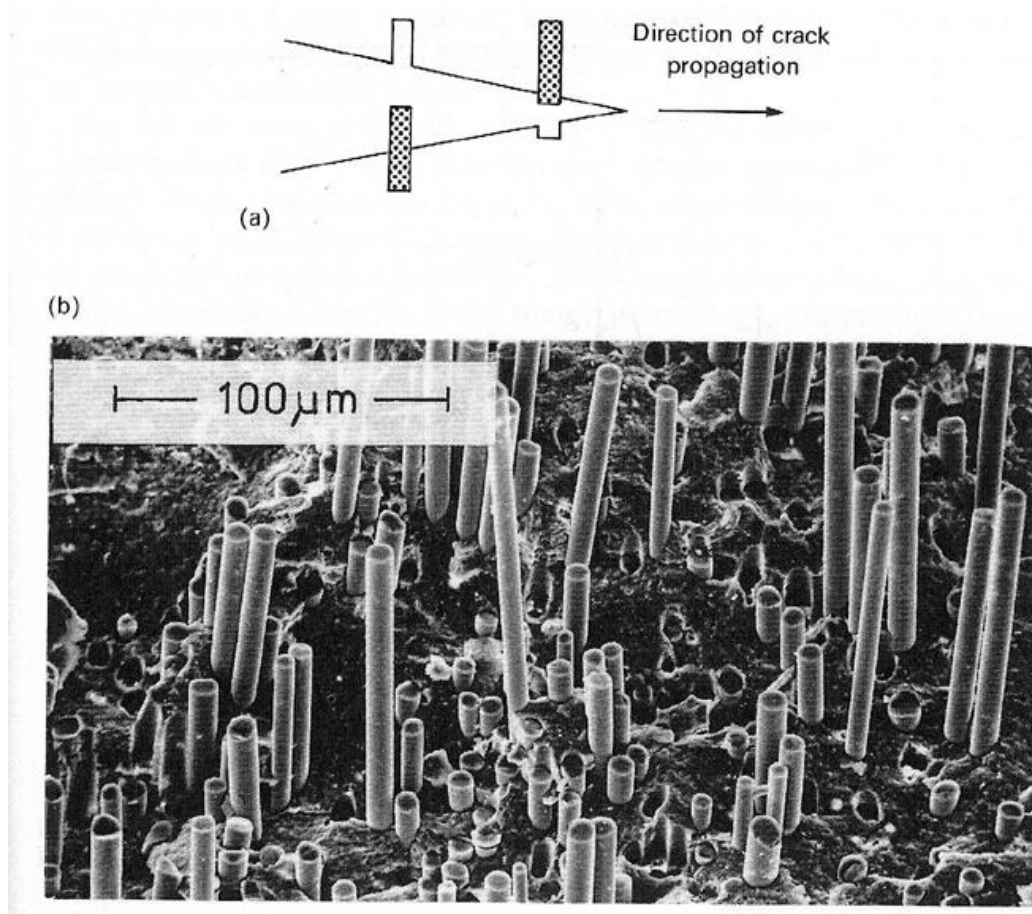


Figure 1. Composite crack propagation path. b) Fiber pullout: Silceram glass ceramic reinforced with SiC fibers [1]

The second iteration of composites occurred in the 1950s in France. A need arose for higher modulus fibers for new applications, i.e. space travel. Sputnik's launch in 1957 marked the beginning of the space race, thus driving the need to develop even stronger/lightweight materials that could break Earth's gravity and survive the high temperature re-entry of space vehicles. Carbon (graphite) fibers and Boron fibers were introduced as a new type of reinforcement to meet the growing demands. Boron fibers presented a stronger replacement than its Carbon counterparts, but manufacturing capabilities

and lower cost pushed Carbon to the forefront. One customer that was not as constrained by cost was the US military. Unfortunately, with all the possible applications of Boron fiber reinforced composites, the material never made a name for itself in industry. One exception is a material developed by DuPont called Kevlar. Initial efforts for applications failed until the new material was spun and formed into a weave. The resulting fibers/weaves were five times stronger than steel.

The third iteration, and the subject of this thesis, was the development of metal matrix composites (MMCs) and ceramic matrix composites (CMCs). CMCs came to fruition with the development of high temperature structural fibers, such as SiC. This was a key hurdle to overcome due to the high temperatures needed for sintering. These SiC fibers decreased the thermal coefficient of expansion, and when used as continuous fibers, increased thermal and strength properties at operating temperatures as well as provided significant creep resistance in the new composites. These SiC fibers merited further development, and continue to do so [1].

II. Background

2.1 Ceramic Matrix Composites

Ceramic matrix composites (CMCs) provide material characteristics very desirable for many engineering applications. They incorporate high strength, high plastic deformation resistance, and temperature/chemical resistance as well as increase the flaw tolerance in comparison to monolithic materials. These attributes make them an ideal replacement for many applications in propulsion systems or thermodynamic cycles. Although there are many types of CMCs, (i.e. oxide/non-oxide, continuous fiber/whisker fiber, etc), continuous fiber CMCs are of most importance for the aforementioned applications. Continuous fiber reinforcement increases crack resistance, or commonly referred to as fracture resistance. Properly designed fiber/matrix interfaces arrest/deflect cracks under normal loading conditions, thus preventing early failure. The fiber can still support the load sufficiently enough to categorize the failure as a “graceful” failure, a fibrous failure similar to that which occurs in wood [2].

The ceramic matrix binds the CMC together and transfers the load to the fibers. Its properties are established by the types of constitutive atoms, type of bonding (ionic or covalent), and its crystal structure. Oxide matrices are typically paired with oxide fibers and non-oxide matrices are paired with non-oxides. Typical types are C/C, C/SiC, SiC/SiC, and Ox/Ox. The properties of the matrix material, which may be nearly identical with respect to chemical composition, typically differ from the fiber properties usually differing by an order of magnitude [3]. The matrix must bind the composite together while delineating the fiber interface to ensure non brittle behavior. A compliant material is coated onto the fiber material, typically called an interphase material. Ideally, the interphase material has a

layered crystal structure that is parallel to the fibers. The interphase should be well bonded to the fiber, but not as thoroughly coupled to the matrix [4].

CMC constituent materials are typically very brittle. Even though the 2 components are brittle, CMCs exhibit excellent strength/toughness. They are damageable elastic materials which, when placed under load, micro cracking occurs initiating fiber/matrix (F/M) de-bonding. This de-bonding decreases stiffness and non-linear stress/strain is exhibited. This de-bonding is ideal behavior for “non-brittle” behavior, however, oxygen diffusion increases causing oxygen embrittlement [5]. This embrittlement is a type of stress corrosion cracking (SCC) which occurs at a higher rate in the region with the highest stress intensity, perpendicular to the applied load, and is commonly referred as environmentally assisted crack growth (EACG). CMCs should be chosen for material properties that are best suited for the temperature/pressure range, chemical resistance, oxidation resistance, and structural applications.

Discontinuous fiber reinforced CMCs (i.e. whisker, particulate nanocomposite) exhibit high yield strengths (YS) and ultimate tensile strengths (UTS) approaching 1000 MPa. Conversely, continuous fiber reinforced CMCs have approximately 75 MPa (YS) and 350 MPa UTS. However, continuous fibers are responsible for establishing a much higher strain to failure (STF) and thus, they have much higher fracture toughness. These fibers also establish the load carrying capability of the CMC (yield strength and ultimate strength).

Defects in CMCs are virtually impossible to eradicate, but when coupled with the material properties between the fibers/matrix, they create “matrix cracking strength”. Typically, the matrix cracking strength and YS of CMCs are one and the same. As the load increases in a CMC cracking initiates, the stress-strain relationship becomes nonlinear. This

fact, coupled with the lower YS/UTS, ensures that most continuous fiber SiC/SiC components are designed to be used well below their yield strength. For short interval applications the high STF property of these continuous fiber CMCs allow them to be used above their proportional limit, giving them exceptional resistance to thermal shock and ensures graceful failure [5].

2.2 Silicon Carbide

Silicon Carbide was discovered in ca. 1905 by Henri Moissan in samples excavated from Diablo Canyon, Arizona in the late 1890s. The aptly named, Moissanite, meteorite contained single SiC crystals with extraordinary isotopic characteristics. The nature of these primitive pre-solar grains is defined in terms of the ratios of $^{29}\text{Si}/^{28}\text{Si}$ and $^{30}\text{Si}/^{28}\text{Si}$. It is speculated that these SiC crystal rich meteorites were collected in the accretion discs as our solar system was formed. However, these isotopic ratios are not dominant in our sun indicating they were formed closer to the center of the Milky Way galaxy. Of significant note, the cubic- β polymorph showed dominant compared to the α -polymorph in contradiction of laboratory experiments, specifically at the high temperatures the crystals must have been subjected to during formation in early stars. β -SiC (3C-SiC) is formed at temperatures below 1700°C, whereas α -SiC (6H-SiC) is formed above 1700°C (see **Figure 2**). This anomaly is yet to be explained [6].

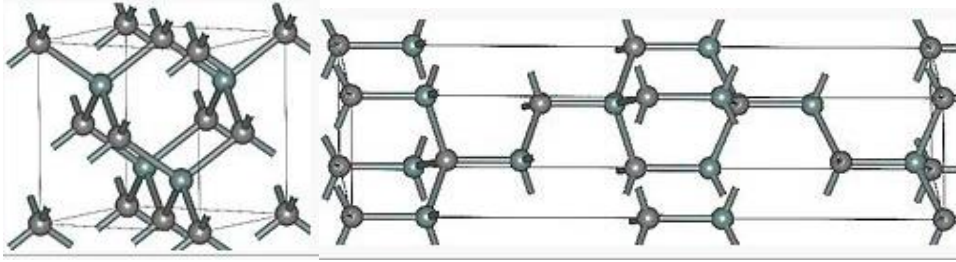


Figure 2. a) β –SiC (3C-SiC) zincblende crystal structure b) α –SiC (6H-SiC) hexagonal crystal structure

2.2.1 Silicon Carbide Based Ceramics

In SiC/SiC CMCs, β –SiC is the type typically used. The Carbon/Silicon atoms are tetrahedral bonded in the zinc-blende structure, containing very strong bonds. A monolithic ceramic SiC material (see **Figure 3**) of this type has high hardness and strength but suffers from low fracture toughness (extremely brittle) compared to other common engineering alloys. Typically, the fracture toughness, K_{1C} of monolithic ceramics do not exceed values of 5 MPa(m^{1/2}) (note: discontinuous fiber CMC (whisker fibers): 7-12 MPa(m^{1/2}), and continuous fiber CMC: 30 MPa(m^{1/2})) [5]. SiC exhibits increased resistance to acids, alkalis, and molten salts up to 800°C. SiC exhibits excellent oxidation resistance at temperatures exceeding 1600°C due to a protective oxidative layer (sacrificial Si to SiO₂). Near stoichiometric SiC (minimal impurities at grain boundaries), demonstrates sustained high temperature strength and chemical resistance [7].



Figure 3. SiC powder in refined/unprocessed state.

2.2.2 Silicon Carbide Fiber

One of the oldest types of SiC fiber manufacturing is chemical vapor deposition (CVD). As seen in **Figure 4** and **Figure 5** high temperature resistant material is selected, drawn into a fine diameter wire, and heated to the desired temperature for material deposition (typically Tungsten/ β -Carbon for SiC deposition). This process creates a large diameter fiber ($>75\ \mu\text{m}$) and is typically unsuitable for weaving [4].

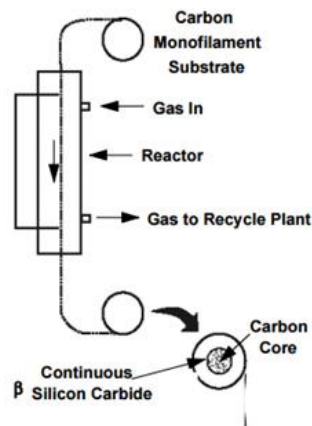


Figure 4. Chemical Vapor Deposition (CVD) process for manufacture of SiC fibers [5].

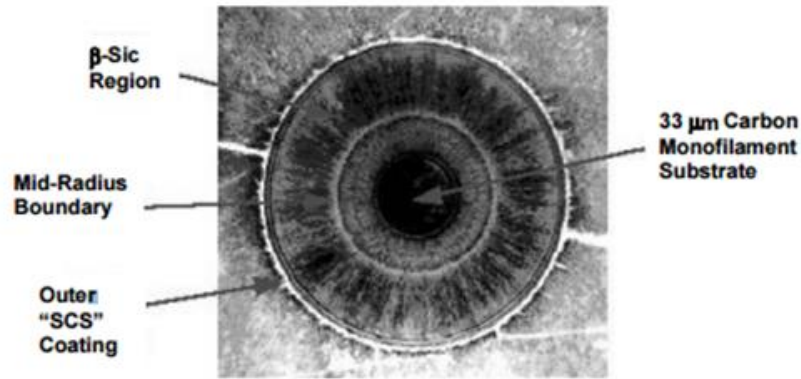


Figure 5. Cross sectional view of CVD processed SiC fiber [5].

This type of SiC fiber is called SCS, was first fabricated by Textron consists of stoichiometric SiC with a columnar grain structure which radiates outward from the core. The diameters of SCS can range from 79-140 μm [5].

Currently, most SiC fibers that are commercially available have diameters less than 20 μm . They are in multifilament bundles (fiber tows) and are created using pyrolysis of organosilicon polymers (see **Figure 6** and **Table 1**).

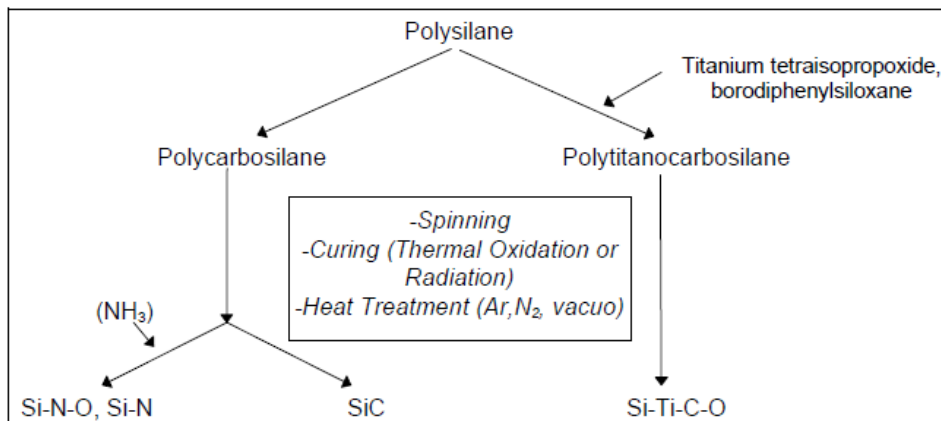


Figure 6. Generalized polymer pyrolysis fiber process diagram [5].

Table 1. Process for commercial and developmental polymer-derived ceramic fibers [4].

Company	Fiber	Spin Method	Cure Method	Ceramic Composition
<u>NCK a</u>	CG	melt	air oxidation	Si-C-O
	NICALON	melt	electron beam	SiC + C
	HI-NICALON S	melt	electron beam	SiC
<u>Ube b</u>	TYRANNO	melt	air oxidation	Si-C-O-Ti
		melt	electron beam	Si-C-Ti
	TYRANNO-Z	melt	air oxidation	Si-C-O-Zr
		melt	electron beam	Si-C-Zr
<u>MER/UMc</u>	—	solution dry	thermal + chemical	SiC
<u>3M/UF d</u>	—	solution dry	thermal + chemical	SiC + C or SiC
<u>DCC e</u>	SYLRAMIC	melt	air oxidation	SiC + TiB ₂
<u>Bayer f</u>	—	-NMe Me -N - Si -NMe -NMe H -NMe N - B N - Me chemical		Si-N-B-C-O or Si-N-B-O

2.3 Creep

Creep is a time-dependent deformation under an applied load usually occurring at high temperature (commonly referenced at temperatures above $0.5 \cdot T_m$), but can also occur at room temperature in certain materials. In CMCs creep is typically seen at a low strain-rate and intermediate stress loading condition [8]. As discussed in Section 1, CMCs represent a significant advance in aerospace and thermal applications. In a jet engine combustion

chamber, these materials would be subjected to high temp (up to 2000°C), long duration, and cyclic loading. Creep deformation ultimately concludes with a brittle rupture if steps are not taken to prevent catastrophic failure. As seen in **Figure 7** Creep is divided into three regimes: primary, secondary and tertiary.

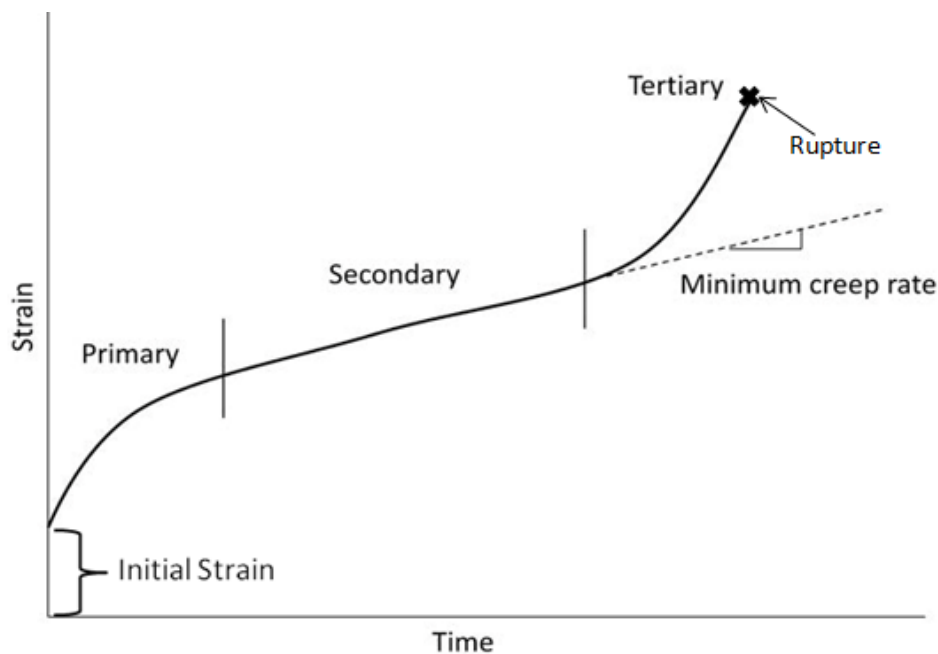


Figure 7. Three stages of creep deformation: Primary, Secondary and Tertiary.

Primary creep begins with an extremely high rate and slows with time. Secondary creep, the region of most interest for CMCs, has a uniform rate “quasi-steady-state regime” [9]. And finally, tertiary creep has an accelerated creep rate and ultimately terminates when the material breaks/ruptures. Tertiary creep in non-brittle materials is associated with both necking and formation of grain boundary voids. In ceramic materials, tertiary creep does not occur. As stated, secondary creep is of most interest in CMCs. Steady state creep at elevated temperatures is driven by multiple mechanisms as seen in **Table 2**.

A common formulation of steady state creep is derived from the Arrhenius equation [10].

$$\dot{\epsilon} = B \frac{D\mu b}{kT} \left(\frac{b}{d}\right)^p \left(\frac{\sigma}{\mu}\right)^n \quad (1)$$

where $\dot{\epsilon}$ is the strain rate, B is a constant, D is the diffusion coefficient, μ is the shear modulus, b is magnitude of the Burgers vector, k is the Boltzmann constant, T is the absolute temperature, d is the grain size diameter, σ is the applied tensile stress, p is the grain size power law exponent and n is the stress power law exponent. The diffusion coefficient D , is given by the expression:

$$D = D_o \exp\left(\frac{-Q}{RT}\right) \quad (2)$$

where D_o is a frequency factor, Q is the creep activation energy, and R is the universal gas constant [11]. As seen in **Equation (3)**, a power law equation is a very common approach to determine creep mechanism. Experimental creep data can be used to determine values stress exponent, n [12] (see **Table 2**).

$$\dot{\epsilon} = A\sigma^n \quad (3)$$

Table 2. Creep mechanisms in fine-grained polycrystalline ceramics [13].

<i>n</i> stress exponent	<i>p</i> grain size exponent	Mechanism
1	2	Diffusional creep through lattice (Nabarro-Herring creep)
1	3	Diffusional creep along grain boundaries (Coble creep)
2	1	Grain boundary sliding and interface- reaction controlled creep
4	3	Grain boundary sliding and cavity growth
3-5	0	Dislocation creep: 3 –glide controlled, 5 –climb controlled
>5		Cavity growth controlled creep

III. Material and Test Specimen

3.1 Material

This research studied Hi-Nicalon™ S fiber tows produced by Nippon Carbon Co Ltd of Tokyo, Japan (see **Figure 8**). Hi-Nicalon™ S is a near stoichiometric β -SiC fiber produced via near-oxygen free utilizing de-carbonization pyrolysis and electron beam curing. It has significantly higher modulus, better creep resistance, and better oxidation resistance than previous generations of Hi- Nicalon™ fibers [14].



Figure 8. Hi-Nicalon™ S fiber tow on a spool [14]

The fiber tows consisted of approximately 500 filaments with an average diameter of $12\ \mu\text{m}$ [15]. The cross sectional area of the fiber tow calculated as $5.658 \times 10^{-8}\ \text{m}^2$ was used in all engineering stress calculations. Mechanical strength properties reported for small-

diameter SiC fibers, such as Hi Nicalon Type STM are typically obtained from single fiber tests and are usually higher than the values obtained for multi-filament fiber tows. Notably, the average strength reported for a single Hi NicalonTM Type S fiber is 1900 MPa, while the average strength reported for a Hi NicalonTM Type S fiber tow is 1450 MPa [16]. However, CMCs are reinforced with woven or braided fiber tows rather than single fibers. Due to the weaving process, fibers are tightly packed and 90% of neighboring fibers contact one another. These fiber tows are the fundamental unit of any CMC. Hence mechanical testing of fiber tows offer better insight into performance of the composite than testing of single filaments as fiber tows. Typical properties of Hi-NicalonTM S fiber tow, as provided by the manufacturer, are summarized in **Table 3**.

Table 3. Typical properties of Hi-NicalonTM S fiber tow; data reproduced from NGS Advanced Fibers Co., Ltd. [17]

Number of filaments	500
Filament diameter (μm)	12
Product Form	Tow
Sizing Agent	PVA
Linear density, tex (g/km)	198
Oxygen content (wt%)	0.8
Modulus of Elasticity (GPa)	380

3.2 Test Specimen

Test specimens were prepared utilizing the three tab method developed by Steffens [18]. This method was also used by Robertson [19], Sprinkle [11], and Piper [20]. A detailed step-by-step description of the specimen preparation process is given in Appendix A1 of Steffens [19].

Each fiber was fastened utilizing three fiberglass tabs (primary, secondary, and tertiary). The primary tab is 1.0 in x 1.5 in (0.0254 m x 0.0381 m), the secondary tab is 1.0 in x 1.0 in (0.0254 m x 0.0254 m), and the tertiary tab is 0.75 in x 0.75 in (0.0191 m x 0.0191 m) (see **Figure 9**).

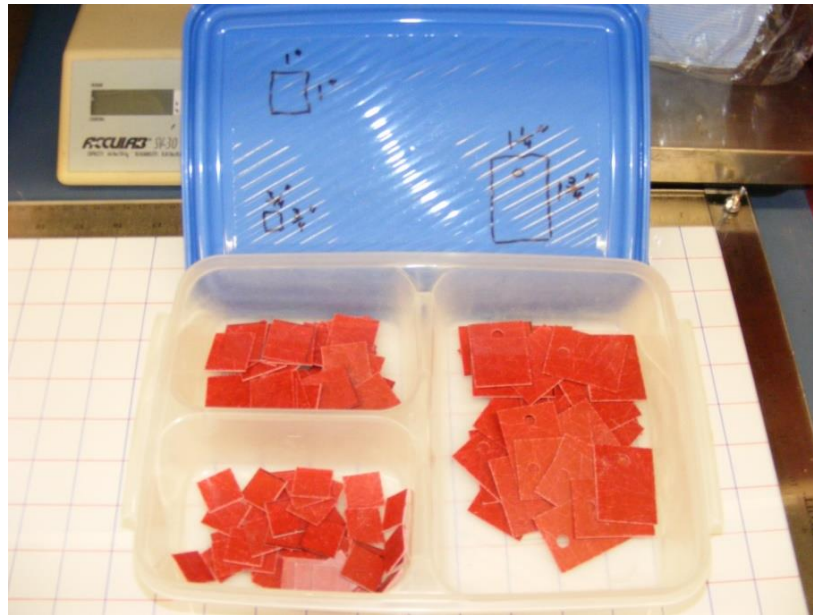


Figure 9. Fiberglass tabs used for fiber tow testing.

A hole was punched along the centerline of each primary tab. The hole is used to suspend the specimens from the hook fixture for tensile creep testing. The primary and

secondary tabs were sanded to prevent sharp edges from impinging on the samples during handling and testing. A schematic of the three-tab arrangement is shown in **Figure 10**.

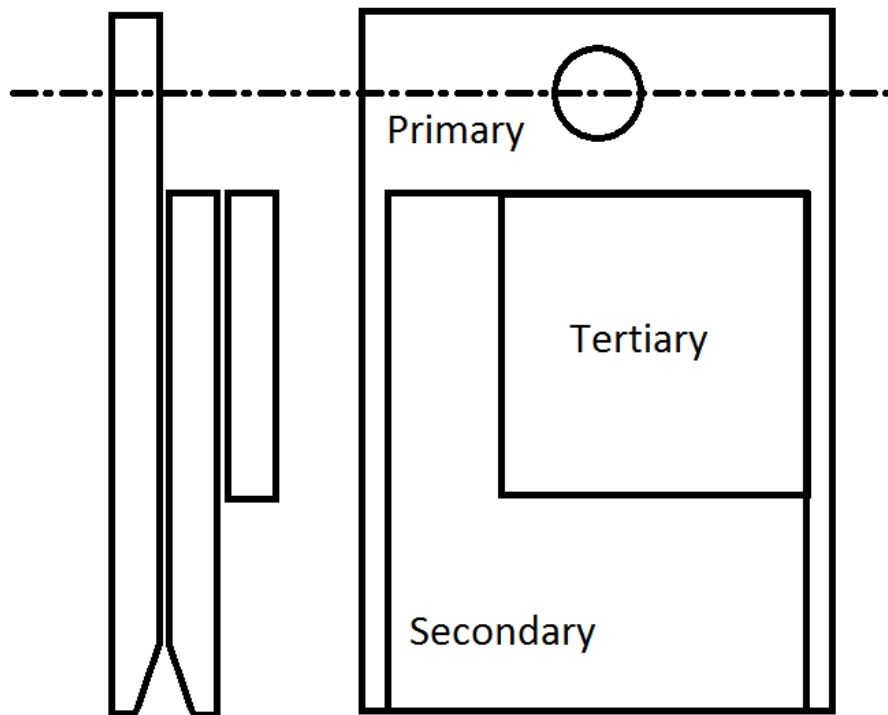


Figure 10. Tab layout for fiber tow testing.

For test specimen preparation the primary tabs were secured on the grid of the sample preparation board (see **Figure 11**) with a 7 in (0.178 m) gap between the tabs along one of the gridlines and another gridline bisecting the hole punched in the primary tab. A length of fiber tow was cut from the spool, aligned along the gridline and attached to the primary tabs with tape (see **Figure 12**). The secondary tab was attached using a two part epoxy. The epoxy used required a 5-min cure for handling and a recommended 24 hour cure for working. After 5 minutes, the excess fiber was folded over the secondary tab and the tertiary tab was

then applied using epoxy. After 5 minutes, the excess fiber protruding from the tertiary tab was removed using a razor blade (see **Figure 13**).

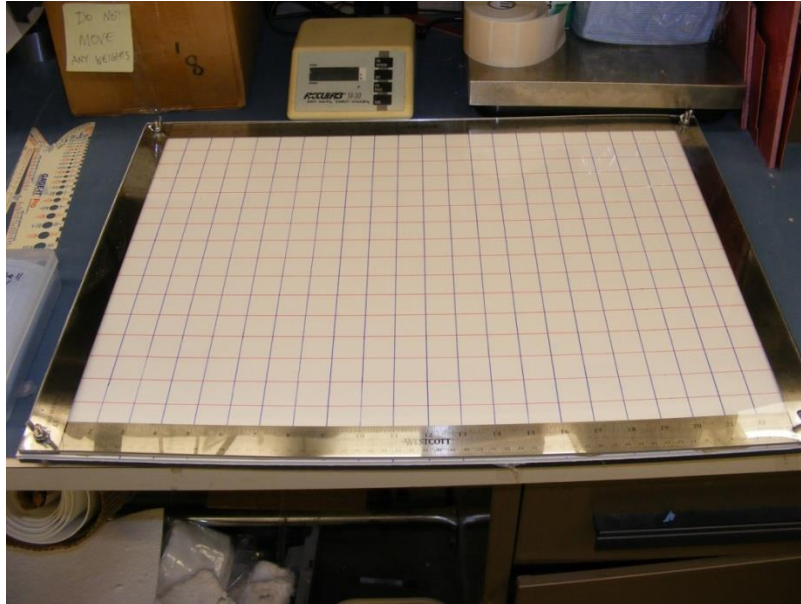


Figure 11. Sample preparation board.

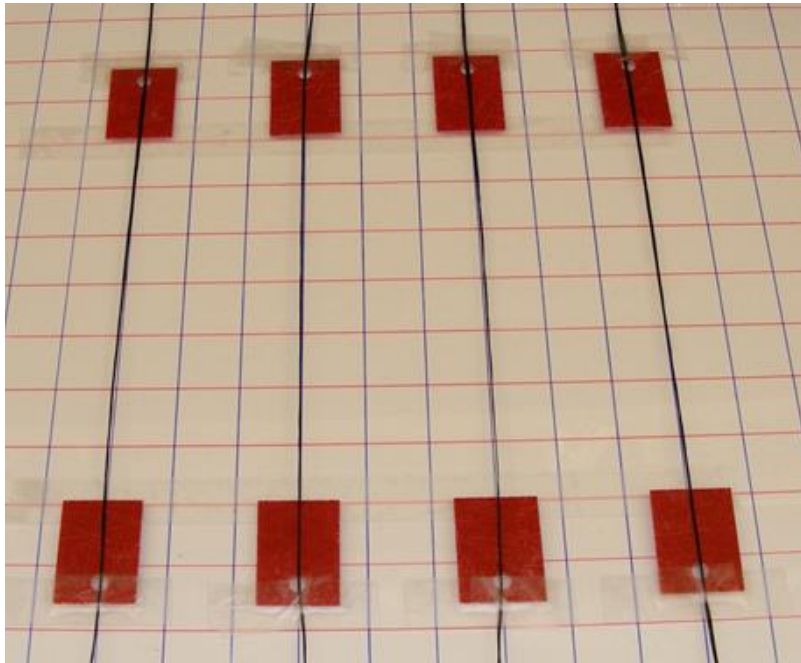


Figure 12. Placing primary tab and attaching fiber tow.

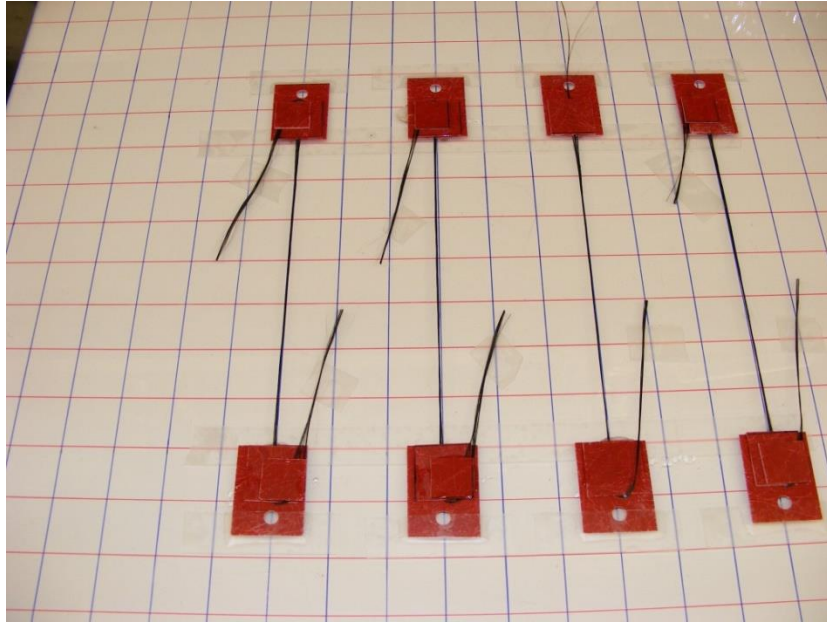


Figure 13. Test specimen preparation process.

IV. Experimental Arrangements and Procedures

4.1 Experimental Facility

The tests for this research were conducted at AFIT in the Mechanics of Advanced Aerospace Materials Laboratory, Department of Aeronautics and Astronautics. The baseline fiber-tow testing facility was originally built by Armani [13] for testing of oxide fiber tows at elevated temperature in air and in steam environments. Steffens [21] and Shillig [22] modified the experimental setup for testing of SiC fiber tows. Shillig discovered that steam environment caused severe degradation of SiC fiber tows during creep tests. Shillig reported that the bottom sections of SiC fiber tows tested at elevated temperature in steam exhibited signs of active oxidation, while the top sections of the fiber tow exhibited signs of passive oxidation. Recognizing that steam entered through a feeding tube located at the bottom of the test chamber, it was concluded that steam leached Si from the SiC fibers as it traveled upwards through the test chamber and became saturated with silicic acid ($\text{Si}[\text{OH}]_4$) as it reached the top of the test chamber. Steam became chemically altered as it traveled upwards along the fiber tow causing the oxidation mechanism to change from active oxidation in the bottom section of the tow to passive oxidation in the top section of the fiber tow. Steam entering the test chamber had to be saturated with silicic acid in order to produce consistent oxidation of the fiber tow specimen.

Subsequently, Robertson [19] and Sprinkle [11] modified the fiber tow testing facility to deliver silicic acid-saturated steam to the test chamber. Furthermore, the modified facility permitted heating steam to test temperature prior to entering the test chamber. It should be noted that testing SiC fiber tows at elevated temperature in silicic acid-saturated steam effectively mimics the conditions imposed on the SiC fibers reinforcing SiC/SiC

composite. When a SiC/SiC composite is subjected to mechanical loading in steam, fiber-bridged matrix cracks form on the surface of the composite. If these cracks are exposed to steam environment, steam enters through the cracks and reacts the SiC matrix, leaching Si from the matrix to become saturated with $\text{Si}[\text{OH}]_4$. The silicic acid-saturated steam then travels into the interior of the composite to attack the oxidation prone SiC fibers. Robertson [24] successfully used the modified experimental facility to test Hi Nicalon™ S fiber tows in creep at 800 °C in air and in silicic acid-saturated steam. Piper [20] explored creep performance of Hi Nicalon™ S fiber tows at 700°C in air and in silicic acid-saturated steam. In this research, we aim to investigate creep performance of Hi Nicalon™ S fiber tows at 900 °C in air and in silicic acid-saturated steam.

A detailed description of the modified test facility is given by Roberson [24]. A brief summary of the fiber tow testing facility is provided below. All tests employed an alumina susceptor (tube with end caps), which fits inside the furnace. The specimen test section is located inside the susceptor, with the ends of the specimen passing through slots in the susceptor (see Fig. 16). The use of alumina susceptor for testing in air provides for a more uniform temperature distribution along the test specimen. When testing in steam, silicic acid-saturated steam is introduced into the susceptor (through a feeding tube near the bottom of the susceptor) in a continuous stream with a slightly positive pressure, expelling the dry air and creating a near 100% silicic acid-saturated steam environment inside the susceptor.

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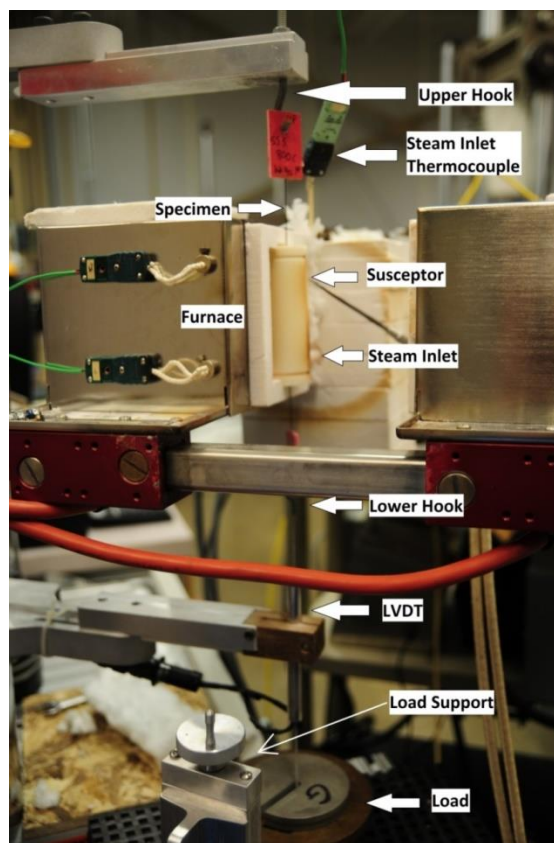


Figure 14. Fiber tow specimen mounted in the creep testing rig. Reproduced from Robertson [19].

A high resolution linear variable differential transformer (LVDT) was used to measure the displacement of the specimen throughout the test. Temperature profiles were measured throughout the length of the furnace with a K-type thermocouple utilizing a hydraulic ram actuator and MTS digital controller. Using the calculated temperature profiles, an effective gauge length of the fiber tow specimen can be calculated. Using the effective lengths, engineering strain can be calculated from the recorded displacement of the LVDT. Technique for determining temperature profiles and methods for calculating strain from displacement measurements are discussed in sections 4.2 and 4.3 below.

For tests in silicic acid-saturated steam, a steam generator manufactured by Micropyretics Heaters International (MHI), model HGA-H was used. Previous research conducted by Armani [13], Steffens [21], Shillig [22], Robertson [19], and Piper [20] utilized both HGA-H and HGA-S steam generators. The HGA-S model is identical to the HGA-H in terms of internal components and function, as well as additional thermocouples for temperature readout and control.

A peristaltic pump, Cole Parmer ® model 7518-10, was used to deliver 16-16.6 ml of de-ionized water to the steam generator. As flow rates above or below the prescribed design limits could cause damage to the steam generator, an alumina tube orthogonal to the susceptor steam inlet tube was used to regulate steam flow into the test chamber to prevent damage to the SiC fiber. The de-ionized water was supplied from a 50 gal reservoir.



Figure 15. MHI HGA-S-CX1300 heater (a) and controller (b) [23].

After leaving the HGA-H/S steam generator, the steam entered into an alumina tube that contained sacrificial silica in order to saturate the steam with silicic acid (see **Figure 17** and **Figure 18**). The alumina tube was placed inside two MHI CX1300 heaters. (see **Figure 15(a)**), The MHI CX1300 heaters equipped with heating coils manufactured by I Squared R Element Co. and encased in RATH KVS 174/400 insulation. The two CX1300 heaters were controlled by an IBPAN controller shown in **Figure 15(b)**. The output of the IBPAN controller was sent to a transformer for the appropriate voltage adjustment before powering the CX1300 heaters. The CX1300 heaters were controlled by a variable rheostat. As the steam moves through the alumina tube filled with silica wool, it becomes saturated with silicic acid and is heated to the desired test temperature. Then the saturated steam heated to the test temperature enters the test chamber. The overall experimental facility is shown in Figure 16.

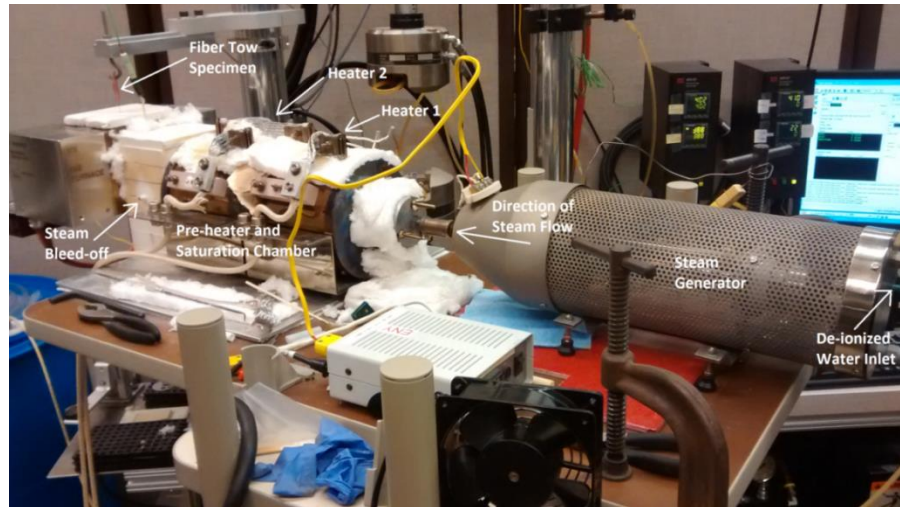


Figure 16. Creep test facility configured for saturated steam tests. Reproduced from Robertson [19]

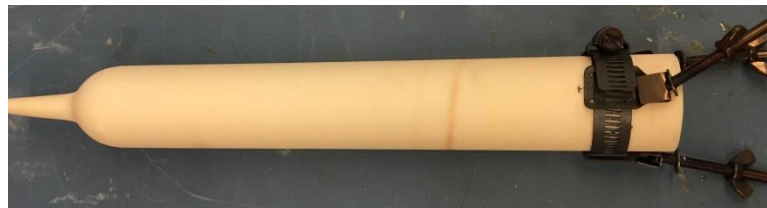


Figure 17. Alumina tube used for steam saturation with silica wool. Reproduced from Piper [20].

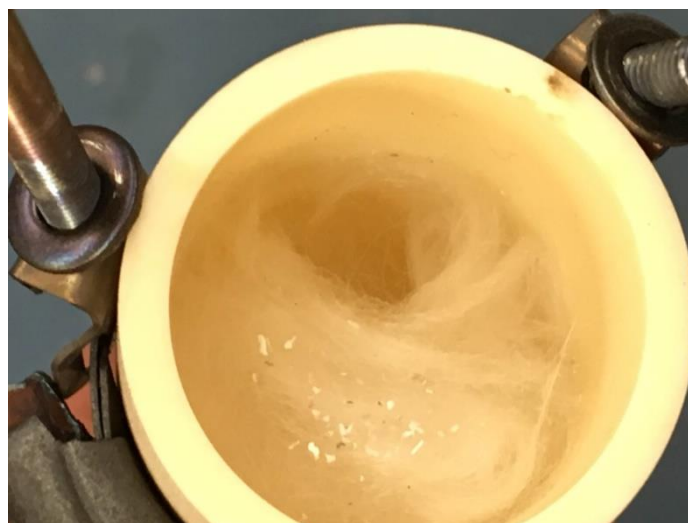


Figure 18. Silica wool inserted into the tube. Reproduced from Piper [20].

4.2 Temperature Profiles

Temperature profiles of the test chamber were taken to validate the test conditions and determine the effective length, L_{eff} , of the specimen. The determined effective length was used to calculate the engineering strain from the LVDT displacement as described in Section 4.3 below. Using the ram actuator of the MTS machine and the MTS controller software, a rigid 10 in K-type thermocouple (accuracy of $\pm 3^\circ\text{C}$) was moved along the 100-mm length of the succesor. Temperature readings were taken along the centerline of the furnace at one mm increments and recorded in a data file using the MTS controller software.

The temperature profiles obtained in air and in saturated steam at 900°C are shown in **Figure 19** and **Figure 20**. Note that the zero position corresponds to the midpoint of the test chamber. Position of +60mm corresponds to the top of the test chamber and position of -60mm to the bottom of the test chamber.

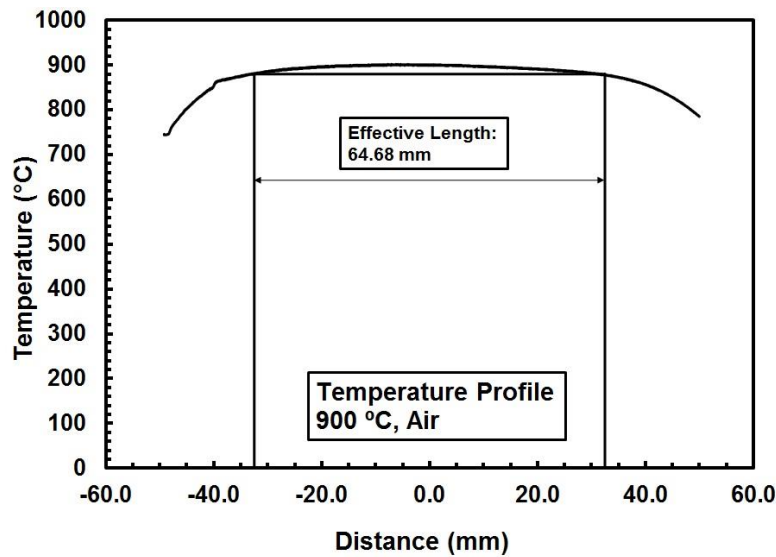


Figure 19. Temperature profile obtained in air at 900°C .

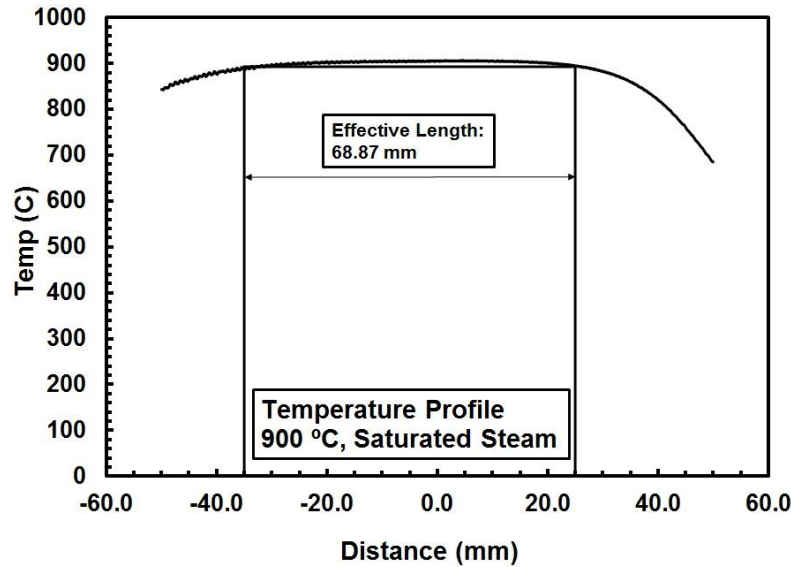


Figure 20. Temperature profile obtained in saturated steam at 900°C.

4.3 Strain Measurement

As seen in **Figure 14**, tensile creep tests were performed using a dead-weight creep rig. The elongation of the fiber tow specimen was measured with an LVDT (Schaevitz M12-30) connected to the bottom tab of the fiber tow specimen. The rod extending from the bottom of the LVDT core held the dead weight. An MTS FlexTest 40 digital controller was used to record the displacement data.

The nature of the fiber tow test specimens does not permit the use of direct contact strain measurement using extensometry. Therefore, indirect methods must be used to determine the strain in the fiber tow specimen. The elongation of the fiber tow specimen was measured with an LVDT (Schaevitz M12-30) connected to the bottom tab of the fiber tow specimen. An MTS FlexTest 40 digital controller was used to record the displacement (fiber elongation) data. Strain was calculated from the specimen elongation measurements using published methods [24], [25], [26]. Because the cold grip method was used, specimen

elongation was measured outside the furnace. Thus the total recorded specimen elongation was the sum of contributions from parts of the specimen located in different temperature zones: the hot zone (at uniform test temperature of 900°C), the temperature gradient zone and the cold zone. Temperature profiles measured for the furnace were employed to determine the effective gauge lengths of 64.68 mm at 900°C in air and 68.87 mm at 900°C in silicic acid-saturated steam, which were used to calculate creep strain and strain rate in the hot zone. This method was successfully used to determine strain and strain rates from displacement data obtained in elevated-temperature creep tests of oxide fiber tows [13] and SiC fiber tows [19], [11], [20]. The detailed description of this method to determine strain and strain rate from displacement measurement is given elsewhere [13]. A brief description is offered below.

Consider a fiber tow test specimen of length 2L. Taking the midpoint of the specimen gauge length as zero, creep is considered to occur over the length from -L to L. The creep strain and creep strain rate can be calculated as:

$$\varepsilon_m = \frac{\Delta l}{2L} = \int_0^t \dot{\varepsilon}_m dt \quad (4)$$

$$\dot{\varepsilon}_m = \frac{\text{measured extension rate}}{2L} = \frac{1}{2L} \int_{-L}^L \dot{\varepsilon} dl \quad (5)$$

Note that the total measured strain and strain rate calculated with **Equation (4)** and **Equation (5)** account for the variations in strain and strain rate along the length, 2L, of the specimen subjected to a temperature profile. Intuitively, the amount of strain will be greatest

at the hottest section. The strain and strain rates at the desired test temperature at the center of the furnace are denoted by the subscript 0 . The strain at the center of the furnace is calculated as the time integral of the strain rate at the center of the furnace and also described as the overall change in length of the specimen, Δl , divided by a hypothetical length, $(2L)_{eff}$, the effective gauge length. The effective gauge length is described as the gauge length obtained under the peak temperature and zero strain is achieved under the lower temperature. The strain at the desired maximum temperature can be calculated as:

$$\varepsilon_0 = \int_0^t \dot{\varepsilon}_0 dt = \frac{\Delta l}{(2L)_{eff}} \quad (6)$$

The strain rate at the maximum temperature at the midpoint of the furnace can be expressed in terms of the effective gauge length as:

$$\dot{\varepsilon}_0 = \frac{\text{measured extension rate}}{(2L)_{eff}} = \frac{1}{(2L)_{eff}} \int_{-L}^L \dot{\varepsilon} dl \quad (7)$$

The ratio of **Equation (5)** to **Equation (7)** can be written as:

$$\frac{\dot{\varepsilon}_m}{\dot{\varepsilon}_0} = \frac{\Delta l}{(2L)_{eff}} \quad (8)$$

Stress is constant in dead-weight creep testing and temperature can be considered as a function of location along the specimen according to the temperature profile. Applying these variables to the general power creep law yields:

$$\dot{\epsilon} = A\sigma^n \exp\left(\frac{-Q}{RT(l)}\right) \quad (9)$$

Combining **Equation (5)**, **Equation (8)** and **Equation (9)**, the ratio can be expressed as measured strain rate to actual strain rate as a function of temperature only:

$$\frac{\dot{\epsilon}_m}{\dot{\epsilon}_0} = \frac{1}{2L} \int_{-L}^L \exp\left\{\frac{-Q}{R}\left(\frac{1}{T(l)} - \frac{1}{T_0}\right)\right\} dl \quad (10)$$

This ratio can be expressed as a numerical summation of increments of length, h, where $L=hk$ and k is an integer. The ratio of measured to actual strain rate becomes:

$$\frac{\dot{\epsilon}_m}{\dot{\epsilon}_0} = \frac{1}{2k} \sum_{i=-k}^k \exp\left\{\frac{-Q}{R}\left(\frac{1}{T(l)} - \frac{1}{T_0}\right)\right\} \quad (11)$$

The effective gauge length is now calculated as:

:

$$(2L)_{eff} = 2L \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_0}\right) \quad (12)$$

The effective gauge length can now be used to determine the strain and strain rate of the fiber tow specimen using the displacement calculations from the LVDT. This approach, along with the temperature profiles determined in Section 4.2 and creep activation energy of 177 kJ/mol reported for Hi-Nicalon™ S [27] was used to calculate effective gauge lengths in air and in steam at 900°C. The calculated effective lengths in air and in steam are shown in Table 4.

Table 4. Effective gauge lengths of Hi-Nicalon™ S fibers at 900C

	Air	Saturated Steam
Effective Length (mm)	64.68	68.88

This approach differs from that used by Hammond [28] and Yun et al. [29] in that they assumed that the majority of creep deformation occurred only within the furnace hot zone and therefore used the flat portion of the temperature profile to determine the gauge length. This approach is both subjective because it relies on human determination of the flat zone and is dependent of the flatness of the temperature profile and the sharpness at which the temperature drops off at the edges of the hot zone. Alternatively, this method is somewhat more independent of the shape of the temperature profile.

4.4 Experimental Procedures for Tensile Creep Testing in Air and in Silicic Acid–Saturated Steam

As discussed in Section 4.1 above, the test facility utilizes a steam generation system to introduce silicic acid saturated steam into the susceptor containing the test specimen. For detailed procedures for specimen mounting and creep testing the reader is referred to Appendix A2 of Steffens [21] and in Appendix A of Piper [20].

V. Results and Discussion

5.1 Creep of Hi-Nicalon™ S Fiber Tows at 900°C

Hi-Nicalon™ S fiber tows were subjected to tensile creep tests at 900°C in dry air and in silicic acid-saturated steam. Creep run-out was set to 100h. Creep-rupture test results produced in this work are summarized in **Table 5** below. Creep strain accumulation, steady-state creep rate, and rupture time are shown for each creep stress level and environment.

Table 5. Summary of tensile creep test results for Hi-Nicalon™ S fiber tows at 900°C in laboratory air and in silicic acid-saturated steam.

Specimen ID	Test Environment	Creep Stress (MPa)	Creep Lifetime (h)	Steady-State Creep Rate (s^{-1})	Creep Strain (%)
S1	Steam	3.5	100†	9.0 E -10	0.066
S16	Steam	497	63.84	2.0 E -9	0.140
S13	Steam	600	55.21	5.0 E -9	0.303
S11	Steam	736	21.5	8.0 E -8	0.260
S3	Steam	800	0.07	8.0 E -6	0.250
A1	Air	3.5	100†	9.0 E -10	0.078
A3	Air	736	100†	5.0 E -9	0.033
A2	Air	802	2.94	2.0E -8	0.048
A5	Air	1180	0.117	9.0 E -5	0.240

† Creep run-out defined as 100 h at creep stress. Failure of specimen did not occur when the test was terminated

We note that interpreting results obtained in creep tests of fiber tows is not a trivial endeavor. Numerous assumptions are made in order to facilitate the analysis of creep test data. To facilitate the calculation of creep stress (load) levels we assume that each fiber tow has the same number of intact fibers and that each fiber has the same cross sectional area. While this assumption is not unreasonable, it does not always accurately represent the realistic fiber tow. The silicic acid-saturated steam generated and transferred through the

steam generator train is assumed to be contaminate free, although it is reasonable to expect some transference of particulate contaminants from the Alumina tube. Additionally, in elevated temperature tests oxidation of the fibers in the tow also takes place, causing the changes in fiber cross-section. A constant cross-sectional area of the fiber tow is used in stress calculations, yet the cross-sectional area is unlikely to remain constant throughout the entire test. Many fibers maintain close proximity to one another inside the fiber tow, a uniform contact area is assumed. Finally, it is difficult to determine whether the progressive strain accumulation with time observed in the creep experiment is indeed due to creep deformation of the fibers. It is possible that individual fibers (or small groups of fibers) fail progressively during the creep test. The load is then transferred to the remaining fibers causing the stress carried by the intact fibers to increase during the test. In this case the increasing strain is not due to creep deformation but rather due to continuously increasing stress.

Representative creep strain vs. time curves obtained at 900°C in air (**Figure 21**, **Figure 22**) and in saturated steam (**Figure 23**, **Figure 24**) are shown in Figures x and y, respectively. Creep curves produced in all tests exhibit primary and secondary creep regimes, but no tertiary creep. At 900°C primary creep transitions into secondary creep fairly early in creep life; primary creep persists during the first 8-10 h of the creep test.

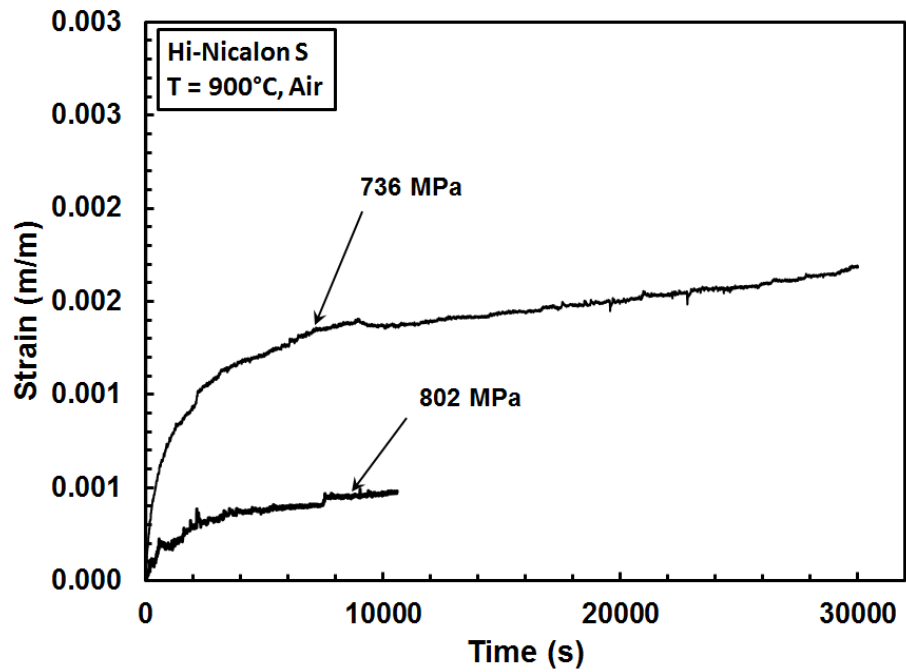


Figure 21. Creep strain vs. time for Hi-Nicalon™ S fiber tows at 900°C in air.

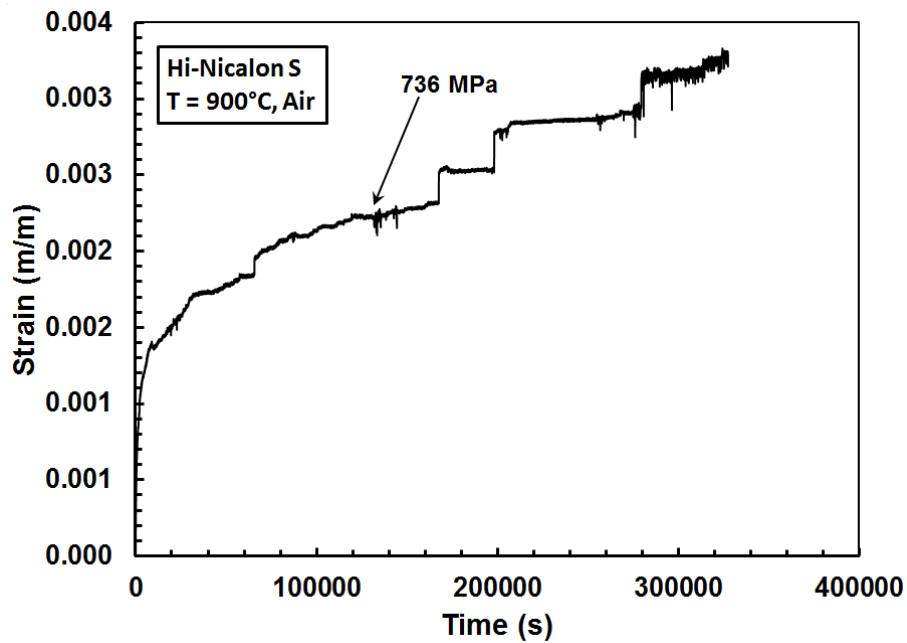


Figure 22. Creep strain vs. time for Hi-Nicalon™ S fiber tows at 900°C in air.

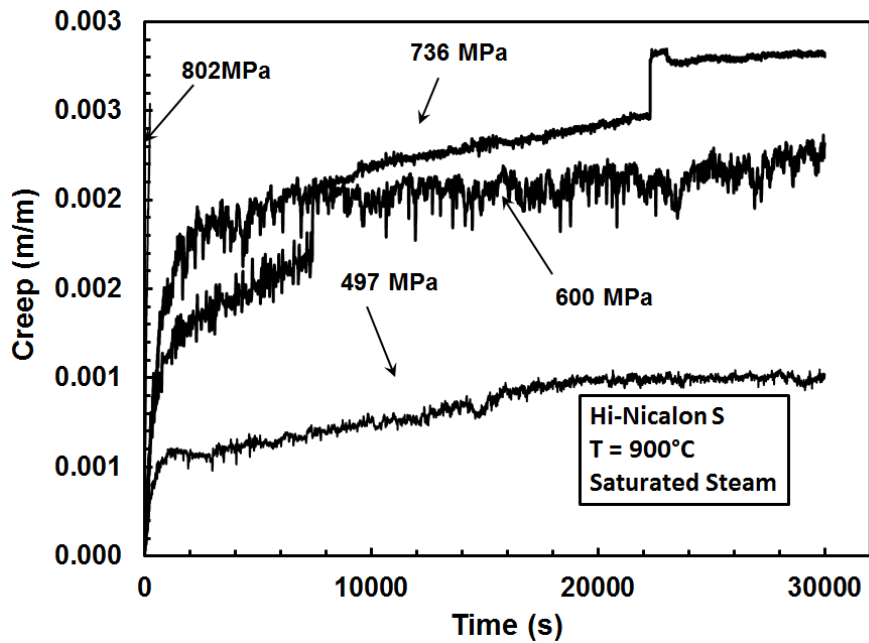


Figure 23. Creep strain vs. time for Hi-Nicalon™ S fiber tows at 900°C in silicic acid-saturated steam.

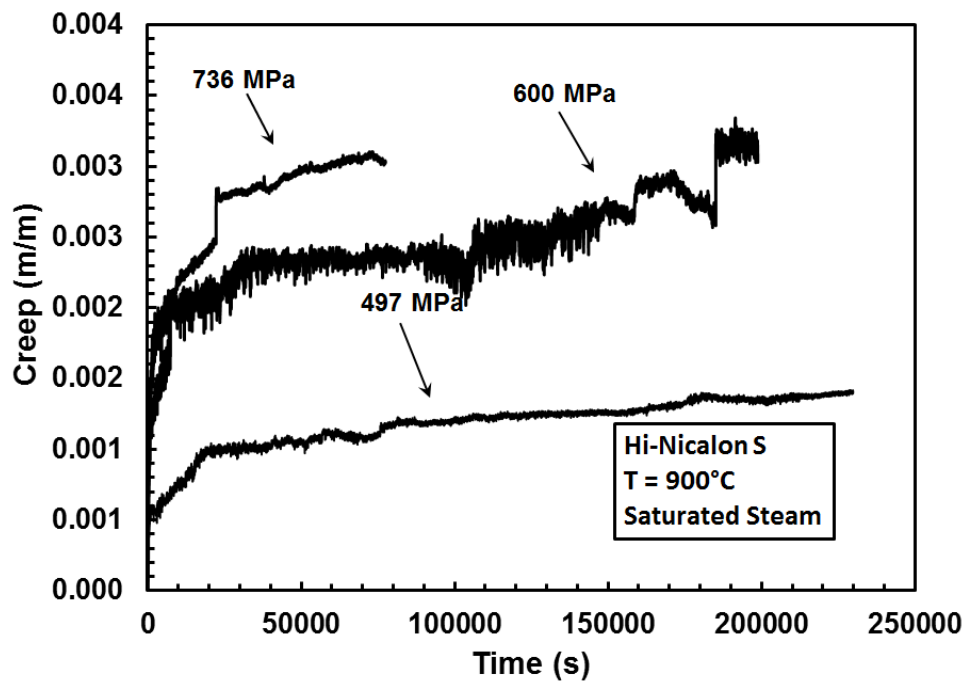


Figure 24. Creep strain vs. time for Hi-Nicalon™ S fiber tows at 900°C in silicic acid-saturated steam.

At 900°C the presence of steam has a noticeable effect on creep strains and creep lifetimes. At 900°C in air, creep run-out was achieved at 736 MPa. In contrast, at 900°C in saturated steam, creep run-out was achieved only at near zero stress of 3.5 MPa. Creep strain produced at 736 MPa at 900°C in saturated steam is nearly an order of magnitude higher than that obtained in air. Detrimental effects of steam are evident.

Steady-state creep was reached in all experiments and dominated the majority of the creep lifetime. Creep rate as a function of applied stress is presented in Error! Reference source not found.. The presence of silicic acid-saturated steam increases creep rates of the Hi-Nicalon S fiber tow. At 900°C the creep rates of the Hi-NicalonTM S fiber tow in saturated steam are approximately one order of magnitude higher than those obtained in air.

Fitting the creep results obtained at 900°C in silicic acid saturated steam with a temperature-independent power law equation [12]:

$$\dot{\epsilon} = A\sigma^n$$

where $\dot{\epsilon}$ is the minimum creep rate, A is a temperature-dependent coefficient that accounts for the activation energy and other variables, and σ is the applied stress, yields a stress exponent $n \approx 5.69$. Mechanical failure of the steam generation system during this effort prevented further testing in air due to time constraints, further testing in air is needed to calculate the corresponding stress exponent. The stress exponent $n \approx 5.69$ obtained at 900°C in saturated steam suggests cavity growth controlled creep as the primary creep mechanism. Roberston [19] found the stress exponent of $n \approx 4.1$ at 800°C in saturated steam. Piper [20] determined the stress exponent as $n \approx 5.3$ at 700°C in saturated steam. These results suggest that as the temperature increases, the creep mechanism operating in Hi-NicalonTM S fibers changes from climb controlled dislocation towards cavity growth controlled creep.

Additional testing is needed to reduce the data scatter and obtain more accurate values of the stress exponent. Detailed microstructural evaluation of the tested samples would lead to a more definitive conclusion regarding the controlling creep mechanisms.

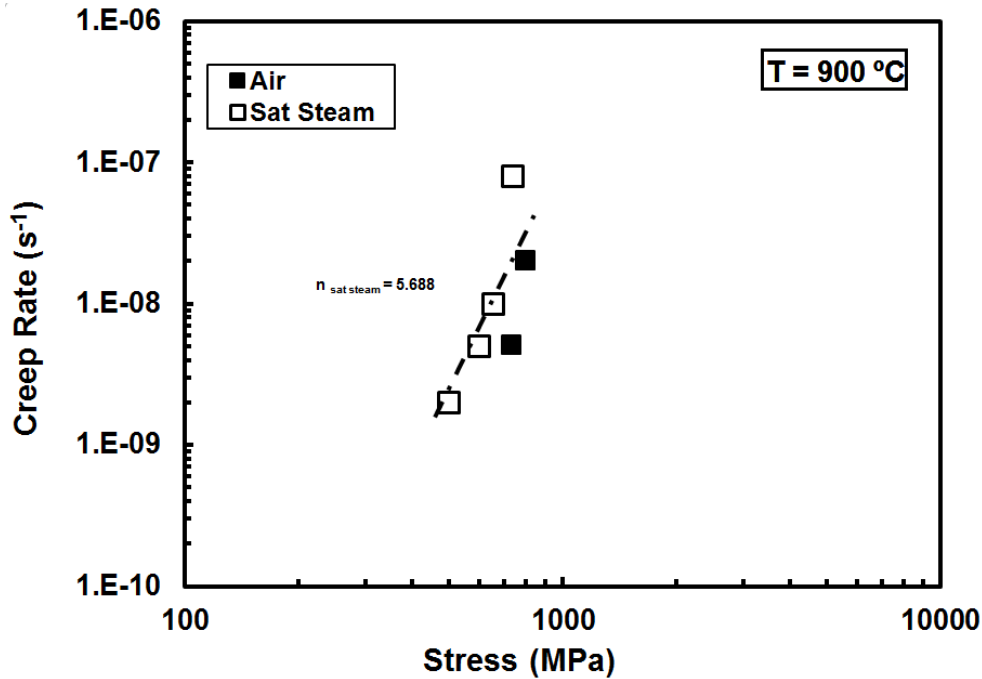


Figure 25. Steady-state creep strain rate vs. applied creep stress for Hi-Nicalon™ S fiber tows at 900°C in air and in saturated steam.

The stress-rupture behavior at 900 °C in air and in saturated steam is summarized in **Figure 26** together with the results obtained at 900°C in air by Shillig [22]. As expected, the creep lifetime decreases with increasing applied stress. At 900°C, saturated steam reduced creep lifetimes by approximately one order of magnitude. Notably, a similar one order of magnitude decrease in creep lifetimes was reported for Hi-Nicalon S fibers at 700°C by Piper [20] and at 800°C by Robertson [19].

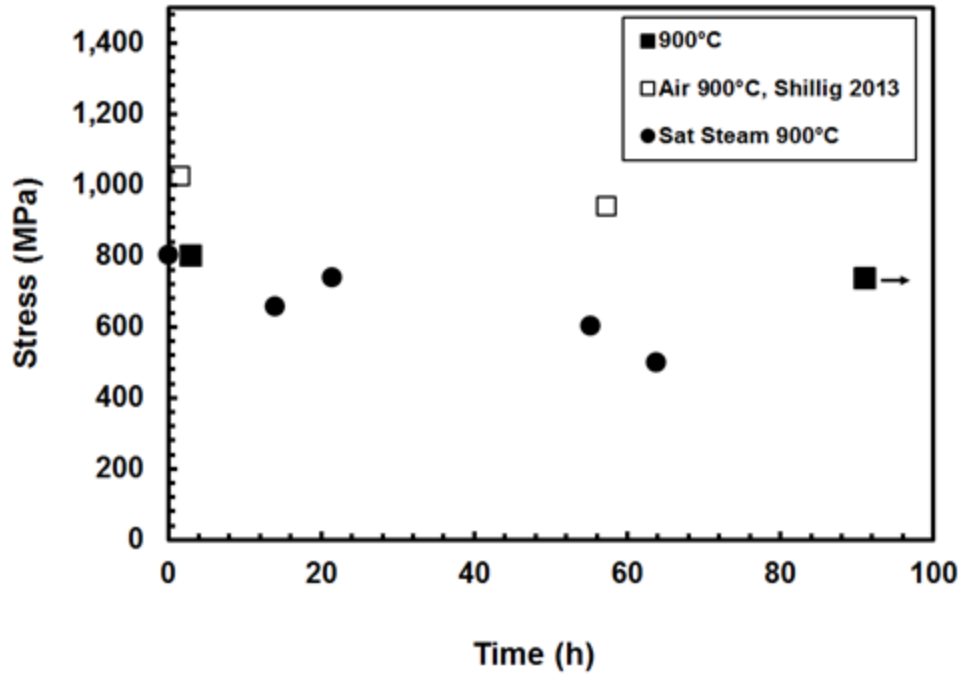


Figure 26. Creep stress vs. time to rupture for Hi-Nicalon™ S fiber tows at 900°C in air and in saturated steam. Data at 900°C in air from Shillig [22] is included. Arrow indicates specimen failure did not occur when test was terminated.

It is instructive to compare the results obtained for Hi-Nicalon™ S fibers in silicic-acid saturated steam with those obtained in unsaturated steam. Creep strain rate vs. applied stress results obtained at 800 and 900 °C in laboratory air, in unsaturated steam and in saturated steam are presented in **Figure 27**. Results at 800°C in air are from Shillig [22] and Robertson [19]. Results at 800°C in unsaturated steam are from Shillig. Results at 800°C in saturated steam are from Robertson. Results at 900°C in air are from Shillig and current effort. Results at 900°C in unsaturated steam are from Shillig. Results at 900°C in saturated steam are from current effort.

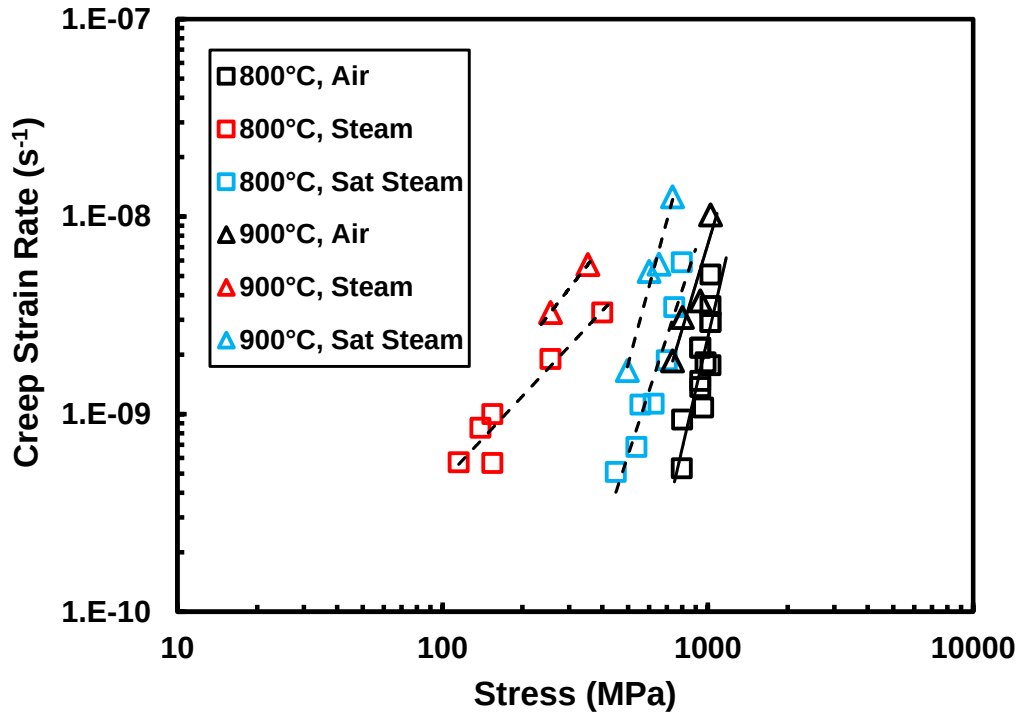


Figure 27. Steady-state creep rate vs. applied stress for Hi-Nicalon S fiber tows at 800 and 900°C in laboratory air, steam and silicic-acid saturated steam. Results from Robertson [19] and Shillig [22] are included for comparison.

Comparison of the results obtained at elevated temperature in unsaturated steam and in silicic acid-saturated steam allows us to determine which of these two environments causes greater degradation of the creep performance of Hi-NicalonTM S fibers. As seen in **Figure 27**, creep rates produced at 800 and 900 °C in silicic acid-saturated steam are an order of magnitude higher than creep rates produced in dry air. However, creep rates produced at 800 and 900 °C in silicic acid-saturated steam are an order of magnitude lower than creep rates in unsaturated steam. Clearly the unsaturated steam causes considerably greater degradation of the creep resistance of Hi-NicalonTM S fibers than the unsaturated steam. This result bodes well for the advanced SiC/SiC CMCs reinforced with Hi-NicalonTM S fibers. As mentioned in Section 4.1 above, steam attacking the SiC/SiC CMC operating in combustion

environment will enter the composite through matrix cracks, will leach Si from the SiC matrix and become saturated with silicic acid as it travels towards the reinforcing fibers. We can expect better performance from the advanced SiC/SiC composites at elevated temperature in steam than that indicated by the early work of Steffens [21] and Shillig [22]. These observations are confirmed when we compare the stress-rupture results obtained 800 and 900 °C for Hi-NicalonTM S fibers in silicic-acid saturated steam with those obtained in unsaturated steam in **Figure 28**, where results at 800°C in air are from Shillig [22], results at 800°C in unsaturated steam are from Shillig, results at 800°C in saturated steam are from Robertson [19], results at 900°C in air are from Shillig and current effort, results at 900°C in unsaturated steam are from Shillig, and results at 900°C in saturated steam are from current effort.

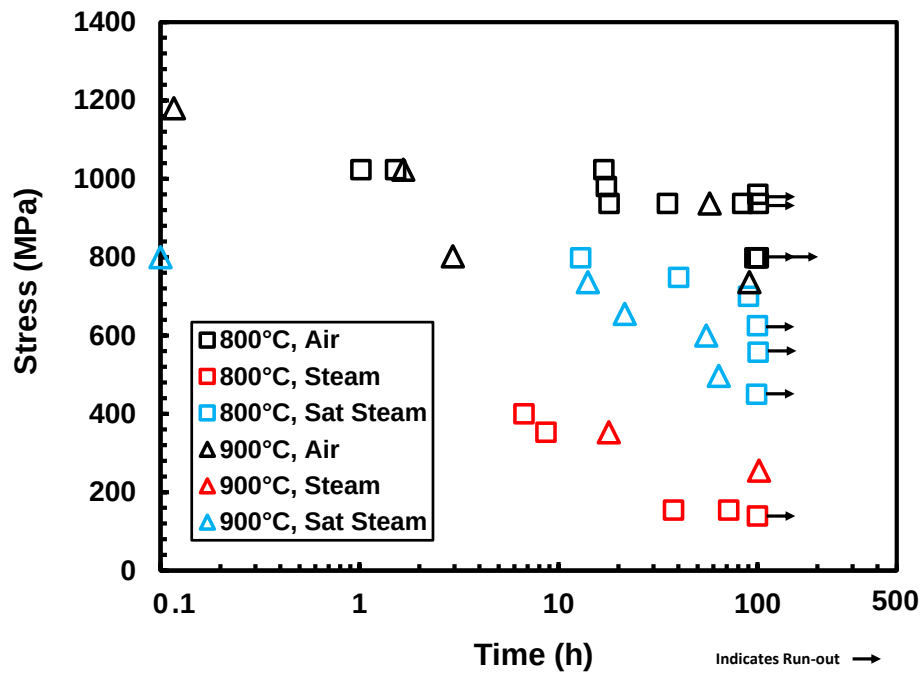


Figure 28. Creep stress vs. time to rupture for Hi-Nicalon S fiber tows at 800 and 900°C in laboratory air, steam and silicic-acid saturated steam. Results from Robertson [19] and Shillig [22] are included for comparison.

5.2 Post-Test Microstructural Analysis of Hi-Nicalon™ S Fiber Tows

Post-test microstructural analysis of the Hi-Nicalon™ S fiber tow specimens was conducted using a FEI Quanta 450 scanning electron microscope (SEM) **Figure 29**. A total of sixteen specimens taken from nine fiber tows, five tested in saturated steam and four tested in air, were analyzed. Fiber tow specimens were prepared for examination with an SEM using standard methods. A detailed description of the SEM specimen preparation is given elsewhere [19], [20], [11]. The method for SEM sample preparation is briefly outlined below.



Figure 29. FEI Quanta 450 Scanning Electron Microscope

Fiber tows were prepared for microstructural analysis by mounting the fibers on an aluminum puck with a 45° surface. First, double-sided carbon tape was applied to the angled surface of the mounting puck. The fibers were then pressed onto the carbon tape such that the portion of the fibers to be analyzed protruded 1-5 mm above the angled surface. After placing the fibers on the puck, a layer of silver paint was applied in order to secure the fibers in

place. The silver paint also provides electric conductivity between the fibers and the aluminum puck, which is required for SEM imaging. After the fibers were securely fixed in place, the remainder of the fiber tow was cut at the bottom of the angled surface to separate it from the specimen to be analyzed.



Figure 30. Hi-NicalonTM S fiber tow specimens tested in creep at 900°C in air and in silicic acid-saturated steam prepared for examination with an SEM

All fiber tow specimens tested in this work were examined with an SEM. In the case of fiber tow specimens that failed during creep test, three samples were examined with an SEM: (1) fiber tow fracture surfaces, (2) a section of the fiber tow located above the fracture and (3) a section of the fiber tow located below the fracture. In the case of the fiber tow specimens that achieved creep run-out of 100 h, the section near the mid-point of the fiber tow was examined with an SEM. A summary of the SEM specimens examined in this work is given in **Table 6**.

Table 6. Hi-Nicalon™ S specimens analyzed using scanning electron microscope.

Specimen	Creep Test (MPa)	Creep Test Duration (h)	SEM Specimen Location Within Fiber Tow
A1	3.5	100*	Midpoint
A2	802	2.94	Upper Section
			Lower Section
A3	736	100*	Upper Section
			Lower Section
A5	1180	0.12	Upper Section
			Lower Section
S1	3.5	100*	Midpoint
S11	736	21.50	Upper Section
			Lower Section
S12	654	14	Upper Section
			Lower Section
S13	600	55.20	Upper Section
			Lower Section
S16	497	63.80	Upper Section
			Lower Section

* Run-out, defined as 100 h at creep stress. Failure of specimen did not occur when the test was terminated.

Previous work by Steffens [21] and Shillig [22] demonstrated that unsaturated steam entering the test chamber lead to variations in oxidation process along the fiber tow specimen. Active oxidation was observed in the bottom section of the fiber tow, while passive oxidation and formation of thick silica was reported near the top of the fiber. Conversely, fiber tows tested in silicic acid saturated steam by at 800°C by Robertson [19] and at 700°C by Piper [20] , exhibited passive oxidation along the length of the fiber tow specimens. Both Robertson and Piper observed that tested fibers had uniform appearance and reported consistent silica scale growth on the surface of tested fibers. As in the case of fiber tows tested in saturated steam at 700 and 800°C, fiber tows tested at 900°C in saturated

steam also had uniform surface appearance along the entire length of the fiber tow specimen. Recall that in the case of fiber tows tested in saturated steam at 700 and 800 °C the growth of silica scale on the fiber surface was not discernible under an SEM. In contrast, in the case of fibers tested in saturated steam at 900°C the growth of silica scale becomes clearly visible, as evidenced in **Figure 31** and **Figure 32**. As discussed in 5.1, the fibers within a fiber tow specimen maintain close proximity to one another during testing. Silica scale growth at times appeared to encompass multiple fibers; as evidenced by the scale fracture in **Figure 32**.

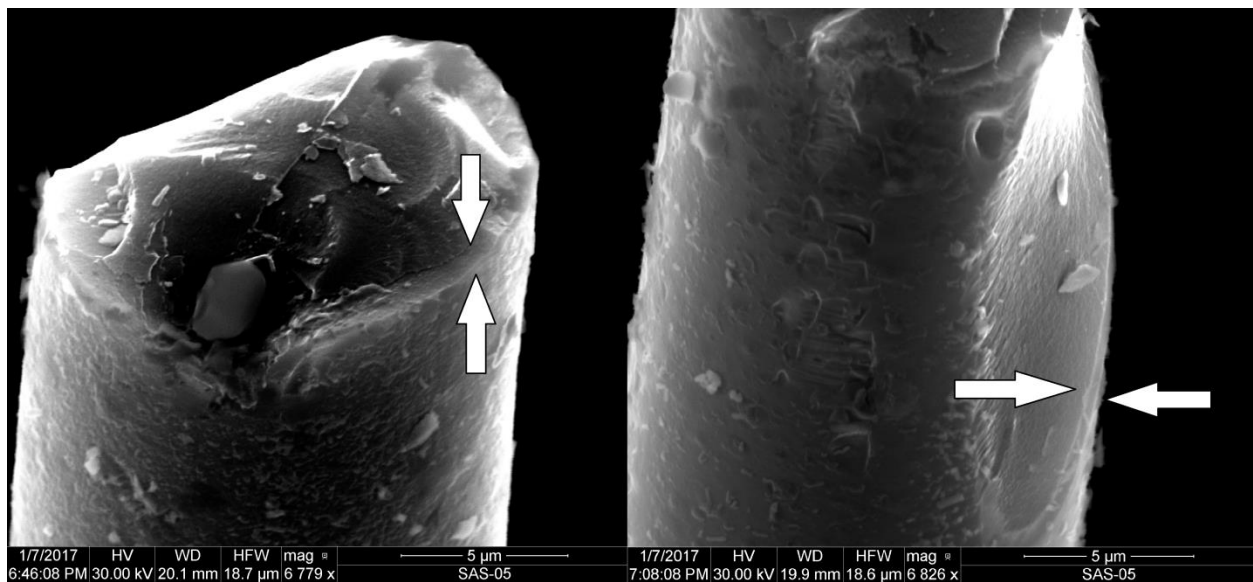


Figure 31. Representative fiber from HI-Nicalon S fiber tow specimen subjected to 3.5 MPa at 900°C in silicic acid-saturated steam for 100 h. Oxide scale is visible (see arrows).

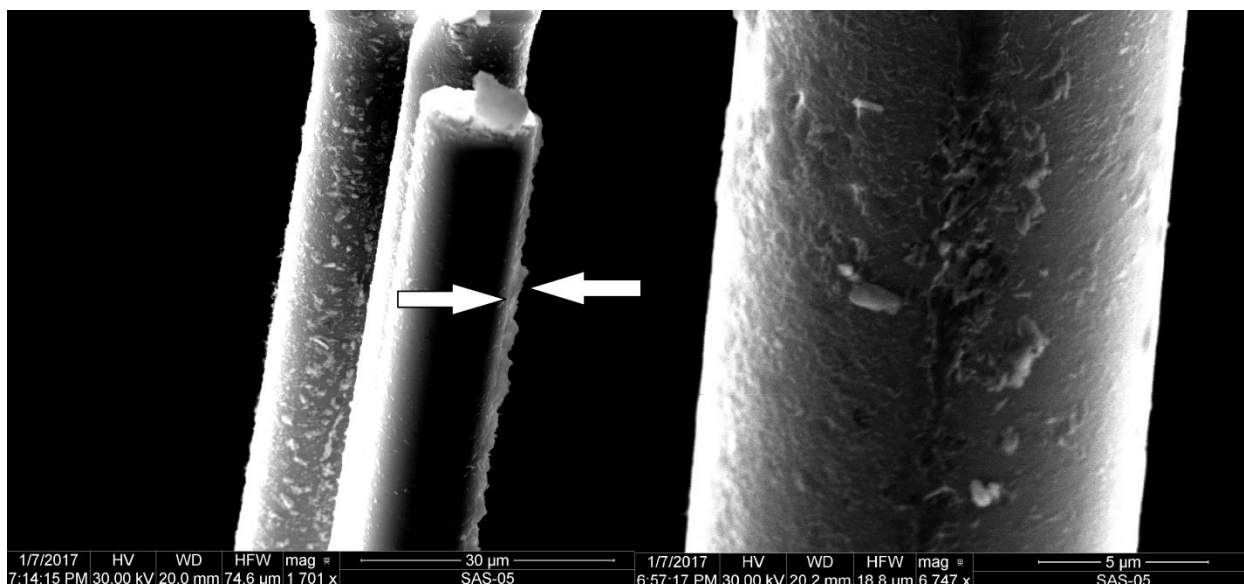


Figure 32. Representative fiber from HI-Nicalon S fiber tow specimen subjected to 3.5 MPa at 900°C in silicic acid-saturated steam for 100 h. Fiber interface bridge is visible (see arrows).

It is recognized that the as-processed SiC fibers contain microstructural defects. Evidence of environmentally assisted crack growth was noted in fracture surfaces of samples tested in saturated steam with creep stress of 600 MPa and 736 MPa (see **Figure 33**). Significant portions of the fiber fracture surfaces appear to be oxidized, indicating that a large crack has formed during creep test prior to final fracture of the fiber tow. Moreover, growth of silica crystals is believed to be seen in the oxidized region of the fracture surface at the leading edge of crack (see **Figure 33b**).

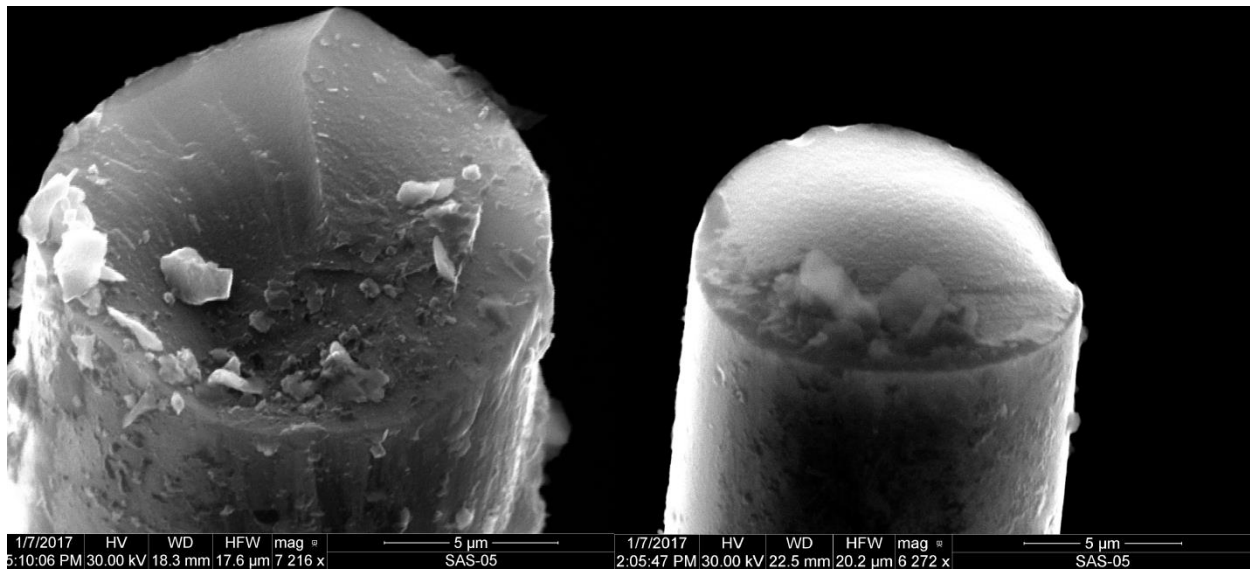


Figure 33. Evidence of environmentally assisted crack growth (EACG) of a) Silicic acid-saturated steam sample, “Steam 13” and b) “Steam 6”

A number of fibers showed dark areas as well as mottled areas running axially along the fiber length, which are presumably a result of contact between fibers during testing (see **Figure 34** and **Figure 35**). In the case of fibers tested in saturated steam, such linear regions were accompanied by visible silica formation. Note that similar darks and mottled areas along the lines of contact between two fibers were also observed at 800°C by Robertson [19] and at 700°C by Piper [20].

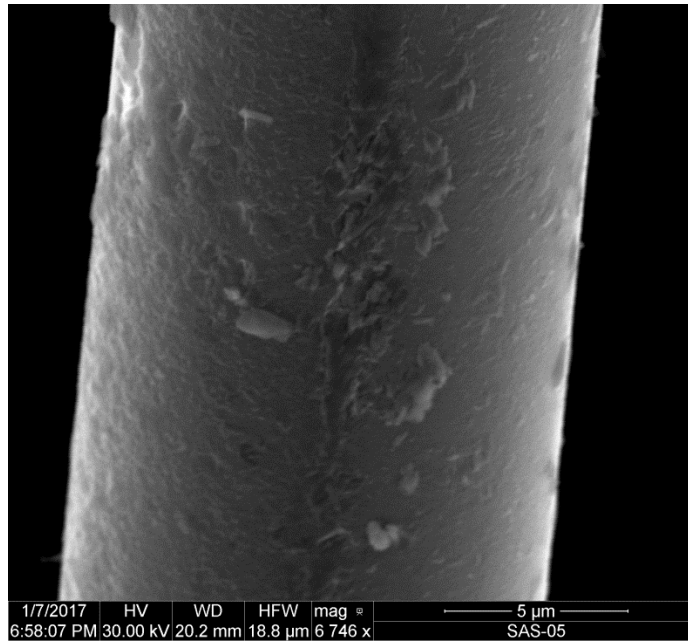


Figure 34. Silicic acid-saturated steam exposure leading to longitudinal fiber scale development during “Steam 1” fiber tow test.

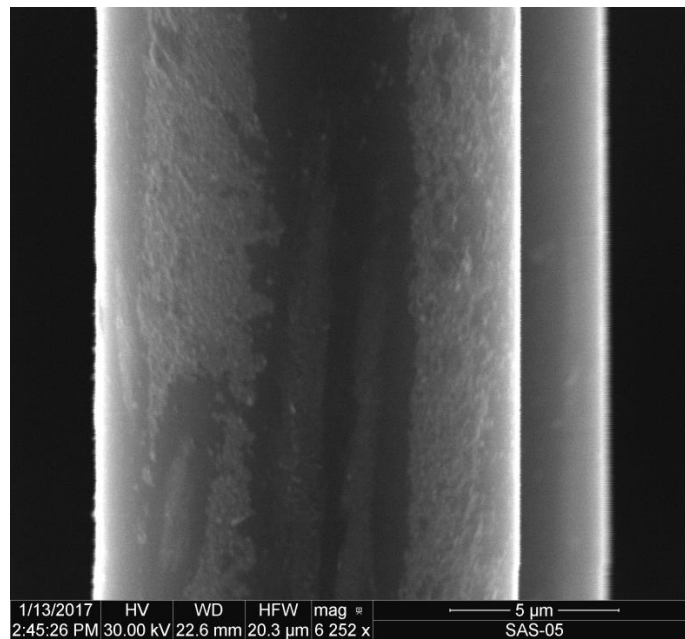


Figure 35. Longitudinal fiber scale development during “Air 3” fiber tow test.

Fracture surfaces of all tested fibers were analyzed using SEM. The fracture surfaces showed evidence of classic brittle fracture. The fracture surface appears to be perpendicular

to the fiber surface. As seen in **Figure 36**, the fracture is initiated on the left side of the fiber (see arrow). The crack propagated radially leading to ultimate fiber fracture. A possible indicator of EACG is visible around the perimeter of the fracture surface.

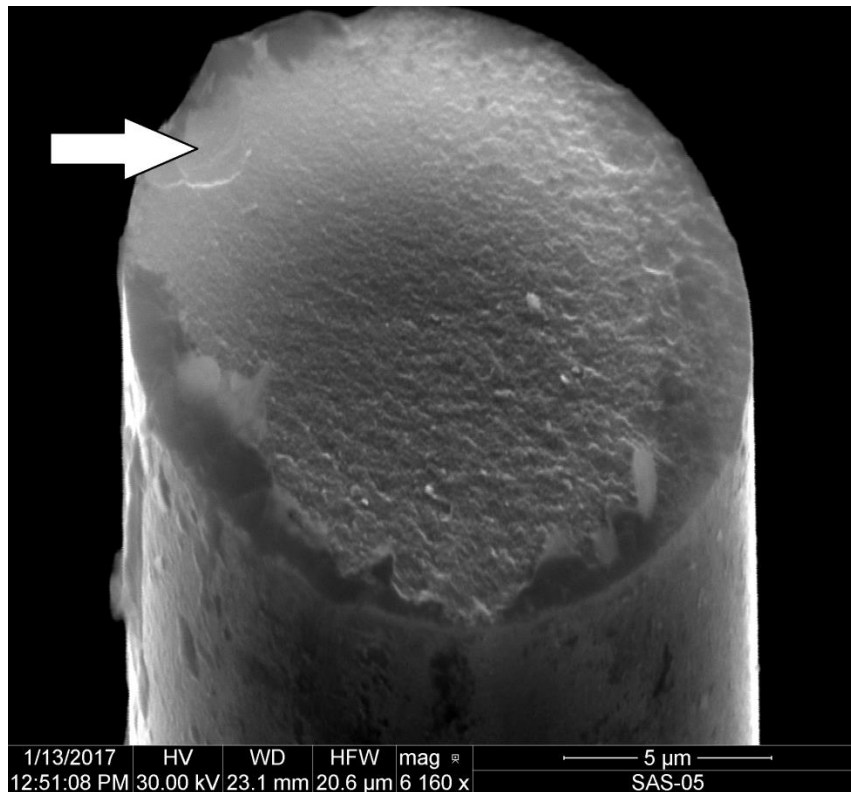


Figure 36. Brittle fracture of “Air 5” fiber tow specimen in air at 900°C. Fracture propagation point seen by arrow.

VI. Conclusions and Recommendations

6.1 Concluding Remarks

The effects of silicic acid-saturated steam on creep of Hi-NicalonTM S fiber tows at 900°C were investigated in creep tests conducted in silicic acid-saturated saturated steam and in laboratory air. The presence of silicic acid-saturated steam significantly degraded creep performance of Hi-NicalonTM S fibers. In saturated steam, creep lifetimes were reduced by approximately one order of magnitude compared to those produced in air. In silicic acid-saturated steam, creep run-out of 100 h was achieved only at near zero stress of 3.5 MPa. At 900°C in air, creep runout was achieved at 736 MPa. Creep rates obtained in silicic acid-saturated steam were an order of magnitude higher than those produced in air.

Results obtained in saturated steam at 900°C as well as those obtained at 800°C [19] were compared with creep results produced at 900 and 800 °C in unsaturated steam [22]. Comparison revealed that unsaturated steam was significantly more damaging to Hi-NicalonTM S fibers than silicic acid-saturated steam. While the presence of saturated steam reduced creep lifetimes by one order of magnitude, unsaturated steam degraded creep lifetimes by two orders of magnitude. While the creep rates in saturated steam were one order of magnitude higher than those in air, creep rates in unsaturated steam were two orders of magnitude higher than the rates obtained at 900°C in air [22].

Microstructural analysis of fiber tows tested in creep in silicic acid-saturated steam at 900°C with an SEM revealed evidence of silica scale growth. A small number of fibers showed evidence of environmentally assisted crack growth.

6.2 Recommendations

Creep behavior of Hi-Nicalon™ S fiber tows has been investigated at 700°C [20] 800°C [19] and 900°C in air and in silicic acid-saturated steam. Notably, silica scale growth was more pronounced at 900°C. The effects of silicic acid-saturated steam on creep of Hi-Nicalon™ at a wider range of temperatures remain to be investigated. Testing at temperatures above 900°C is likely to promote the growth of silica scale on the fibers. Thus the results obtained at temperatures > 900°C will provide additional evidence needed to determine the effects of a well-developed silica scale on creep performance of Hi-Nicalon™ fibers in the presence of silicic acid saturated steam.

SEM images were collected to analyze the failure mechanisms and oxidation of the fibers creep tested at 900°C in silicic acid-saturated steam. This work should be complemented by transmission electron microscopy (TEM) analysis of the fibers. TEM allows higher magnification and a greater level of fidelity in determining the presence of an oxide layer or other deposits on the surfaces of the fibers.

Future creep testing of Hi Nicalon S fibers at elevated temperatures in air or in saturated steam environments should include acoustic emission to capture individual fiber failure events. The acoustic emission data will be instrumental in determining whether progressive deformation of the fiber tow under constant applied load is indeed due creep of fibers or due to progressive failures of individual filaments within the tow.

Appendix A. SEM Images

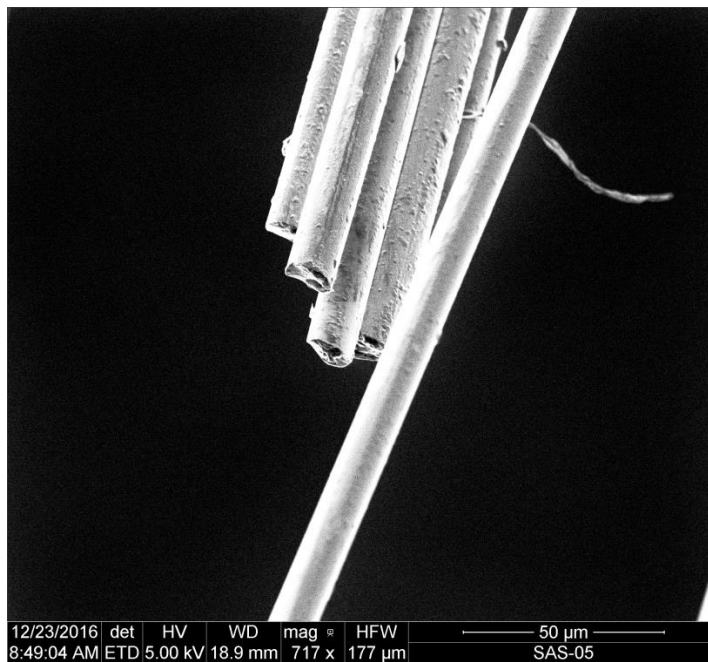


Figure 37. SEM micrograph of the Hi-Nicalon™ S specimen “Virgin Sample” examining surface features

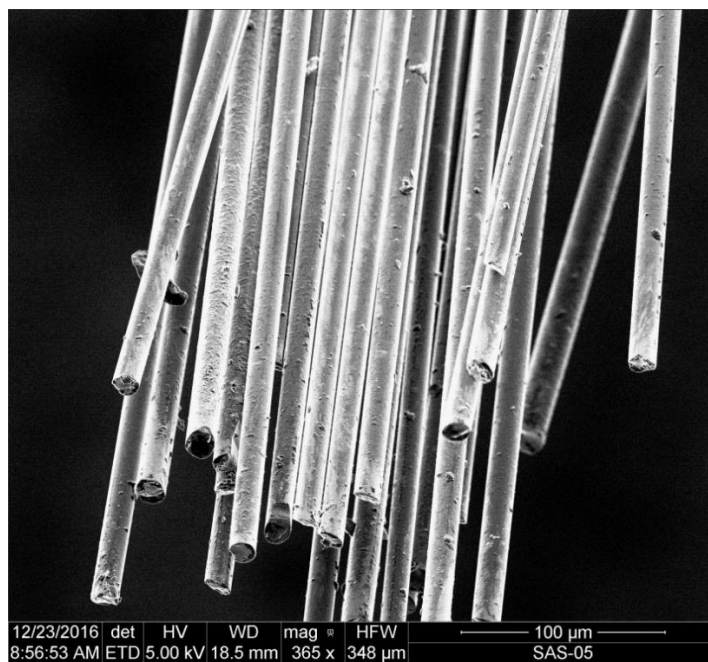


Figure 38. SEM micrograph of the Hi-Nicalon™ S specimen “Virgin Sample” examining surface features

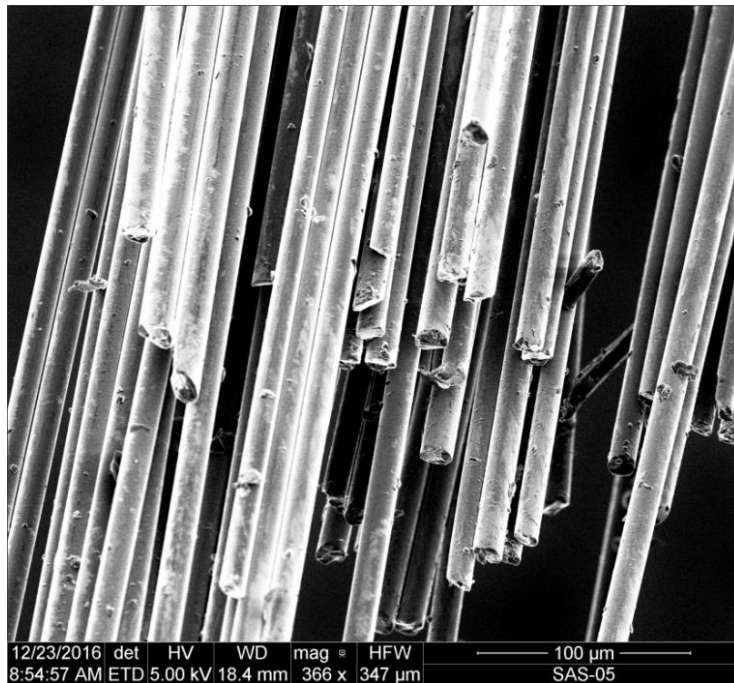


Figure 39. SEM micrograph of the Hi-Nicalon™ S specimen “Virgin Sample” examining surface features

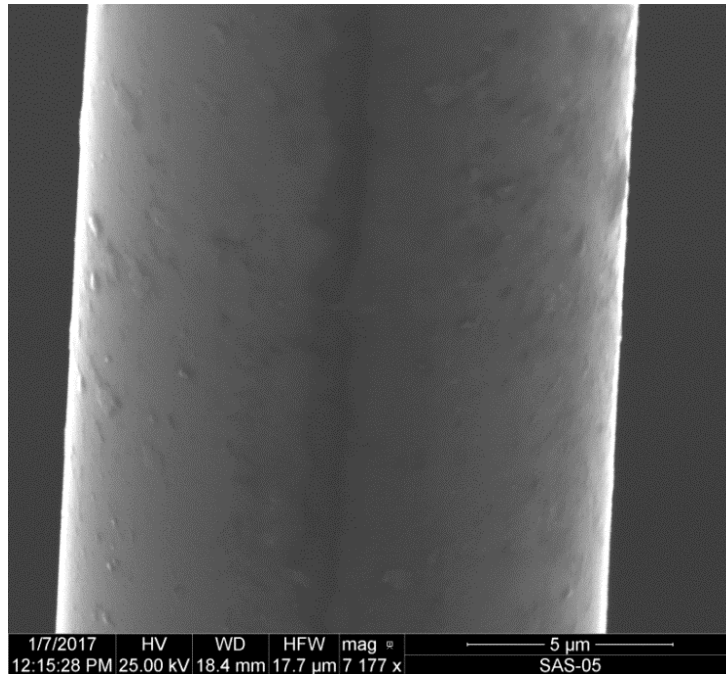


Figure 40. SEM micrograph of the Hi-Nicalon™ S specimen “Virgin Sample” examining surface features

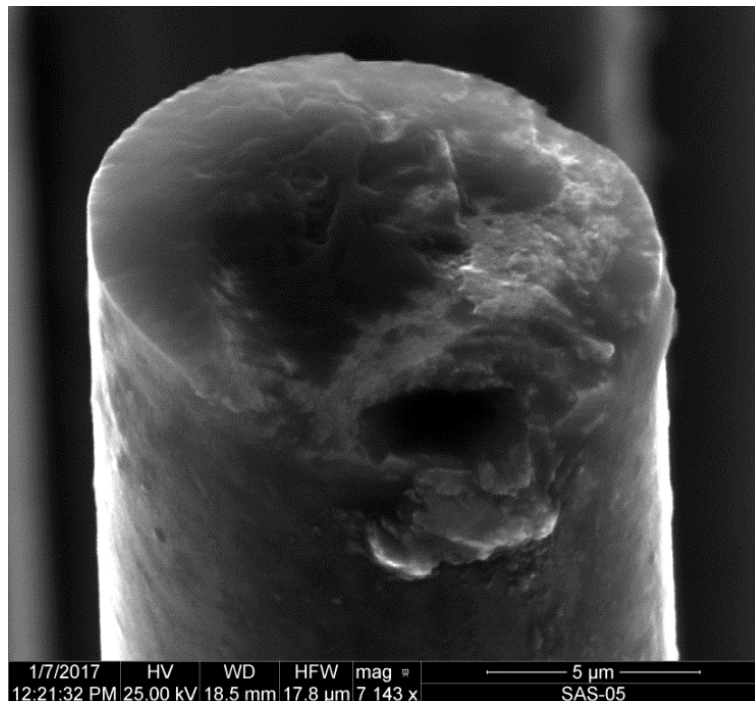


Figure 41. SEM micrograph of the Hi-Nicalon™ S specimen “Virgin Sample” examining surface features

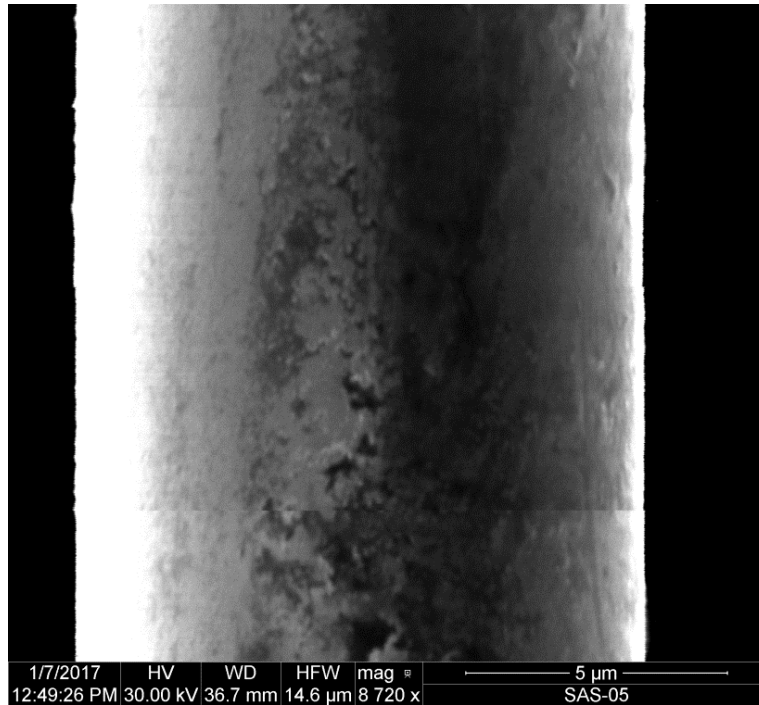


Figure 42. SEM micrograph of the Hi-Nicalon™ S specimen “Air 1” examining fiber surface in middle portion of fiber ($\sigma_{cr} = 3.4695$ MPa, $t_f > 100$ h)

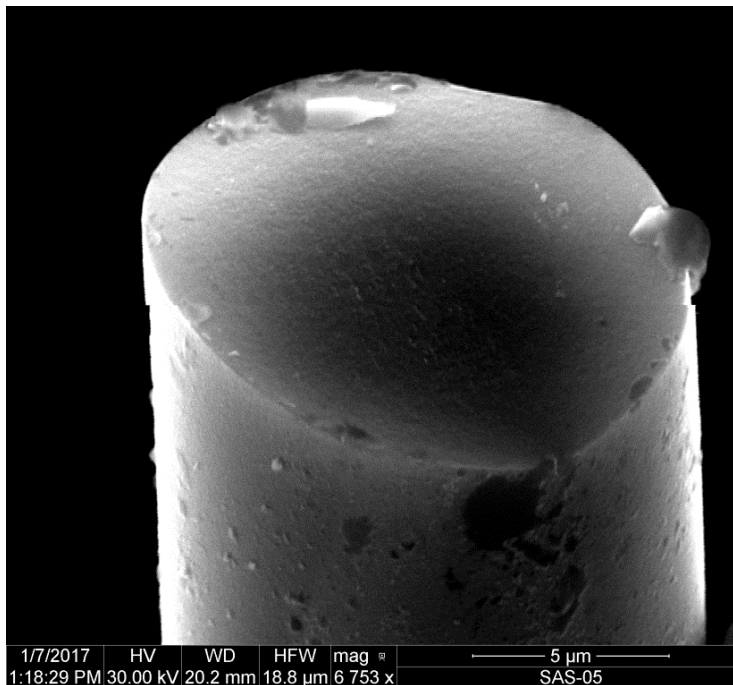


Figure 43. SEM micrograph of the Hi-Nicalon™ S specimen “Air 1” examining fiber surface in middle portion of fiber ($\sigma_{cr} = 3.4695$ MPa, $t_f > 100$ h)

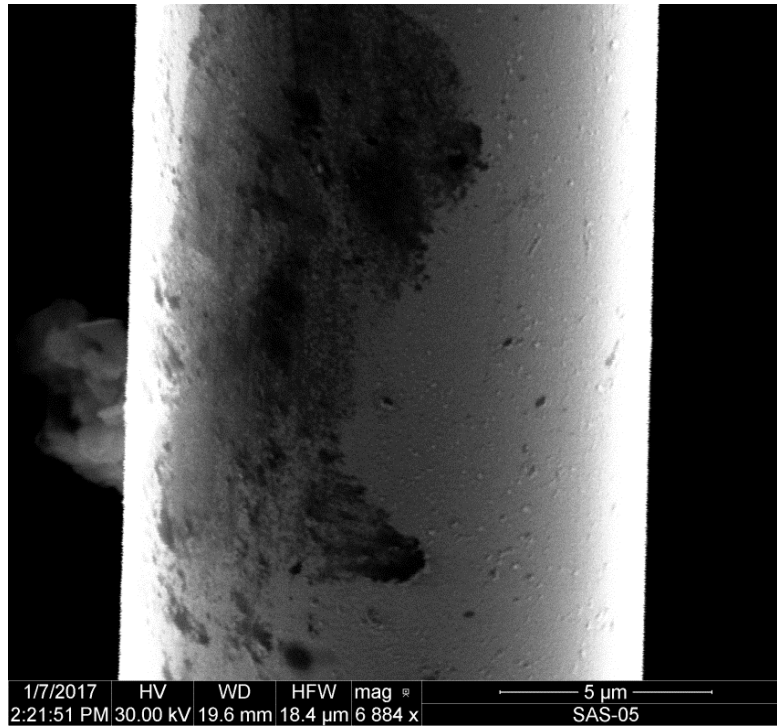


Figure 44. SEM micrograph of the Hi-Nicalon™ S specimen “Air 2” examining fiber surface in lower portion of fiber ($\sigma_{cr} = 802$ MPa, $t_f = 2.94$ h)

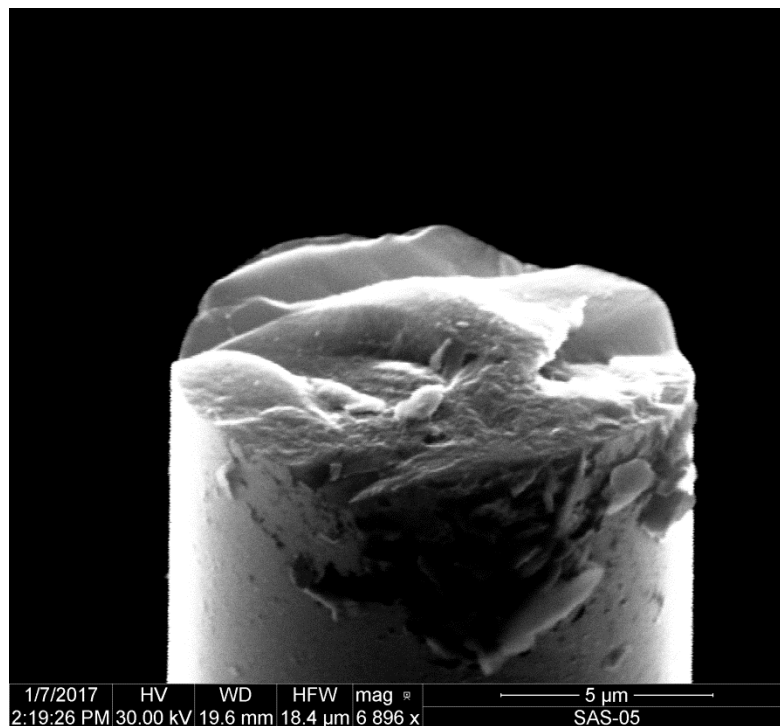


Figure 45. SEM micrograph of the Hi-Nicalon™ S specimen “Air 2” examining the lower fracture surface of a fiber ($\sigma_{cr} = 802$ MPa, $t_f = 2.94$ h)

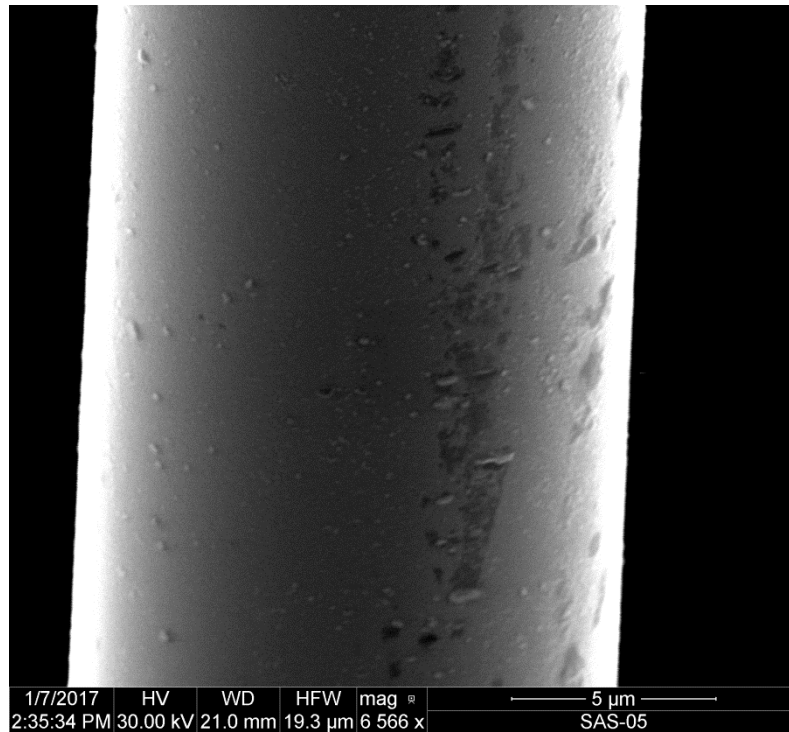


Figure 46. SEM micrograph of the Hi-Nicalon™ S specimen “Air 2” examining fiber surface in upper portion of fiber ($\sigma_{cr} = 802$ MPa, $t_f = 2.94$ h)

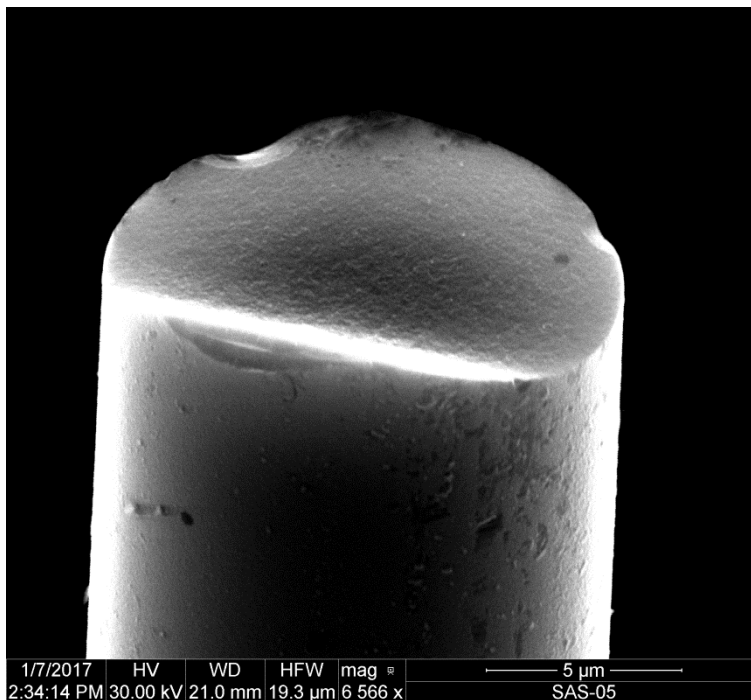


Figure 47. SEM micrograph of the Hi-Nicalon™ S specimen “Air 2” examining the upper fracture surface of a fiber ($\sigma_{cr} = 802$ MPa, $t_f = 2.94$ h)

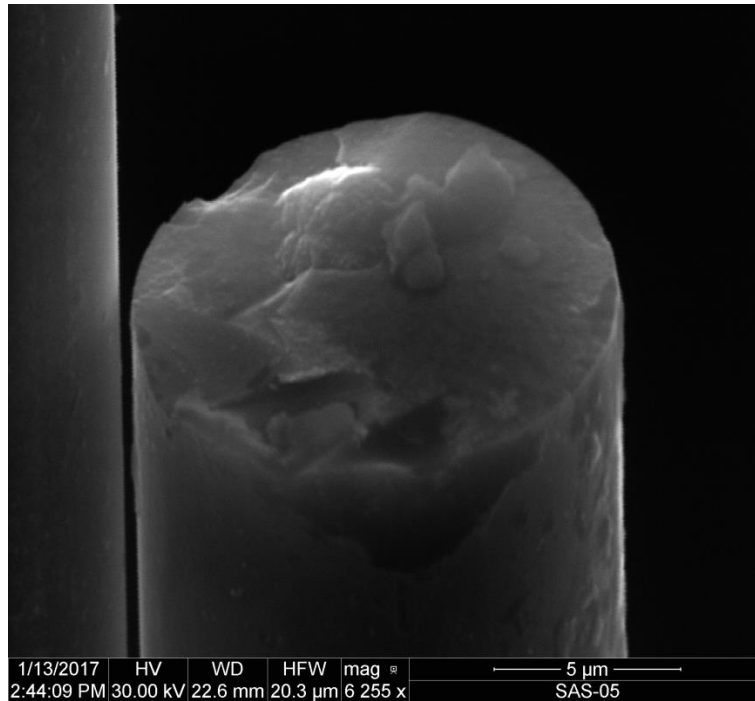


Figure 48. SEM micrograph of the Hi-Nicalon™ S specimen “Air 3” examining the lower fracture surface of a fiber ($\sigma_{cr} = 802$ MPa, > 100 h)

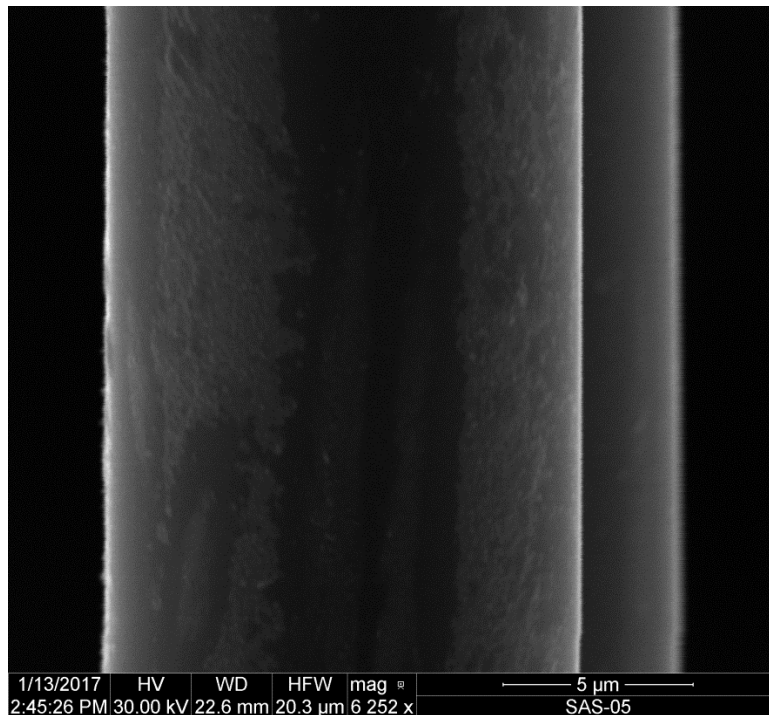


Figure 49. SEM micrograph of the Hi-Nicalon™ S specimen “Air 3” examining fiber surface in lower portion of a fiber ($\sigma_{cr} = 802$ MPa, > 100 h)

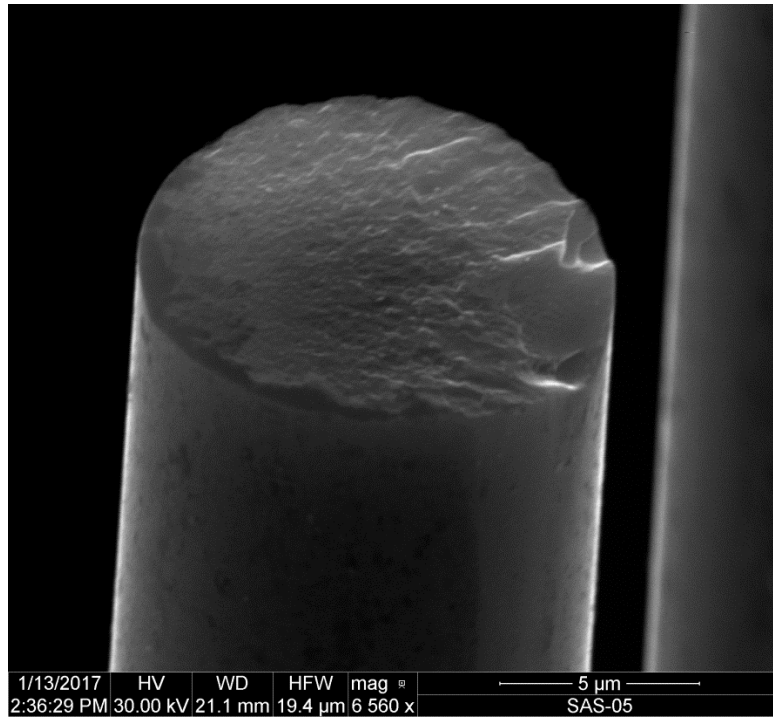


Figure 50. SEM micrograph of the Hi-Nicalon™ S specimen “Air 3” examining upper fracture surface of a fiber ($\sigma_{cr} = 802$ MPa, > 100 h)

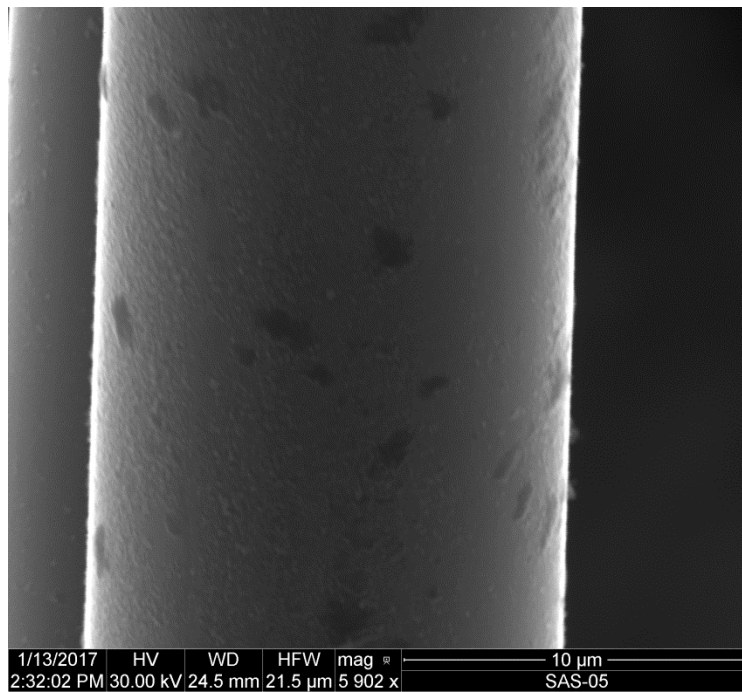


Figure 51. SEM micrograph of the Hi-Nicalon™ S specimen “Air 3” examining fiber surface in upper portion of a fiber ($\sigma_{cr} = 802$ MPa, > 100 h)

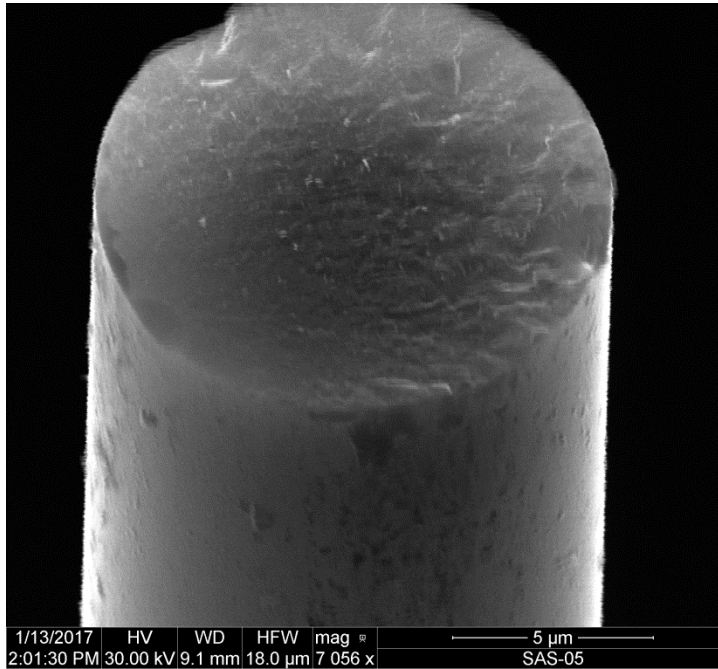


Figure 52. SEM micrograph of the Hi-Nicalon™ S specimen “Air 5” examining lower fracture surface of a fiber ($\sigma_{cr} = 1180$ MPa, $t = 0.12$ h)

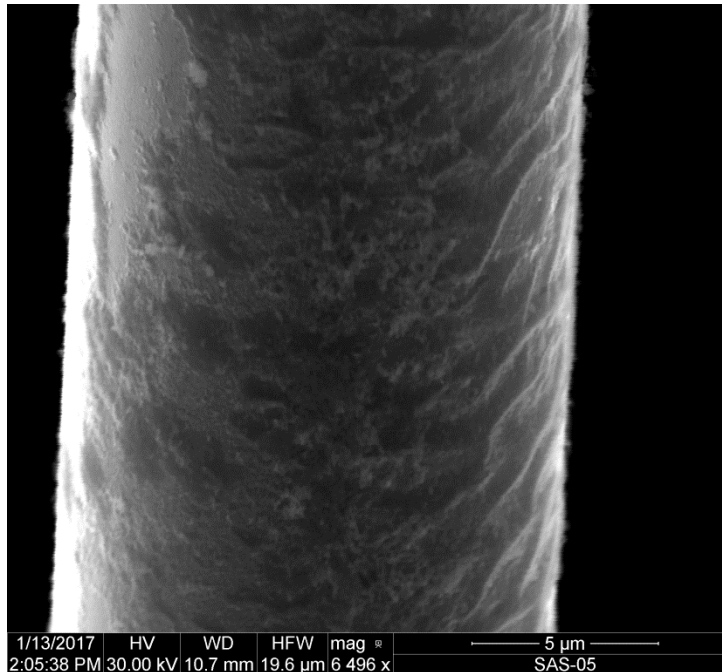


Figure 53. SEM micrograph of the Hi-Nicalon™ S specimen “Air 5” examining fiber surface in lower portion of a fiber ($\sigma_{cr} = 1180$ MPa, $t = 0.12$ h).

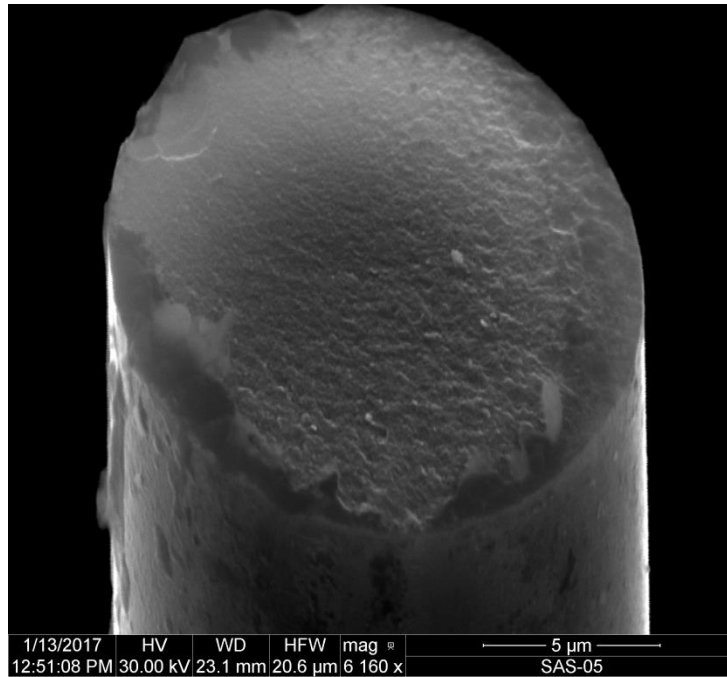


Figure 54. SEM micrograph of the Hi-Nicalon™ S specimen “Air 5” examining upper fracture surface of a fiber ($\sigma_{cr} = 1180$ MPa, $t = 0.12$ h).

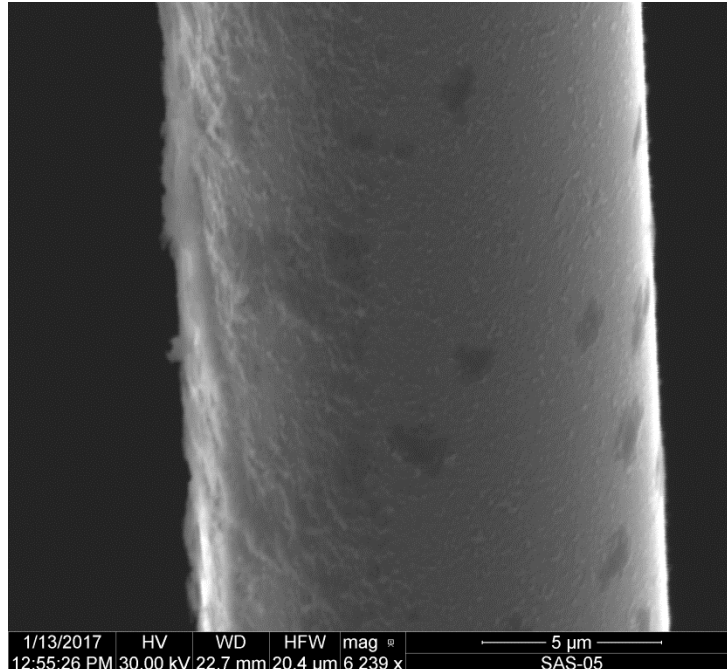


Figure 55. SEM micrograph of the Hi-Nicalon™ S specimen “Air 5” examining fiber surface in upper portion of a fiber ($\sigma_{cr} = 1180$ MPa, $t = 0.12$ h).

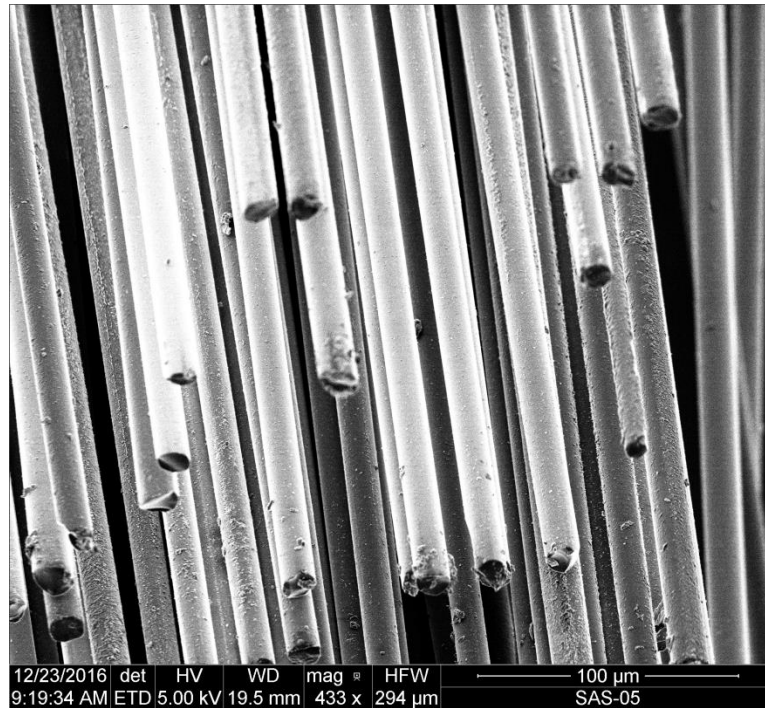


Figure 56. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 1” examining fiber surfaces in middle portion of fiber tow ($\sigma_{cr} = 3.5$ MPa, > 100 h).

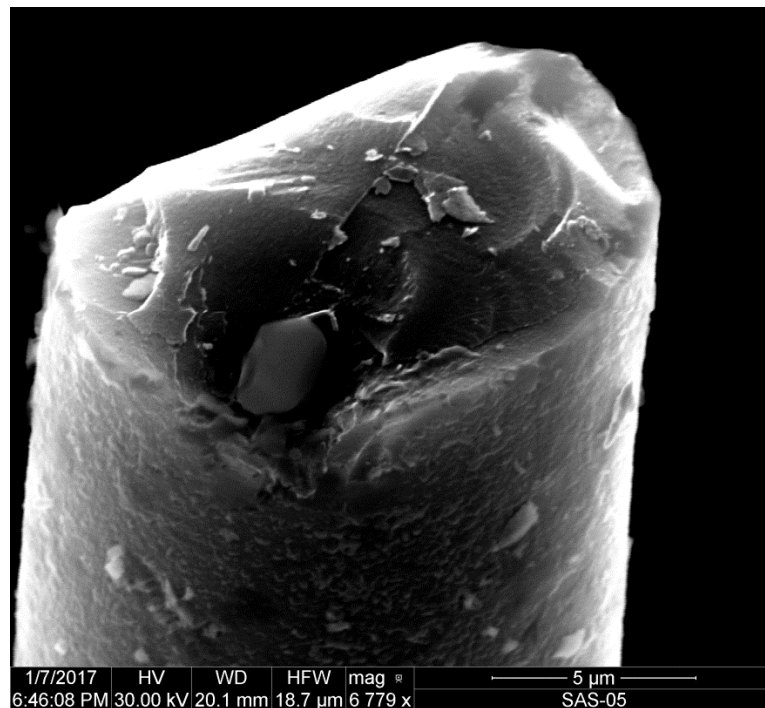


Figure 57. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 1” examining middle cross section of a fiber. Note defined silica scale growth. ($\sigma_{cr} = 3.5$ MPa, > 100 h).

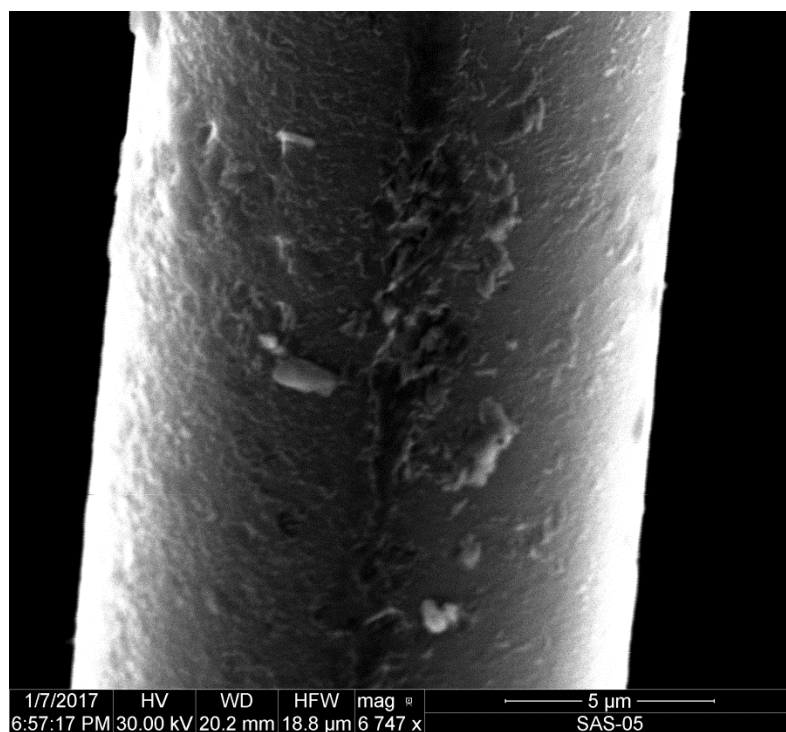


Figure 58. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 1” examining middle surface of a fiber ($\sigma_{cr} = 3.5$ MPa, > 100 h).

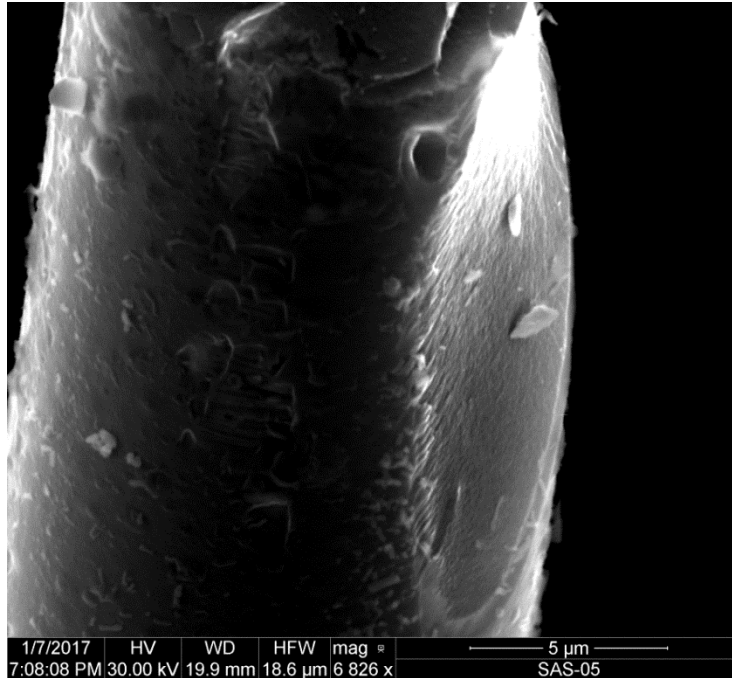


Figure 59. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 1” examining middle cross section of a fiber. Note defined silica scale growth. ($\sigma_{cr} = 3.5$ MPa, > 100 h).

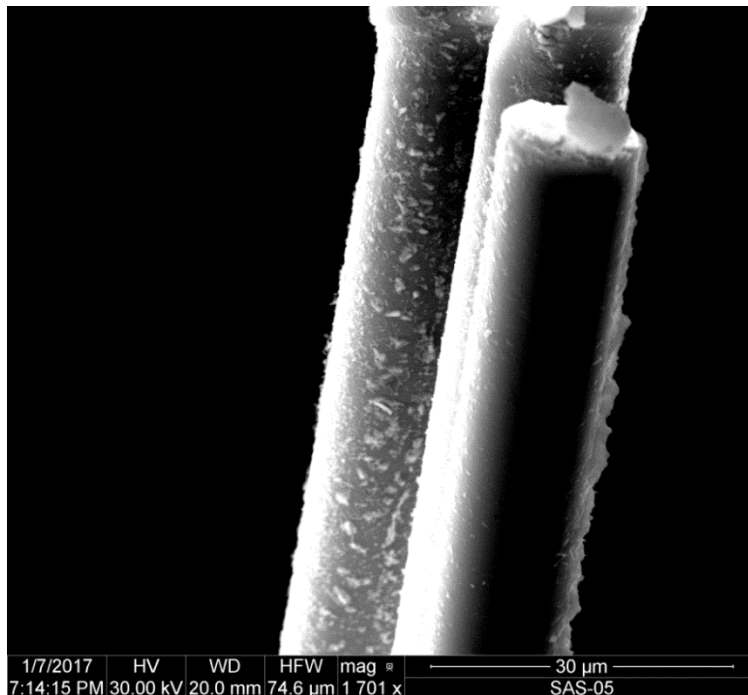


Figure 60. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 1” examining middle cross section of a fiber. Note defined longitudinal silica scale growth from fiber contact. ($\sigma_{cr} = 3.5$ MPa, > 100 h).

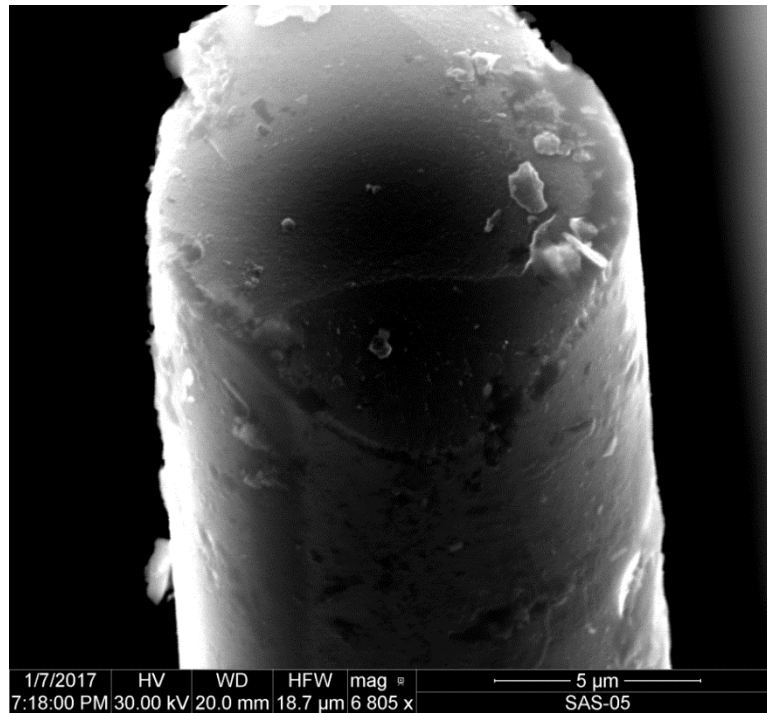


Figure 61. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 1” examining middle cross section of a fiber. ($\sigma_{cr} = 3.5$ MPa, > 100 h).

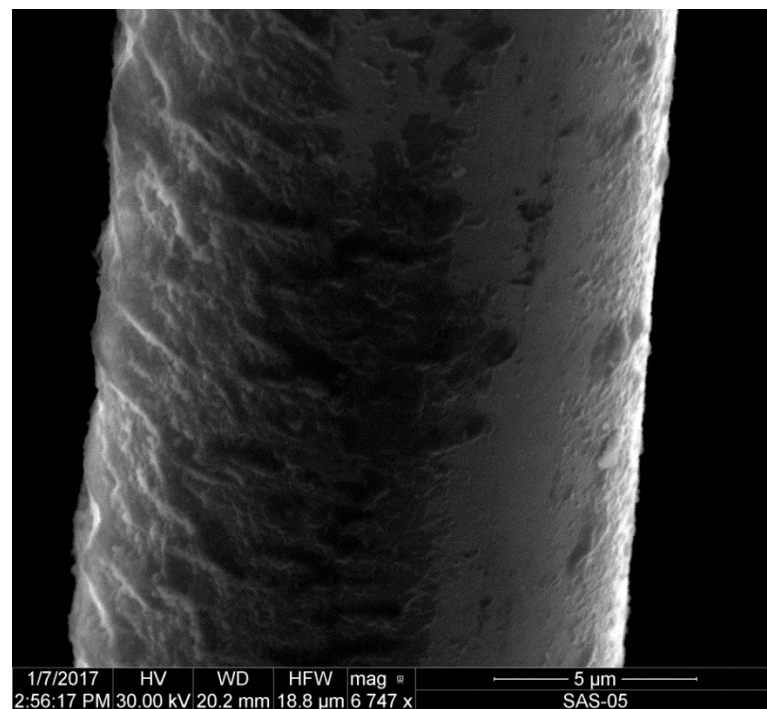


Figure 62. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 16” examining fiber surface on lower portion of a fiber. Note longitudinal silica scale. ($\sigma_{cr} = 497$ MPa, $t = 63.84$ h).

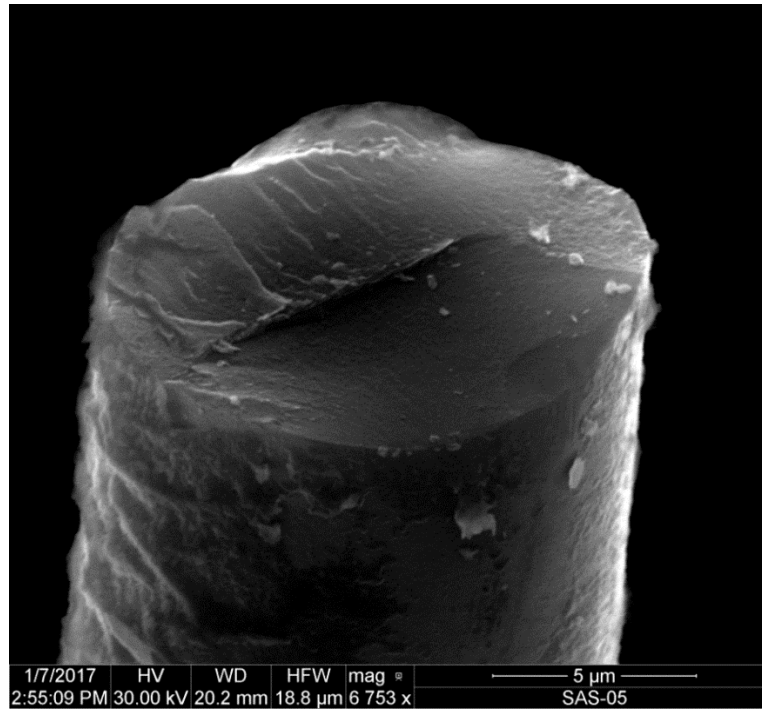


Figure 63. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 16” examining lower fracture surface of a fiber ($\sigma_{cr} = 497$ MPa, $t = 63.84$ h).

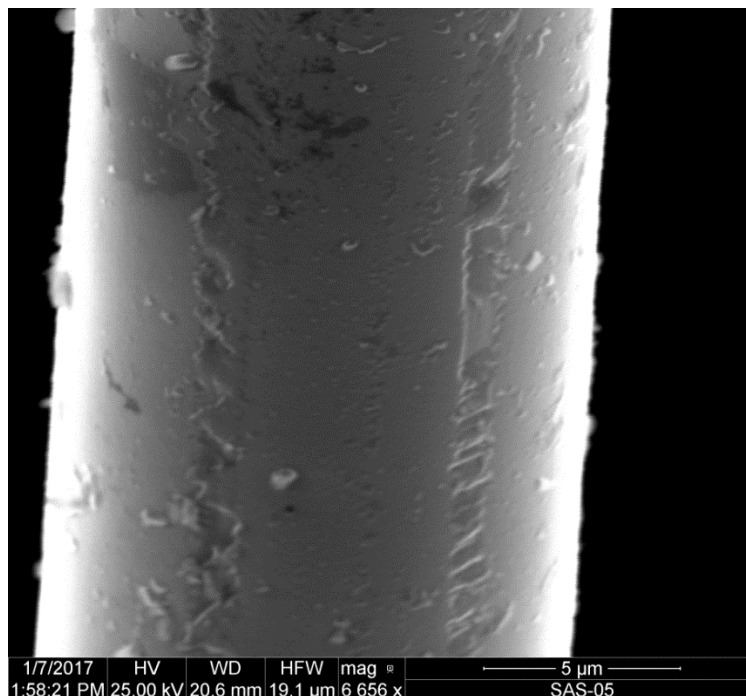


Figure 64. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 16” examining fiber surface on upper portion of a fiber. Note longitudinal fiber-fiber contact scale ($\sigma_{cr} = 497$ MPa, $t = 63.84$ h).

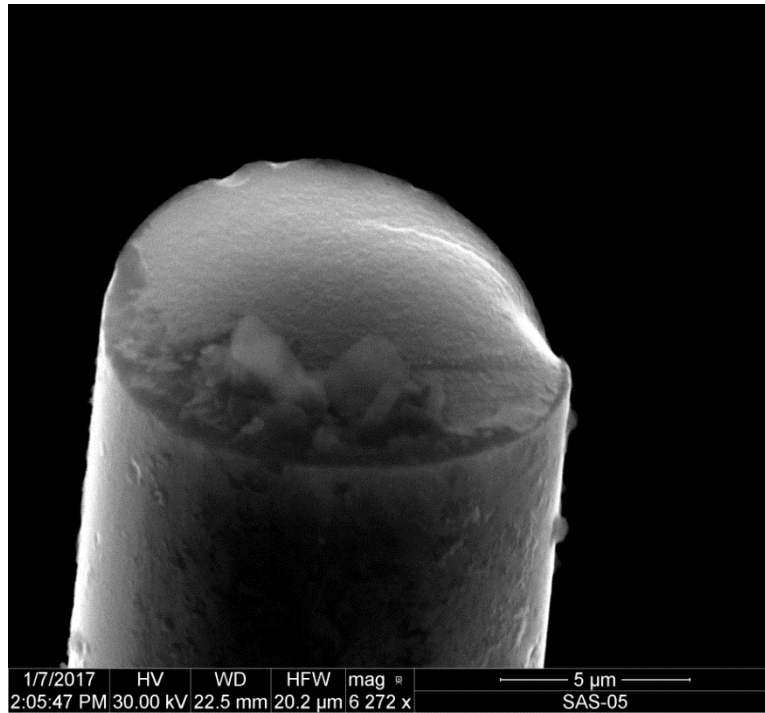


Figure 65. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 16” examining upper fracture surface of a fiber ($\sigma_{cr} = 497$ MPa, $t = 63.84$ h).

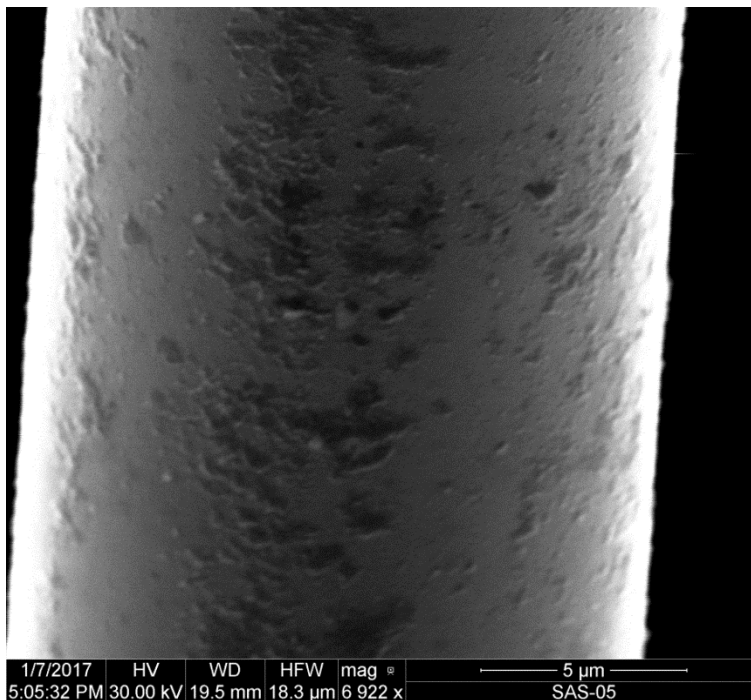


Figure 66. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 13” examining fiber surface on lower portion of a fiber ($\sigma_{cr} = 600$ MPa, $t = 55.21$ h).

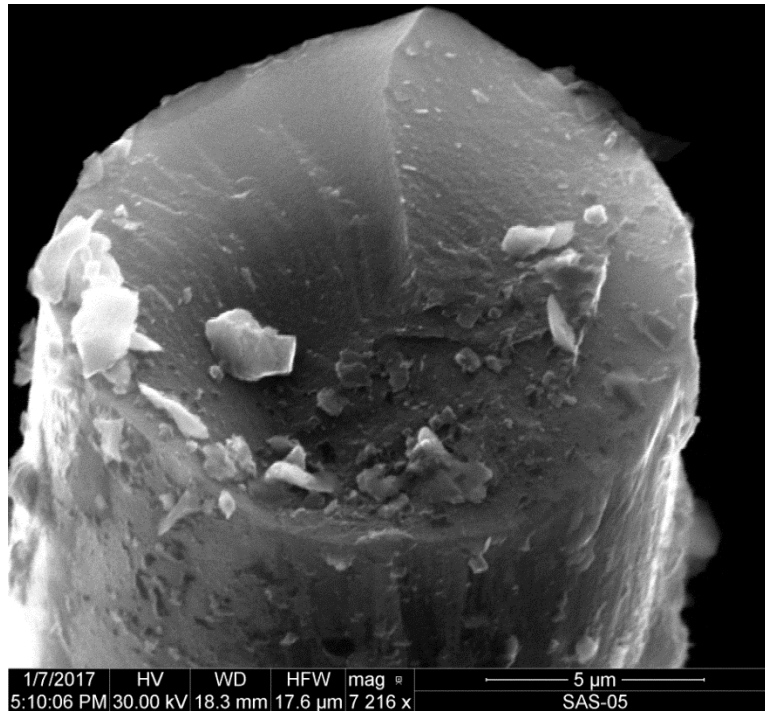


Figure 67. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 13” examining lower fracture surface of a fiber ($\sigma_{cr} = 600$ MPa, $t = 55.21$ h).

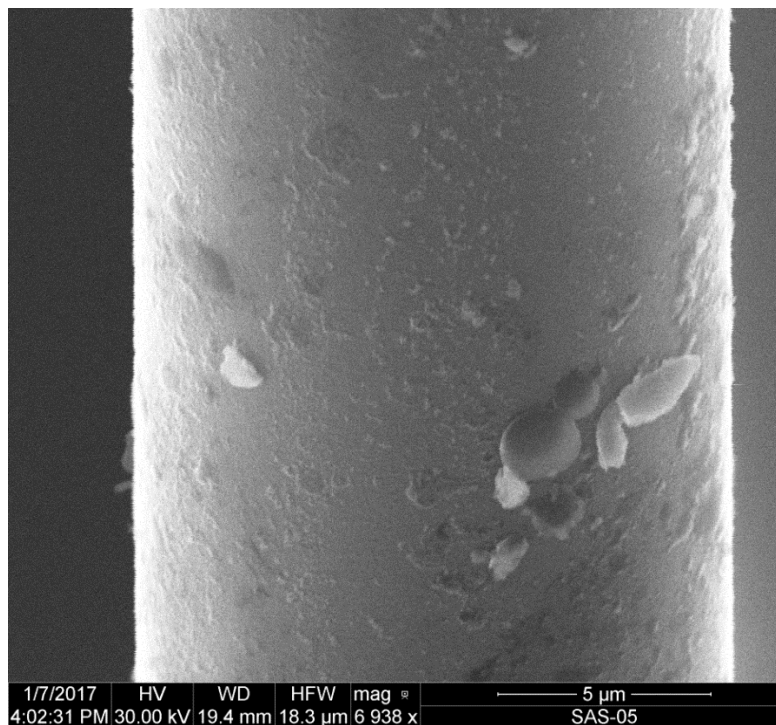


Figure 68. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 13” examining fiber surface on upper portion of a fiber ($\sigma_{cr} = 600$ MPa, $t = 55.21$ h).

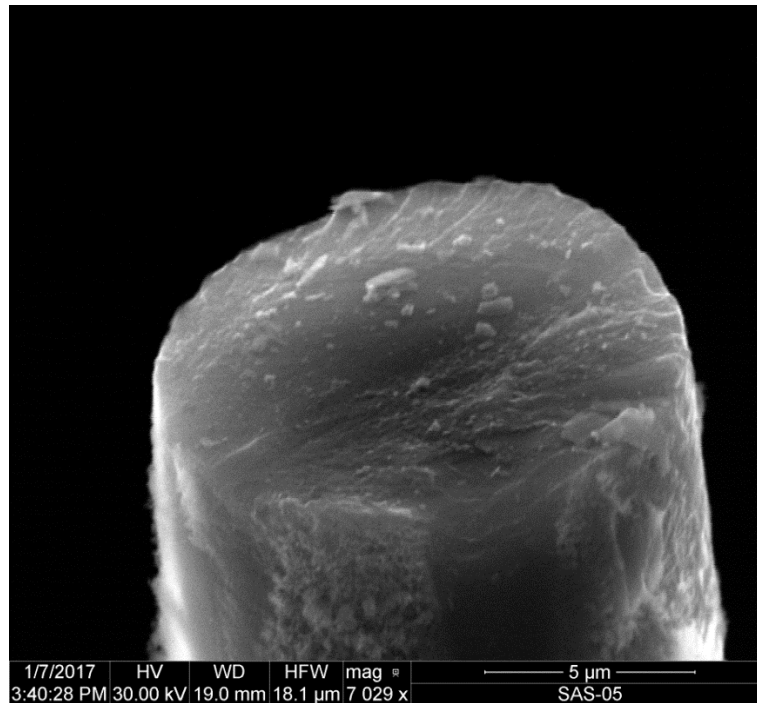


Figure 69. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 13” examining upper fracture surface of a fiber ($\sigma_{cr} = 600$ MPa, $t = 55.21$ h).

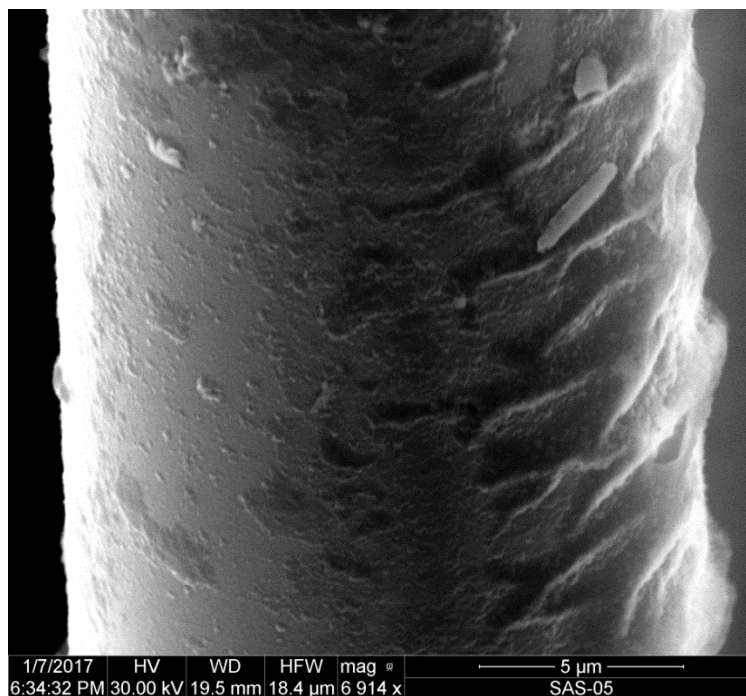


Figure 70. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 11” examining fiber surface on lower portion of a fiber. Note anomalous longitudinal scale ($\sigma_{cr} = 736$ MPa, $t = 21.5$ h).

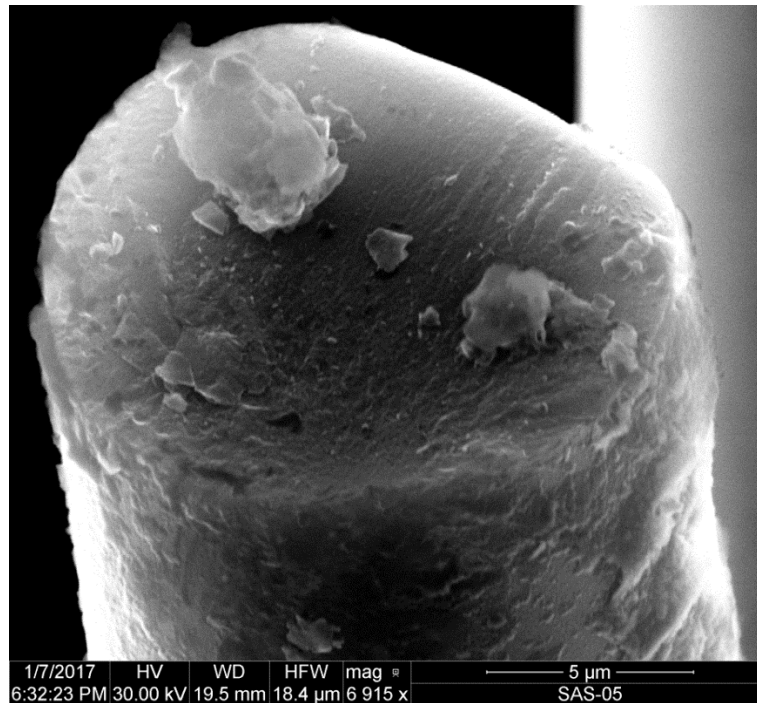


Figure 71. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 11” examining lower fracture surface of a fiber ($\sigma_{cr} = 736$ MPa, $t = 21.5$ h).

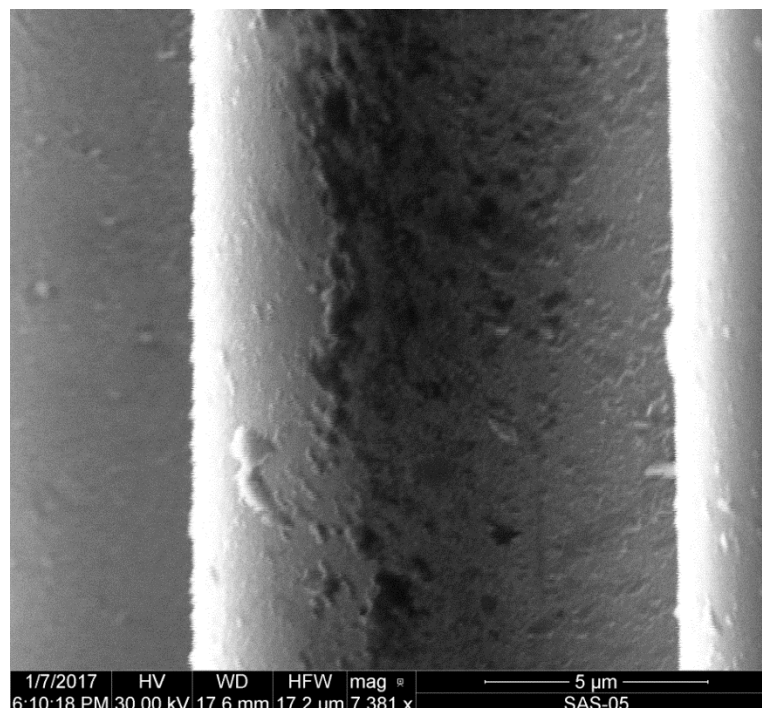


Figure 72. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 11” examining fiber surface on upper portion of a fiber. Note anomalous longitudinal scale ($\sigma_{cr} = 736$ MPa, $t = 21.5$ h).

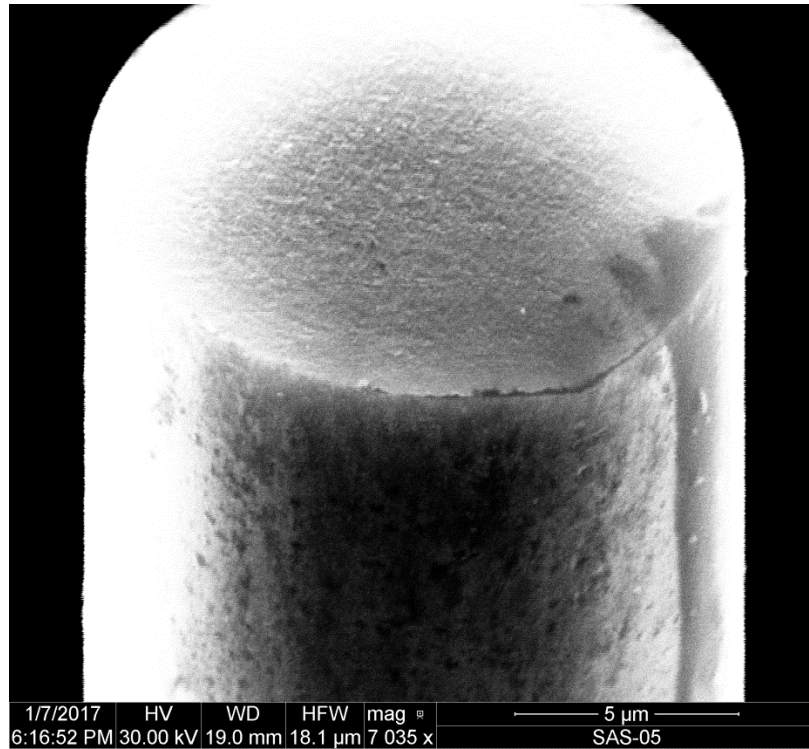


Figure 73. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 11” examining upper fracture surface of a fiber ($\sigma_{cr} = 736$ MPa, $t = 21.5$ h).

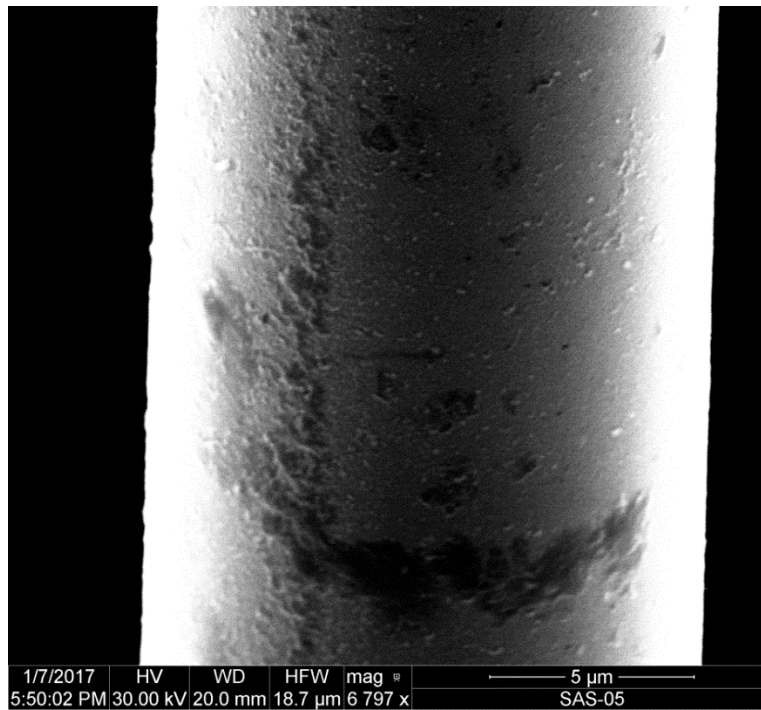


Figure 74. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 12” examining lower surface on lower portion of a fiber. ($\sigma_{cr} = 654.5$ MPa, $t = 14.1$ h).

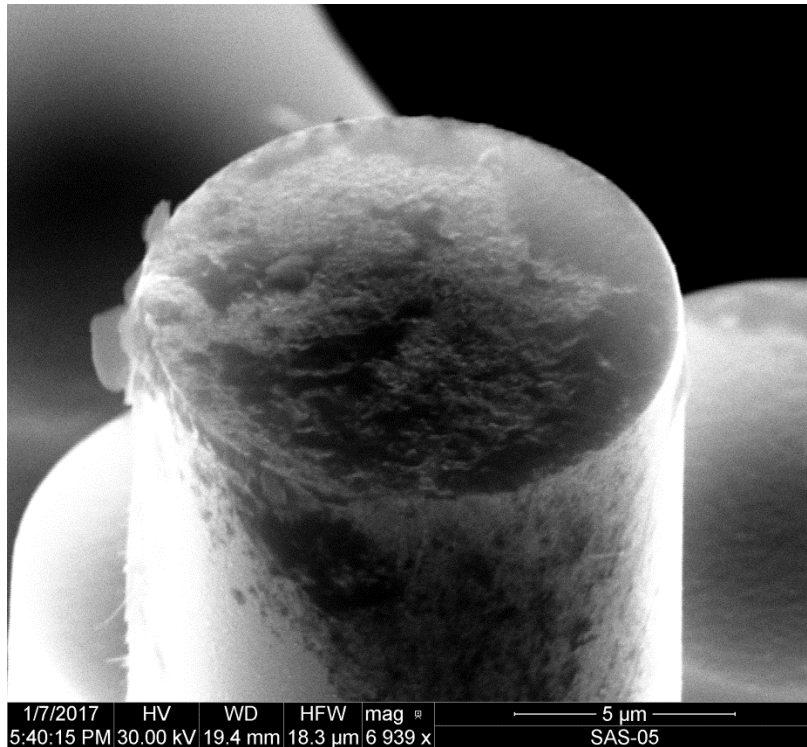


Figure 75. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 12” examining lower fracture surface of a fiber (654.5 MPa, t = 14.1 h).

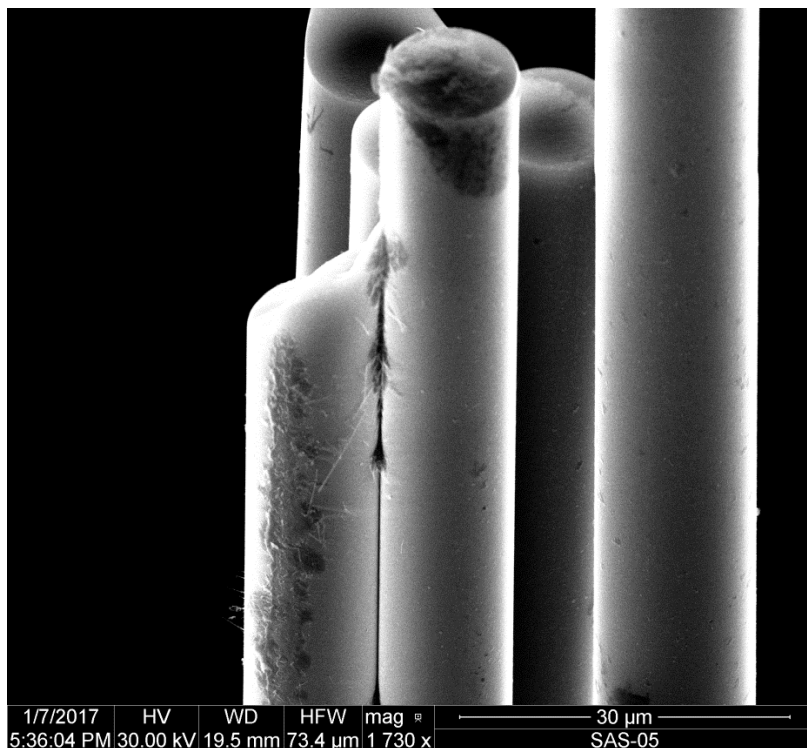


Figure 76. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 12” examining lower fiber tow (654.5 MPa, t = 14.1 h).

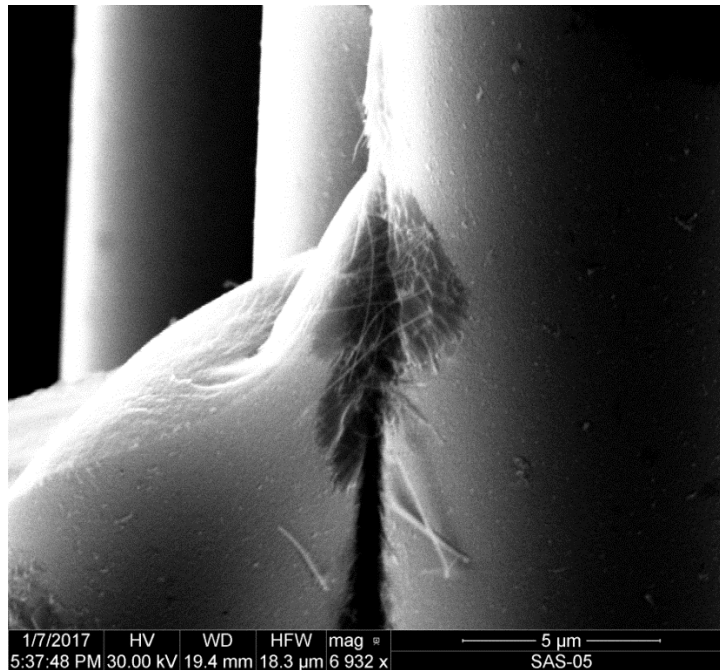


Figure 77. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 12” examining lower fiber tow. Note signs of unsaturated steam exposure (spider webbing/silica leeching at interface) (654.5 MPa, t = 14.1 h).

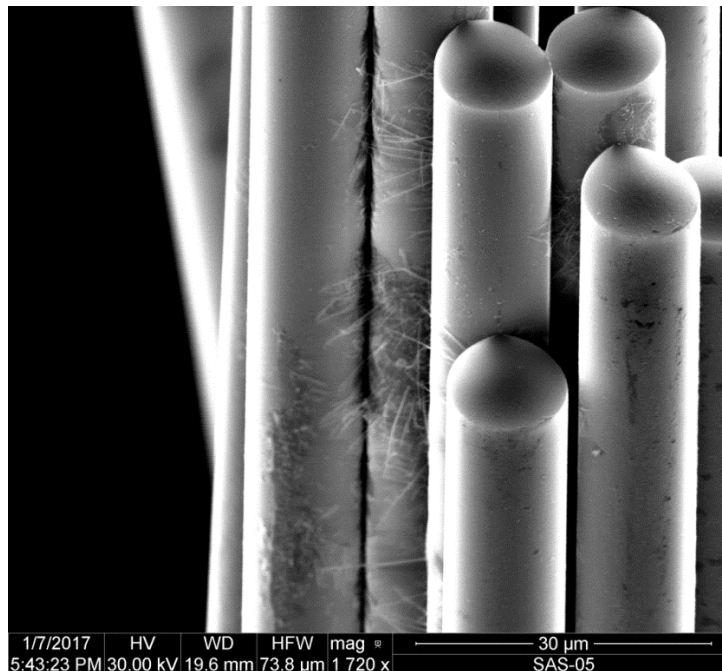


Figure 78. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 12” examining lower fiber tow. Note signs of unsaturated steam exposure (spider webbing/silica leeching at interface) (654.5 MPa, t = 14.1 h).

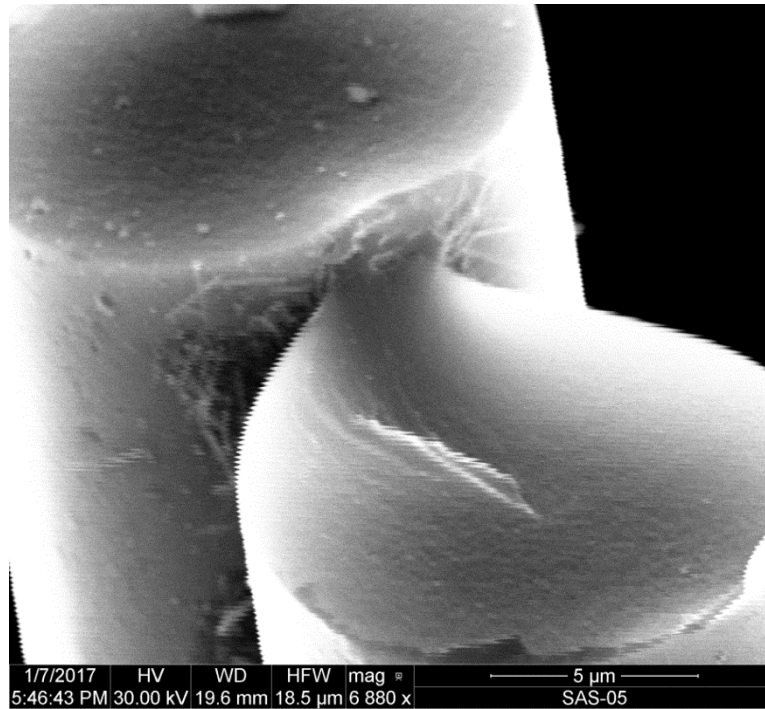


Figure 79. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 12” examining lower fiber tow. Note signs of unsaturated steam exposure (spider webbing/silica leeching at interface) (654.5 MPa, t = 14.1 h).

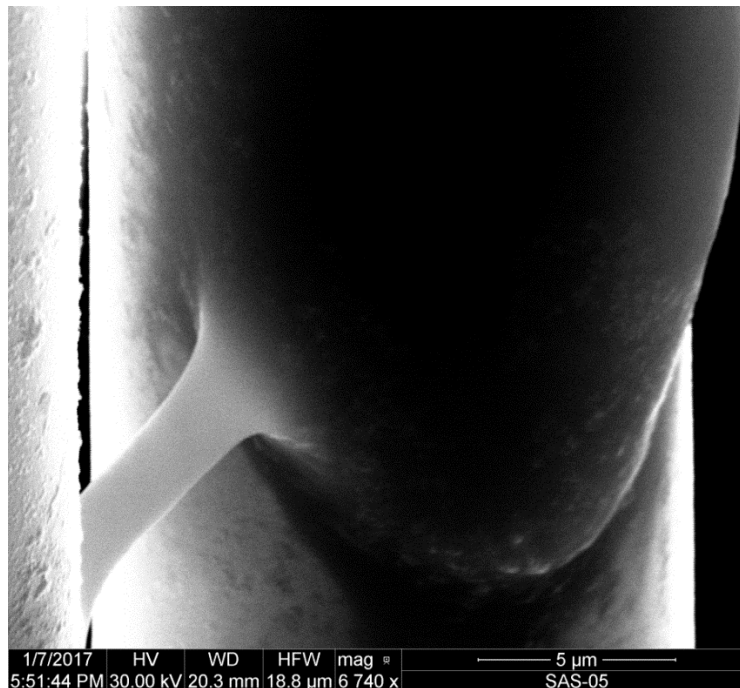


Figure 80. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 12” examining lower fiber tow. Note signs of unsaturated steam exposure (spider webbing/silica leeching at interface) (654.5 MPa, t = 14.1 h).

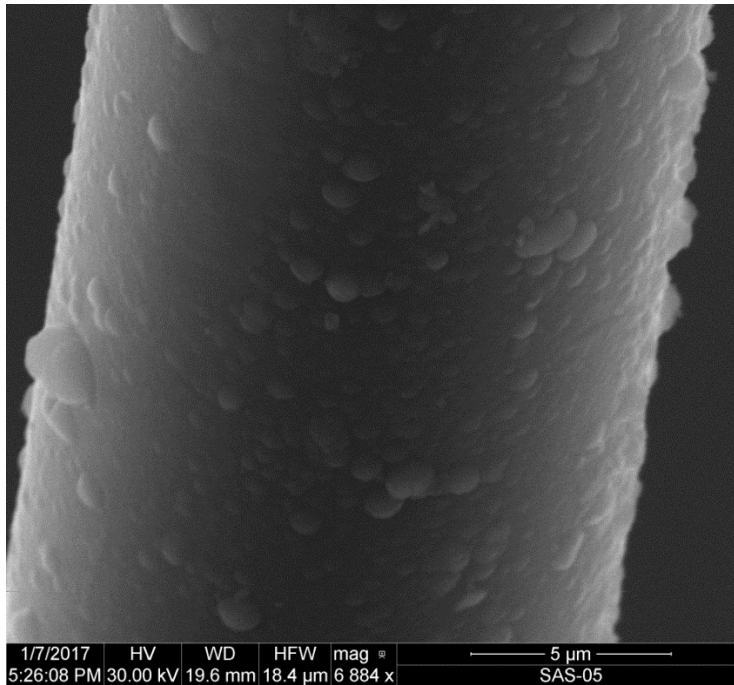


Figure 81. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 12” examining upper fiber tow. Note signs of saturated steam exposure (dewetting [30]/silica deposition) (654.5 MPa, t = 14.1 h).

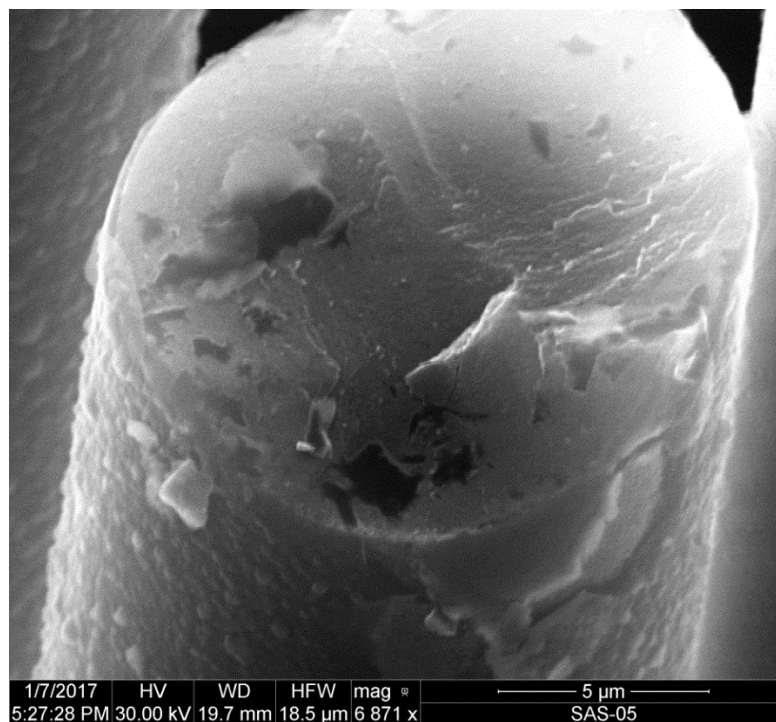


Figure 82. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 12” examining upper fiber tow. Note signs of saturated steam exposure (dewetting [30]/silica deposition) (654.5 MPa, t = 14.1 h).

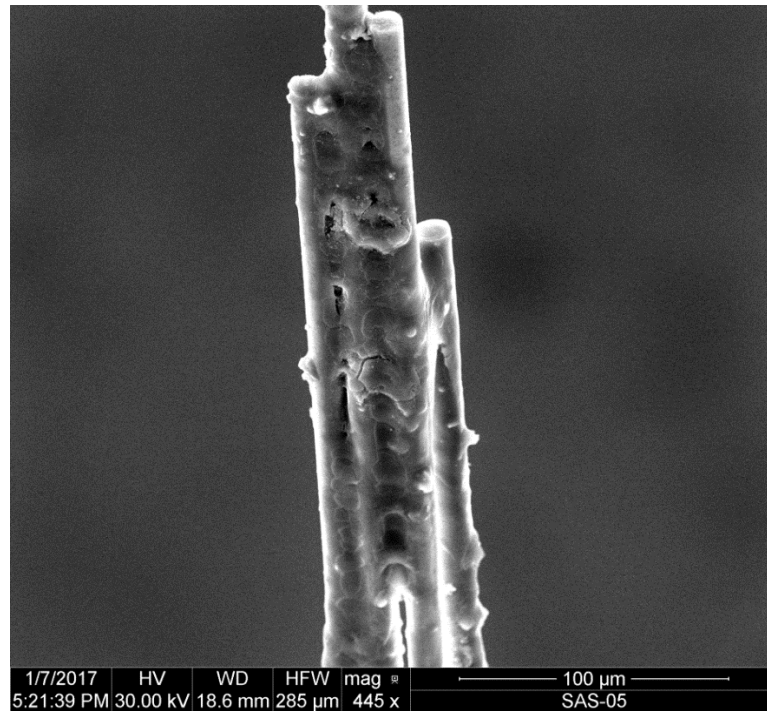


Figure 83. SEM micrograph of the Hi-Nicalon™ S specimen “Steam 12” examining upper fiber tow. Note signs of saturated steam exposure (pronounced silica scale deposition, also reported by Roberston [19]) (654.5 MPa, t = 14.1 h).

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