Importance of Measuring SiC Fiber Diameter Prior to Conducting Fiber Stress Rupture / Creep Tests?

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Outline

• Background and Research Objectives
• SiC fiber testing at NASA GRC
• GRC interest in characterizing SiC fiber durability at intermediate temperature (815°C)
• Laser measurement of fiber diameter
• Consideration of importance of measuring the diameter of each SiC fiber prior to stress rupture and creep testing
• Testing Procedure – Fiber Stress Rupture Testing in Air
• Summary and Conclusions
Background

*Distribution of fiber diameters observed*

CVI SiC/SiC CMC Reinforced With Sylramic™-iBN SiC Fiber
# Background

A significant amount of SiC fiber creep / stress rupture data has been generated and reported in the past by various researchers.

Researchers have often used an *accepted average diameter* value (fiber type-dependent) to determine the load that will be applied to a fiber to generate a specific stress during testing.

*This is due to:*

- the difficulty of measuring fiber diameter,
- the amount of time required to make accurate measurements,
- concerns about the potential for damaging the fiber during the measurement, and
- concerns about the “validity” of using an *average measured diameter.*
**Background**

**What is the Benefit of Measuring Fiber Diameter?**

**Premise**
Testing fibers (creep / stress rupture) without knowing their “actual” diameter, and using an average value, introduces error / scatter unless they have very similar diameters (minimal fiber-to-fiber variation in diameter).

**Reality?**
Measuring the fiber diameter over a specific length of the fiber (e.g., the gage region) will provide an indication of the average value within that range, and the amount of diameter variation¹.

This could (or should) help reduce the amount of scatter in the test data, but we still won’t know the diameter at the point where failure occurs.

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Research Objectives

1) We are currently assessing the difference between using an *average fiber diameter* in stress rupture and creep testing / data analysis vs. using a *measured ≈ “known” diameter for each fiber*. *This is related to*....

2) GRC *fiber stress rupture testing* that is being performed in air and vacuum to assess the effect of environment and stress on fiber longevity *in support of GRC modeling of SiC/SiC CMC stress rupture life at intermediate T*. 

![Image of fiber microstructure]
Assessment of the quality of an As-produced SiC fiber

GRC has relied on room temperature tensile testing of SiC fiber tow (1” gage) to assess the as-produced quality / strength; not on testing of individual fibers

Thus, we essentially test all of the fibers in the tow, and there is no need to measure the fiber diameter to determine failure stress

Determine ave. max. load: 100 N ave. (5 tests), for example

This approach has generally yielded an acceptable means of assessing the quality of a specific type of fiber over a long period of time (as different lots or batches of fiber are delivered)

GRC can also perform FF strength tests on single fibers
SiC Fiber Testing at NASA GRC

**Creep / stress rupture behavior of an As-produced SiC fiber**

GRC has been performing elevated temperature tensile creep testing of individual SiC fibers (air: 4” gage, Ar / Vacuum: 4” gage)\(^2\)

Each test can last hundreds of hours. Thus far, an average fiber diameter has been used to calculate stress. Fiber remnants saved.

\(^2\) A. Almansour, “SiC fibers and SiC/SiC ceramic matrix minicomposites damage behavior,” Advanced Ceramic Matrix Composites: Science and Technology of Materials, Design, Applications, Performance and Integration (Engineering Conferences International), Santa Fe, NM Nov. 2017
Considering making 3 measurements of diameter per fiber prior to testing:

L = 7”, 8.5”, and 10”

Fibers are 12.75” long

LVDTs

Loading Stage

5 Fiber Rig

Stress rupture / creep testing of 5 fibers simultaneously in air
SiC Fiber Testing at NASA GRC

5 Fiber Rig

Attach weights

Close-up view of LVDTs
GRC Interest in Characterizing SiC Fiber Durability at Intermediate Temperature (815°C)

- GRC is conducting fiber stress rupture testing in air and vacuum to assess the effect of environment and stress on fiber longevity at 815°C (and subsequently 1200°C). This will support CMC durability modeling \(^3\).

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**Example for Hi-Nicalon: \( \sigma \) of 800 MPa**

- Max. failure time of about \( 4 \times 10^5 \) s = 111 hr (< 5 days)
- \( 10^5 \) sec = 27.8 hr

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**Fig. 2.** Time-to-failure versus applied stress of Hi-Nicalon single fibers (gold circles) and tows (black squares) at 800°C. Data from Gauthier and Lamon [11].

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GRC Interest in Characterizing SiC Fiber Durability at 815°C and 1200°C

- Start with stress rupture testing of Hi-Nicalon™ in air
- Focus on stress rupture testing of Hi-Nicalon S™ in air and vacuum
  - 815°C at 6 - 8 target stress magnitudes (15 replicas each)
  - 1200°C at 6 - 8 target stress magnitudes (15 replicas each)
Measurement of the diameter of individual SiC fibers

- NASA GRC has begun using a laser fiber diameter measurement system
- We are considering eventually characterizing each SiC fiber prior to testing in creep / stress rupture
AFRL/RXLN
Laser Fiber Measurement

Schematic view:

Fiber in grips

Screen with ruler

20.7 cm

X = 50.8 cm (example)

Uniphase Model 1122P
2 mW Class 3A HeNe laser
\( \lambda = 623.8 \text{ nm} \)

Fiber diameter, \( d \):

\[
d = \frac{\lambda}{\sin \left( \arctan \left( \frac{D}{X} \right) \right)}
\]

For ~10\( \mu \text{m} \) fibers, determination of \( D \) to within 0.05 cm (0.5 mm) results in ~200 nm resolution in \( d \)

Fraunhofer diffraction
Laser Diffraction—Used to Measure Fiber Diameter

Fraunhofer diffraction

Diffraction Pattern Projected on Screen – Measure spacing (D) between pattern minima

D = 3 cm

Moveable Screen
Ceramic Fiber Diameter Variation

08 Aug 2013

Santeri Potticary
Ceramic M&P RT
Air Force Research Laboratory
Hi Nicalon S™ (Potticary Data)
Diameter Meas. Along Fiber Length

Sample 097

Small variation in d
Ave. Dia.= 12.5 μm

Sample 102

Ave. Dia.= 12.6 μm

Sample 105

Ave. Dia.= 12.6 μm

Sample 111

Ave. Dia.= 12.8 μm

- 4 samples have a similar ave. diameter
- Fiber diameter usually within ± 2 μm of ave. diameter
**Fraunhofer diffraction**

- Currently planning to use screen marked with 2 cm intervals (via tape)

D has the greatest impact on the accuracy of the diameter measurement.
Table Showing the Effect of Distance “X” and Pattern Width “D”

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X is the distance from the fiber to the screen/grid
Need accurate measurement of D

Note the effect of D on r²

Fiber diameter, d:

\[
d = \frac{\lambda}{\sin\left(\arctan\left(\frac{D}{X}\right)\right)}
\]
Consideration of the Importance of Measuring the Diameter of Each SiC Fiber Prior to Stress Rupture Testing

CVI SiC/SiC Reinforced with Sylramic™–iBN SiC Fiber

- Ave. dia. ≈ 10 µm        Range:  6 to 16.6 µm
Example of How Fiber Diameter Influences Stress

*Equivalent load applied to 3 different fibers*

Assumed average dia. for Hi-Nicalon™

- **14 µm**
  - $r^2 = 49 \mu m^2$
  - $\sigma = 600$ MPa

- **12 µm**
  - $r^2 = 36 \mu m^2$
  - $\sigma = 817$ MPa
  - 36% higher

- **16 µm**
  - $r^2 = 64 \mu m^2$
  - $\sigma = 459$ MPa
  - 24% lower

Assume uniform cross section in gage region
If Three Diameter Measurements are Made per Fiber, How Would We Use Those Values?

**Determination of load to apply to individual SiC fiber**

- Our furnace hot zone is roughly 3.5 in. (uniform max. T).
- Assume you make 3 measurements to look at the variability.
- Then, do you focus on the minimum measurement (where the highest stress should be) or just average the measured diameters?
- Example: measured values are 12, 14, and 16 µm.
- Ave.: 14 µm, but minimum is 12 µm.
Consideration of the Importance of Measuring the Diameter of Each SiC Fiber Prior to Stress Rupture Testing

- Continue to review the literature on fiber diameter measurements and distributions and further discuss the benefits of making these measurements.
Notes, Procedure, and Issues

- Furnace T was profiled (good uniformity)
- LVDTs calibrated
- Extract fibers from de-sized tow *
- Mount fibers (apply tabs to ends of fibers—12.75” apart)
- Measure diameter of fibers (ideally)
- Position fibers in furnace and attach loads (but do not load)
- Heat to T (815°C)
- Apply loads to fibers by lowering stage *
- Run to failure to determine life at that stress
- Unload and store fiber fragments for additional characterization
- Data analysis (expect to see more creep at higher temperatures)

Currently need to:
- Confirm accuracy of Laser Diameter Measurements using SEM
  - * Improve ability to apply higher stresses to fibers (800 MPa and higher)
  - * Determine the best way to extract fibers from the tow (least damaging to fiber)
- Characterize the extent of SiC fiber oxidation occurring during testing
- See how time-consuming it is to measure fiber diameter
- Maintain dialogue with GRC and AFRL modelers
Summary and Conclusions

- GRC is conducting fiber stress rupture testing in air and vacuum to assess the effect of environment and stress on fiber longevity at 815°C (and subsequently 1200°C).

- This is being done to support GRC modeling of SiC/SiC CMC stress rupture life at those temperatures.

- AFRL has teamed with GRC to share their expertise and to collaborate in this research effort.

- GRC is interested in determining the benefit of conducting fiber diameter measurements. That capability can also be applied to other GRC SiC fiber creep studies.

- We are trying to optimize our fiber test sample preparation and sample loading procedure.

- Comments and advice from other researchers are welcome.
Improvement in Accuracy and Speed of Fiber Strength Measurement

Fraunhofer diffraction: \( a \sin \theta = m \lambda \) for a thin slit or thin solid object (Babinet’s Principle)

For \( L \gg d \) in the diagram below: \( d = \frac{\lambda L}{\Delta x} \)

Fiber diameters \(~10 \, \mu\text{m} \); measurement error improved from 10% to 2%

Implemented RXCC laser diffraction method as part of quality control for improved accuracy and rate of fiber strength measurement