

# **Creep/Stress Rupture Behavior of 3D Woven SiC/SiC Composites with Sylramic-iBN, Super Sylramic-iBN and Hi-Nicalon-S Fibers at 2700F in Air**

**R.T. Bhatt**

**Ohio Aerospace Institute**

**NASA Glenn Research Center**

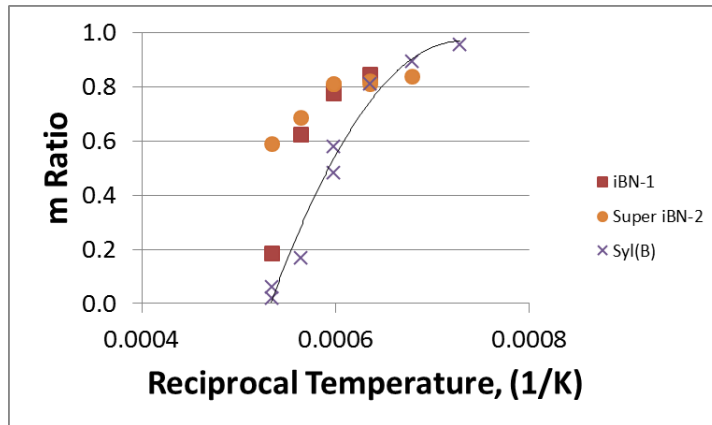
**Cleveland, OH**

**Support from NASA Transformational Tools and Technologies Program**

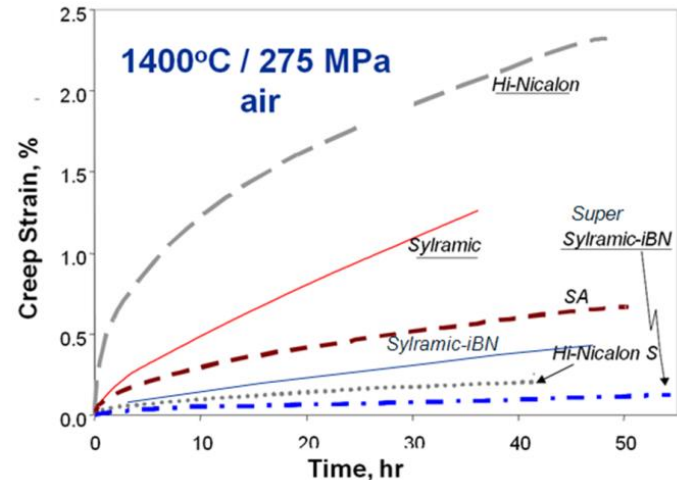
**Presented at the 41<sup>th</sup> Annual Conference on Composites, Materials, and Structures, Cocoa Beach/Cape Canaveral, Florida, January, 2017**

# Background

## Bend Stress Relaxation (BSR) Data



## Tensile Creep Data



- Both BSR and individual filament creep tests indicate that super Sylramic-iBN fibers exhibit better creep resistance than Sylramic-iBN fibers, and the creep resistance of Hi-Nicalon-S fibers is similar to that of super Sylramic-iBN fibers, but the potential benefits and upper use capability of 3D woven SiC/SiC composites with (CVI+PIP) hybrid with super Sylramic-iBN fibers or Hi-Nicalon-S fibers have not yet been investigated.
- According to composite theory, in-plane matrix cracking and ultimate tensile strengths of the composite increase with increasing fiber fraction in the loading direction. However influence of fiber fraction on creep durability is not clearly understood specifically in a composite system with creep mismatch ratio (creep rate of the fiber/creep rate of the matrix) is  $>1$ .

# Objective and Approach

## Objective:

### Short term:

Determine creep durability of 3D woven SiC/SiC composites with super Sylramic-iBN and Hi-Nicalon-S fibers at 2700<sup>0</sup>F in air

### Long term:

Improve durability of 3D woven SiC/SiC composites at temperatures to 2700<sup>0</sup>F by controlling constituent microstructures .

## Approach:

- Fabricate 3D woven SiC/SiC composites with super Sylramic-iBN fibers using the fiber architecture and constitutive vol% similar to those of 3D woven SiC/SiC composites with Sylramic-iBN fibers
- Fabricate 3D woven SiC/SiC composites with Hi-Nicalon-S fibers with fiber architecture similar to 3D woven SiC/SiC composites with super Sylramic-iBN fibers, but with higher fiber fraction.
- Conduct creep tests and determine stress rupture mechanisms of both types of 3D woven composites.

# Material and Characterization

## Fibers/Fiber architectures

Fibers: Sylramic™-iBN#, Super Sylramic™-iBN#, and Hi-Nicalon-S(NOX)

## Fiber Architectures

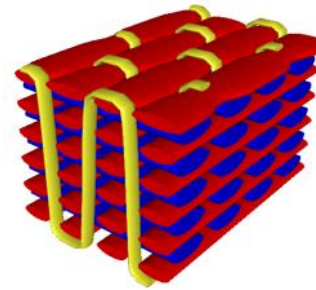
Orthogonal

Modified angle interlock with and without warp stuffers

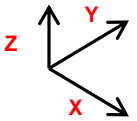
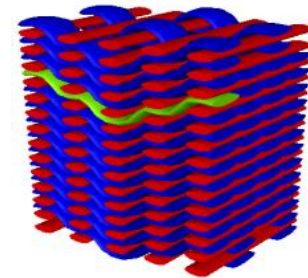
## Fiber Coating

CVI BN/SiC

Orthogonal



Angle Interlock



## Weaving, Coating and PIP Vendors

Weaving: TEAM Inc ( Woonsocket, RI)

Fiber coating: Rolls-Royce High Temperature Composites Inc, Huntington Beach, CA

PIP: Teledyne Scientific & Imaging LLC (Thousand Oaks, CA)

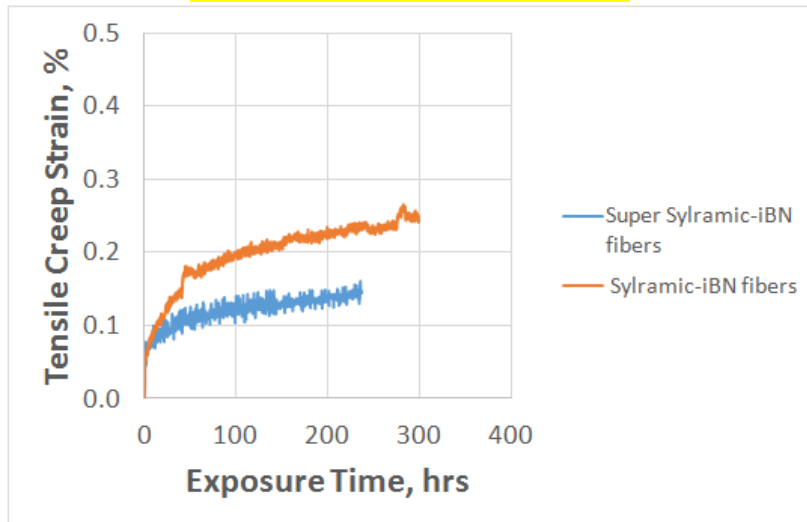
## Characterization: Creep, Tensile, and SEM

Note: # Original Dow Corning Sylramic™ fibers converted to Sylramic™-iBN and super Sylramic™-iBN. Y-Fill stuffers , X-Warp stuffers /weaver, Z- Weaver. The Sylramic and Hi-Nicalon-S fiber preforms were woven with/without a serving fiber, respectively.

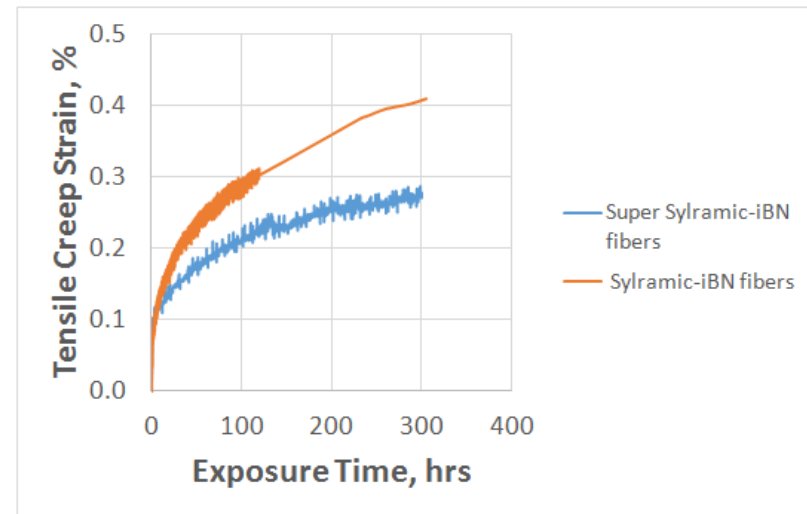
# Influence of Fiber Types on Tensile Creep Behavior of 3D Woven SiC/SiC Composites with (CVI+PIP) Hybrid Matrix

Fiber Architecture: Orthogonal-2 floater ( $V_{fx} \sim 0.07$ ,  $V_{fy} \sim 0.22$ ,  $V_{fz} \sim 0.02$ ,  $V_{CVI/SiC} \sim 0.28$ )

1482°C/69MPa/Air



1482°C/103MPa/Air

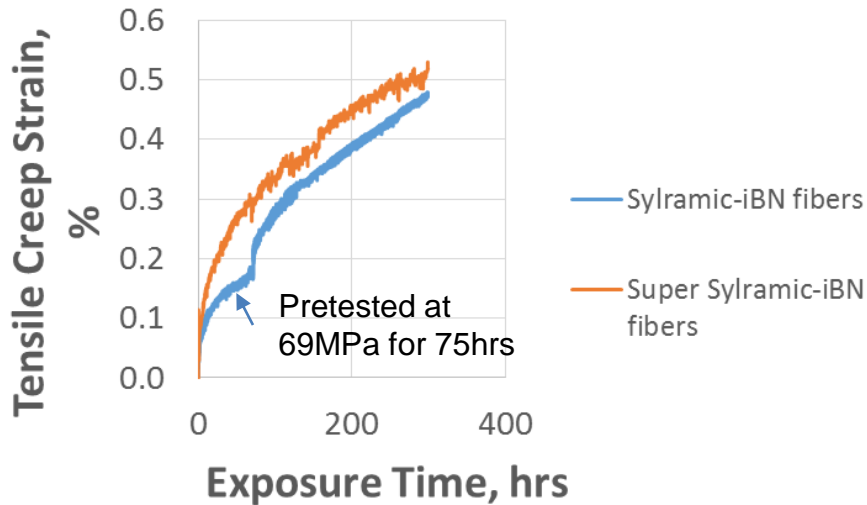


*Note: 3D woven (Orthogonal) SiC/SiC composites with Super Sylramic-iBN fibers show slightly better creep resistance than those containing Sylramic-iBN fibers*

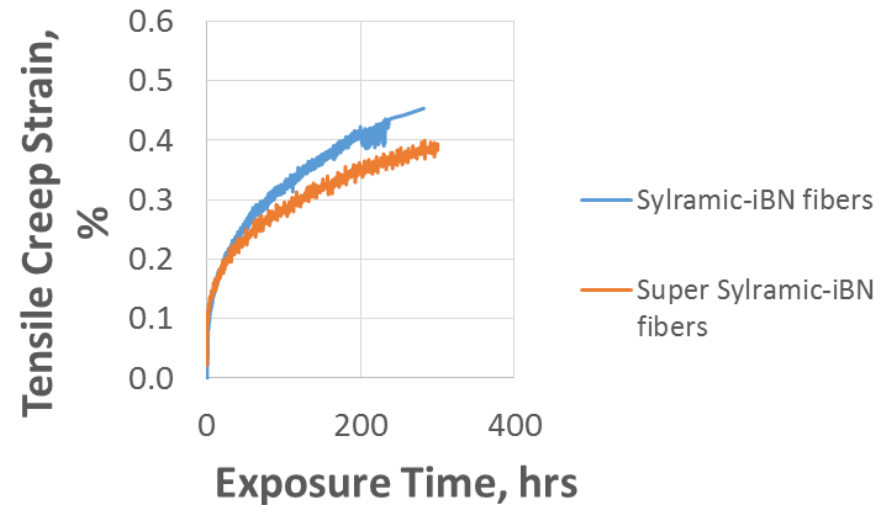
# Influence of Fiber Types on Tensile Creep Behavior of 3D Woven SiC/SiC Composites with (CVI+PIP) Hybrid Matrix

Fiber Architecture: Angle Interlock with Stuffers ( $V_{fx} \sim 0.06$ ,  $V_{fy} \sim 0.22$ ,  $V_{fz} \sim 0.01$ ,  $V_{CVISiC} \sim 0.28$ )

1482°C/138MPa/Air



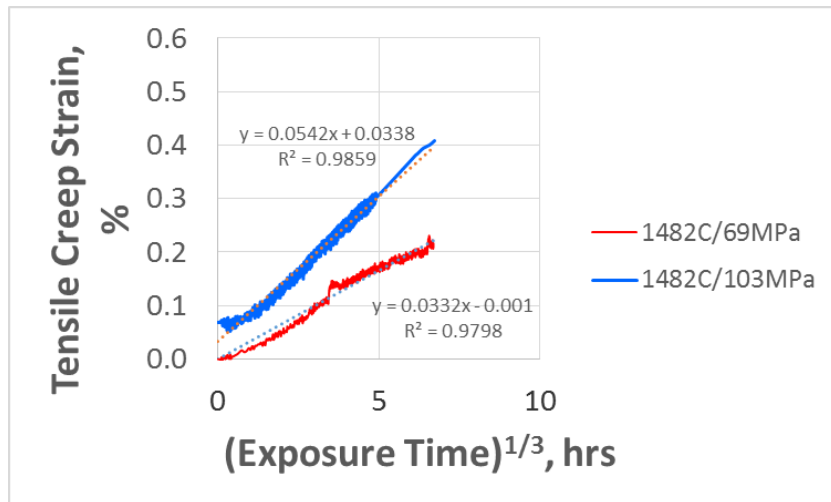
1482°C/120MPa/Air



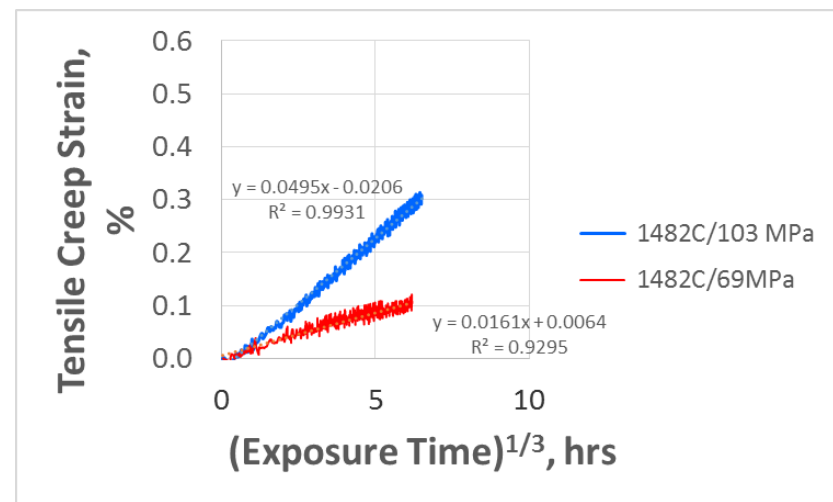
*Note: 3D woven(Angle interlock with stuffers) SiC/SiC composites with Super Sylramic-iBN fibers show nearly the same or slightly better creep resistance than those containing Sylramic-iBN fibers*

# Creep Analysis of 3D Woven SiC/SiC Composites With (CVI+PIP) Hybrid Matrix

Sylramic-iBN fibers



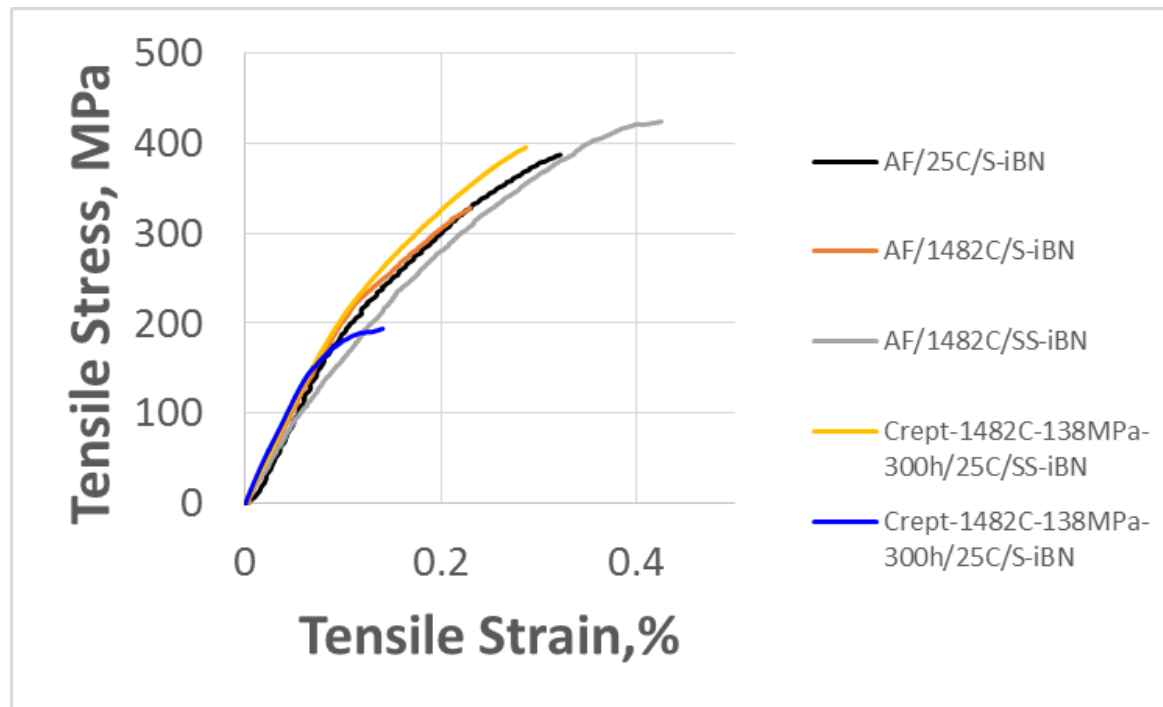
Super Sylramic-iBN fibers



- At 1482<sup>o</sup>C, the accumulated creep strain in 3D woven SiC/SiC composites with (CVI+PIP) hybrid matrix and with Sylramic-iBN or Super Sylramic-iBN fibers linearly varies with  $t^{1/3}$  and shows linear stress dependency. A behavior similar to CVD-derived ultra SCS monofilament SiC fiber and CVD SiC matrix.
- The creep strain can be represented by the equation:  $\epsilon_p (\%) = (C)[\sigma_m] [t \times \exp (-B/T)]^{1/3}$  where  $\sigma_m$  is the stress in CMC matrix,  $T$  is temperature,  $B$  is a constant, and  $t$  is time. The constant  $C$  in the above equation can be determined from the slope of each line in the above figures and the stress.

# Tensile Stress Strain Behaviors of As-Fabricated and Crept 3D SiC/SiC Composites with (CVI+PIP) Hybrid Matrix

Fiber Architecture: Angle Interlock with Stuffers ( $V_{fx} \sim 0.06$ ,  $V_{fy} \sim 0.22$ ,  $V_{fz} \sim 0.01$ ,  $V_{CVISiC} \sim 0.28$ )

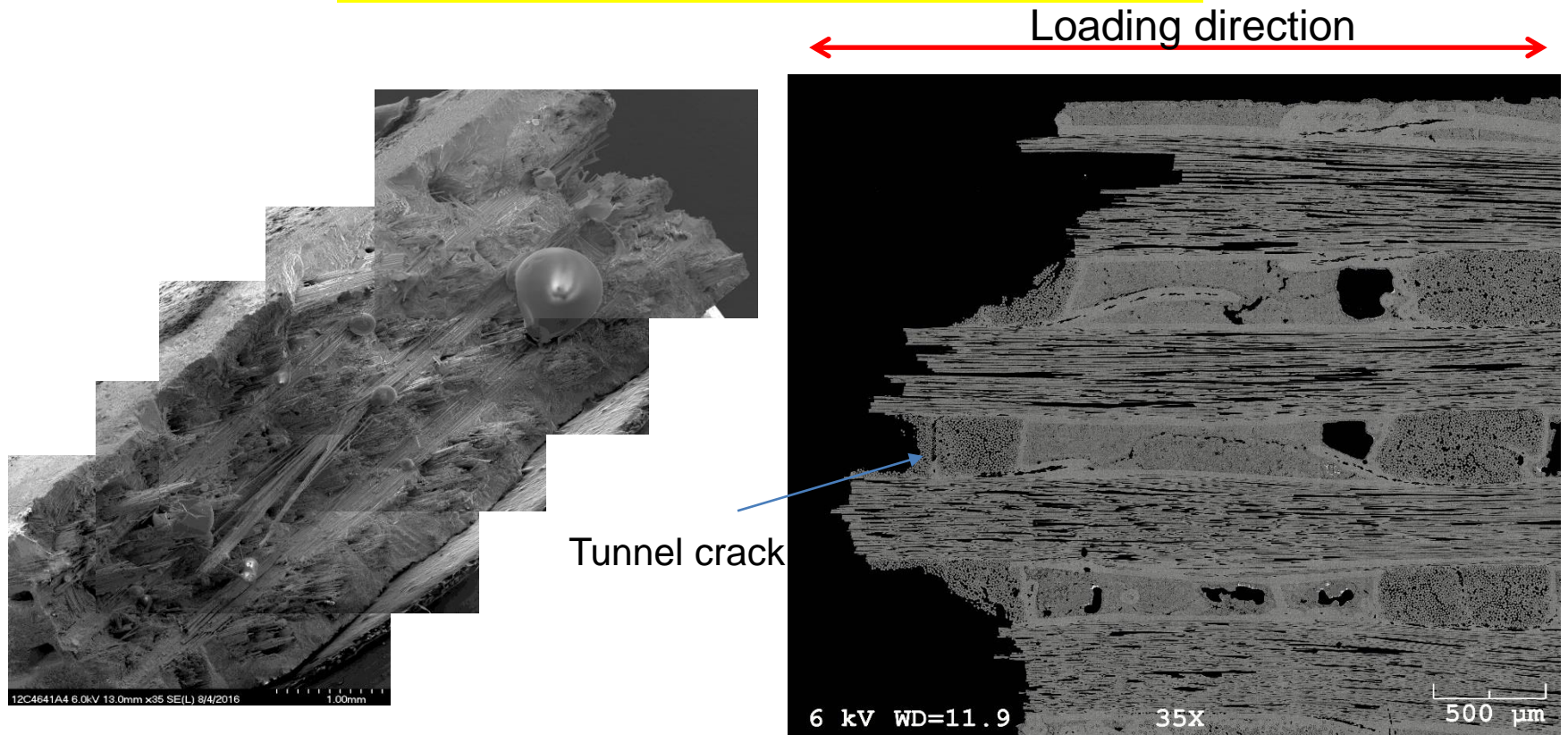


**AF: As-Fabricated, S-iBN: Sylramic™-iBN, SS-iBN: Super Sylramic™-iBN**  
**Note: 3D woven SiC/SiC composites with Sylramic-iBN and Super Sylramic-iBN fibers retain significant % of in-plane tensile properties after 300 hr creep at 1482°C/138MPa**



# SEM Micrographs of the Fracture Surface and Longitudinal Cross Section of a Stress Ruptured 3D Woven SiC/SiC Specimen

Orthogonal: 1482°C/103MPa/277hr/Air

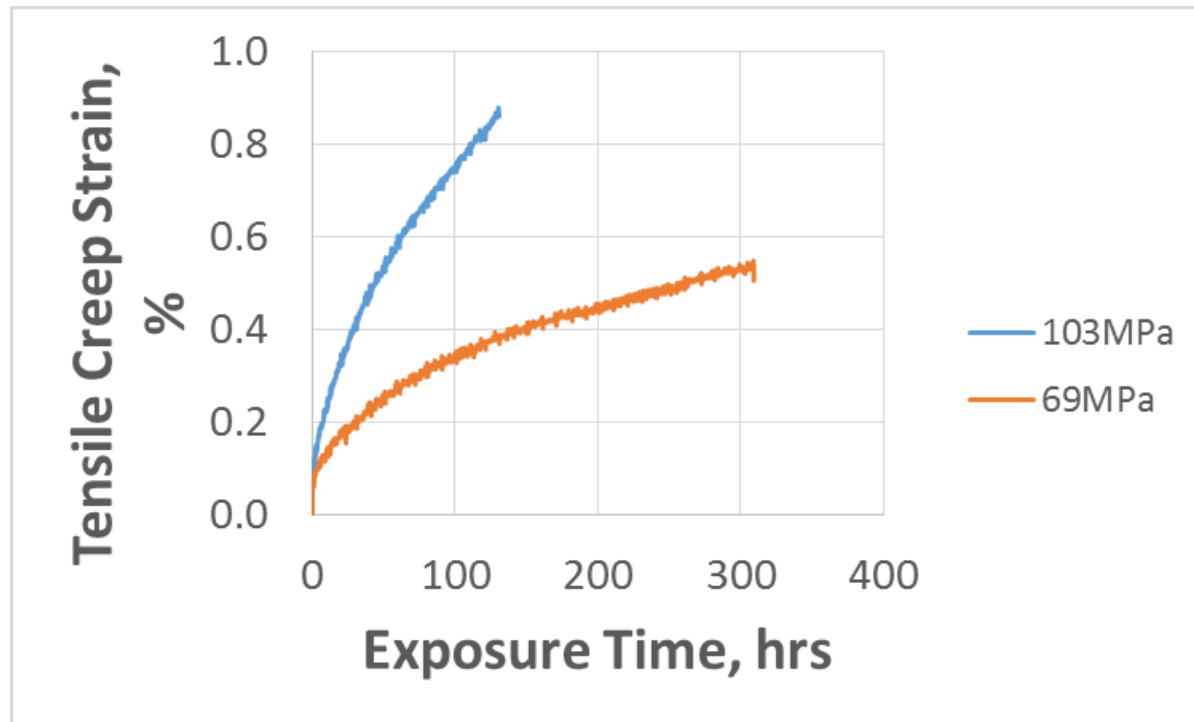


*Note: Crack initiated from the corner/edge of the specimen and then propagated through the tunnel cracks. No secondary cracks found on the longitudinal cross section of the specimen*

# **Tensile Creep Behavior of 3D Woven SiC/SiC Composites with (CVI+PIP) Hybrid Matrix and Hi-Nicalon-S Fibers**

# Tensile Creep Behavior of 3D Woven SiC/SiC Composites with (CVI+PIP) Hybrid Matrix and Hi-Nicalon-S Fibers at 1482<sup>o</sup>C in Air

Fiber Architecture: Angle Interlock without Stuffers ( $V_{fx} \sim 0.14$ ,  $V_{fy} \sim 0.29$ ,  $V_{CVISiC} \sim 0.16$ )



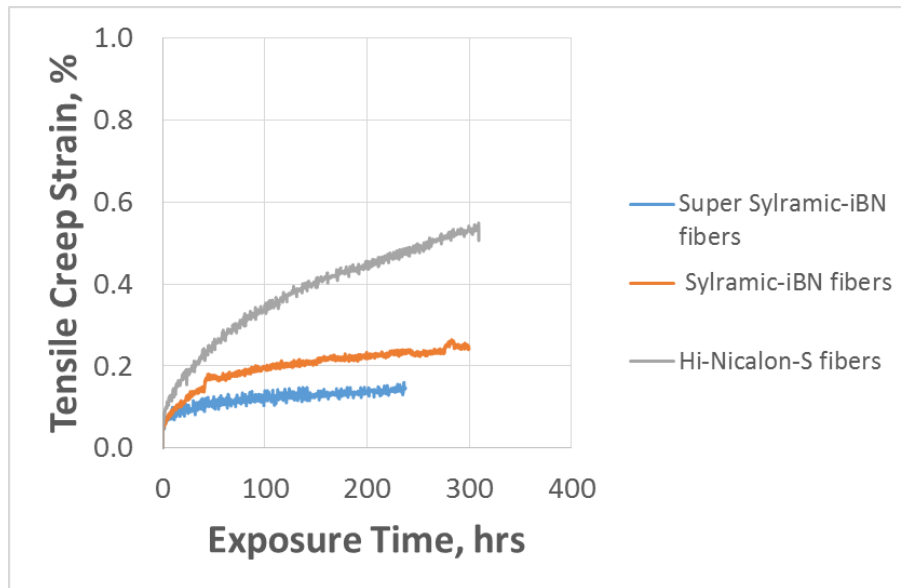
**The 3D woven SiC/SiC composite with Hi-Nicalon-S fibers exhibits fiber dominated creep at 1482<sup>o</sup>C due to (CVI+PIP) matrix cracking during initial creep loading or during creep deformation depending on applied stress. The composite had low volume content of CVI SiC.**

# Comparison of Tensile Creep Behaviors of 3D Woven SiC/SiC Composites with Hi-Nicalon-S and Sylramic Fibers

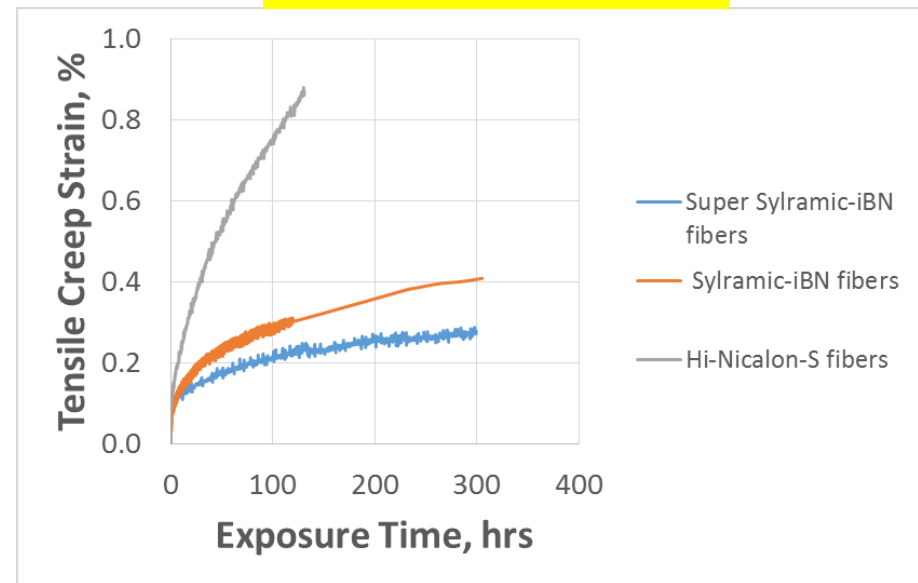
Hi-Nicalon-S: Angle interlock without stuffers ( $V_{fx} \sim 0.14$ ,  $V_{fy} \sim 0.29$ ,  $V_{CVISiC} \sim 0.16$ )

Sylramic-iBN/Super Sylramic-iBN: Orthogonal ( $V_{fx} \sim 0.07$ ,  $V_{fy} \sim 0.22$ ,  $V_{fz} \sim 0.02$ ,  $V_{CVISiC} \sim 0.28$ )

1482°C/69MPa/Air

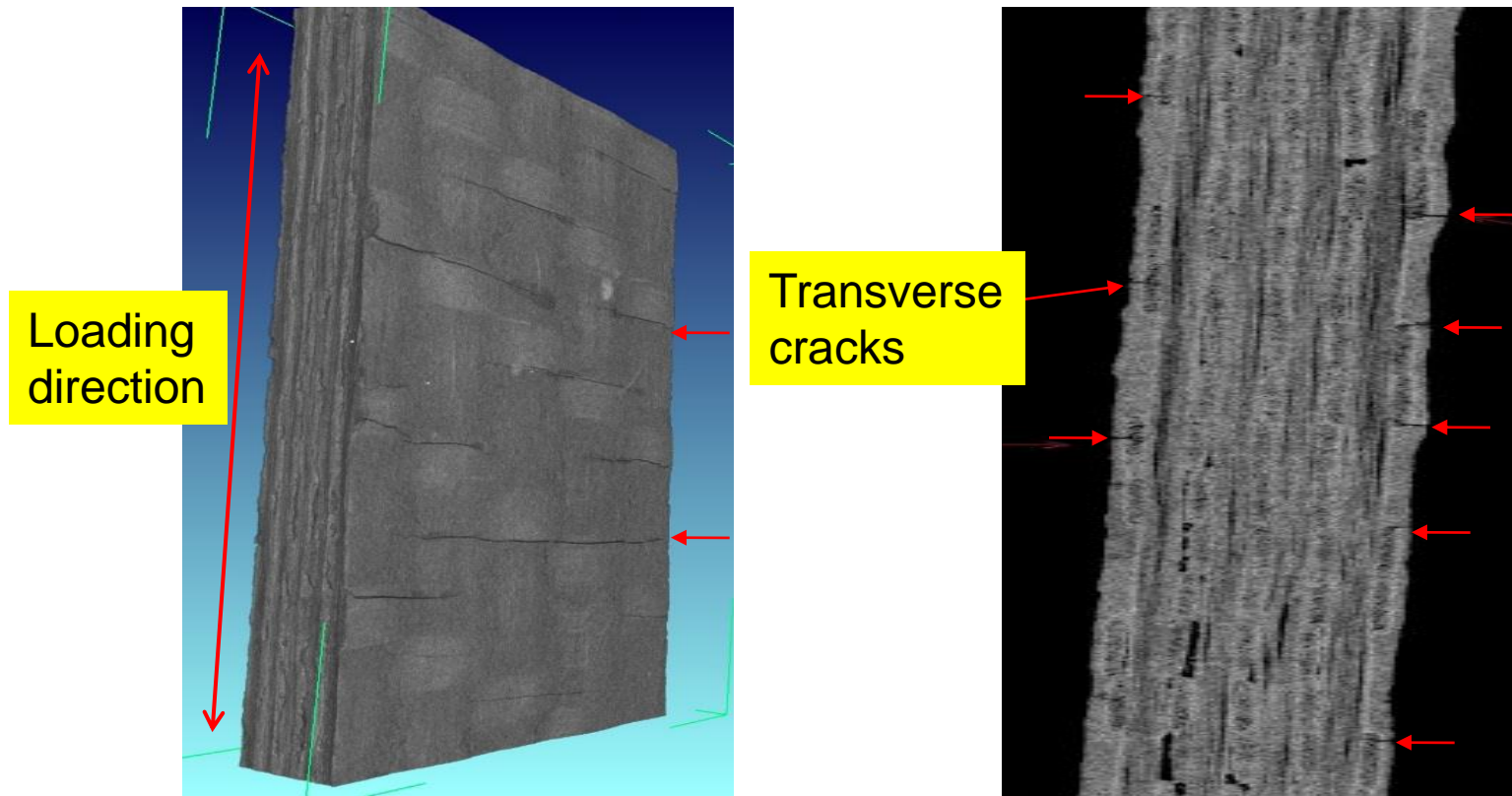


1482°C/103MPa/Air



**The creep resistance of the 3D SiC/SiC composites with Hi-Nicalon-S fibers significantly lower than that of the 3D SiC/SiC composites with Sylramic-iBN or Super Sylramic-iBN fibers because in the former, the long term creep durability is controlled by the fibers and in the latter, it is by the CVI SiC matrix.**

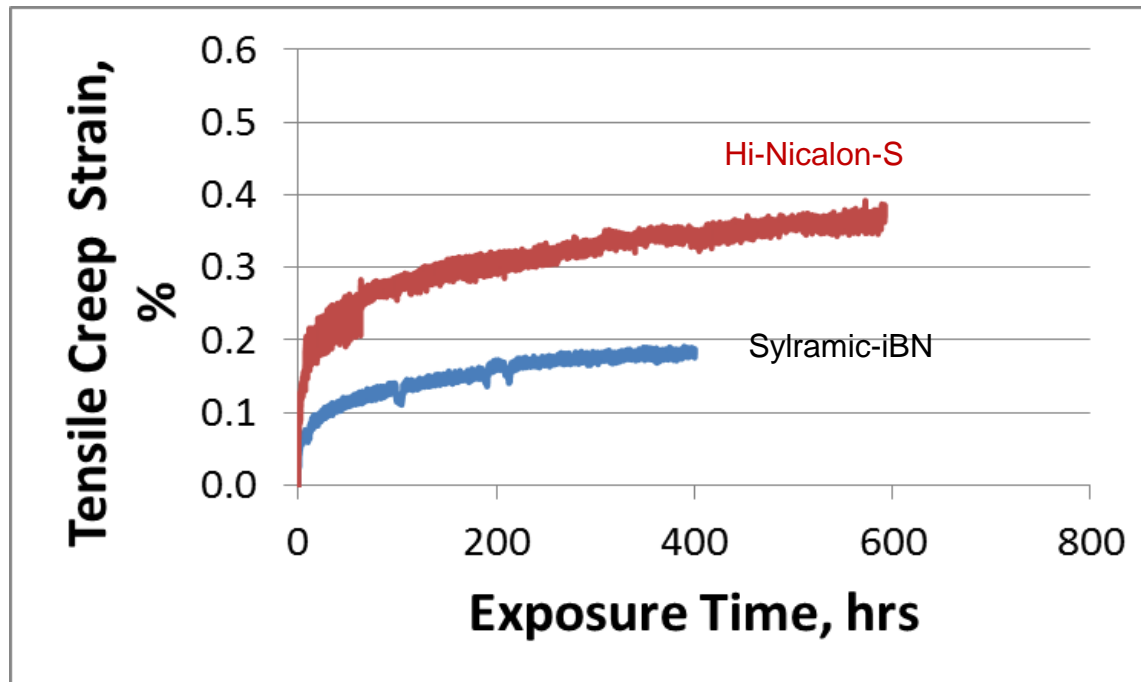
# CT Images of a 3D Woven SiC/SiC Composite Specimen Creep Tested at 1482°C/69MPa/300hrs (Hi-Nicalon-S Fibers)



***The transverse cracks formed during creep deformation***

# Comparison of Tensile Creep Behaviors of 2D Balanced Full CVI SiC/SiC Composites With Hi-Nicalon-S and Sylramic-iBN Fibers Tested at 1450°C/69MPa/Air

( $V_{fx} \sim 0.17$ ,  $V_{fy} \sim 0.17$ ,  $V_{CVISiC} \sim 0.45$ )

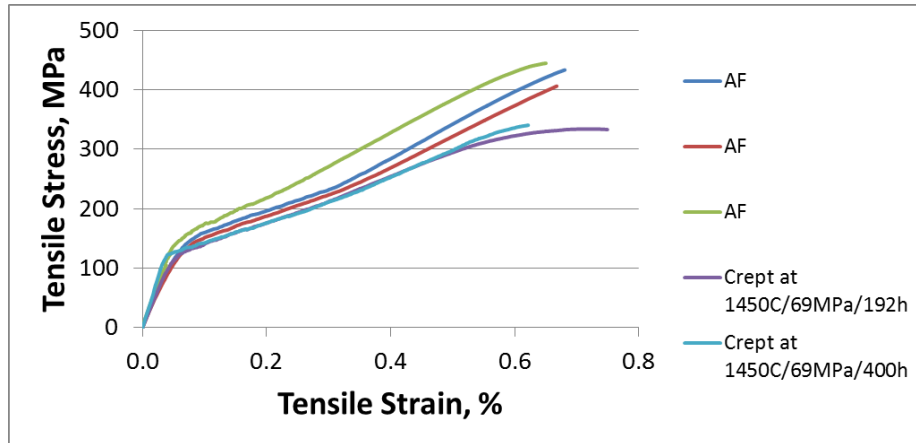


**At a fixed stress level, 2D Hi-Nicalon-S CVI SiC/SiC composites show higher primary creep strain than 2D Sylramic-iBN CVI SiC/SiC composites. In both composites, the long creep durability is controlled by the CVI SiC matrix**

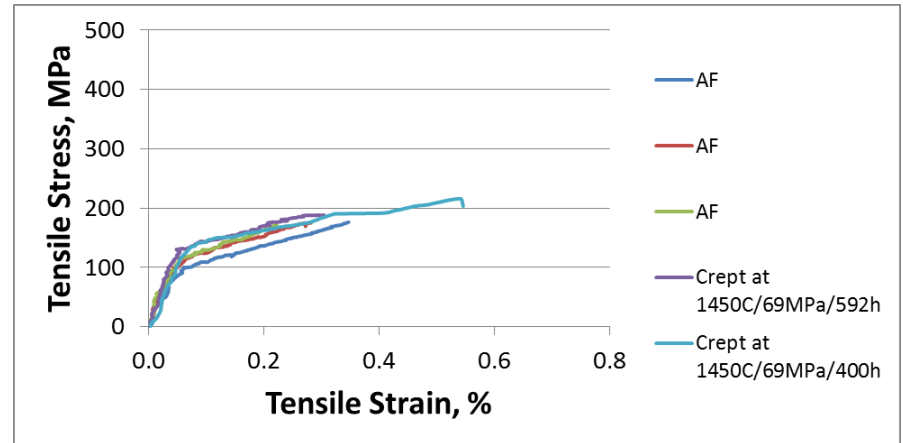
# Tensile Stress Strain Behaviors of As-Fabricated and Crept 2D Full CVI SiC/SiC Composites with Hi-Nicalon-S Fibers

( $V_{fx} \sim 0.17$ ,  $V_{fy} \sim 0.17$ ,  $V_{CVISiC} \sim 0.45$ )

Room temperature



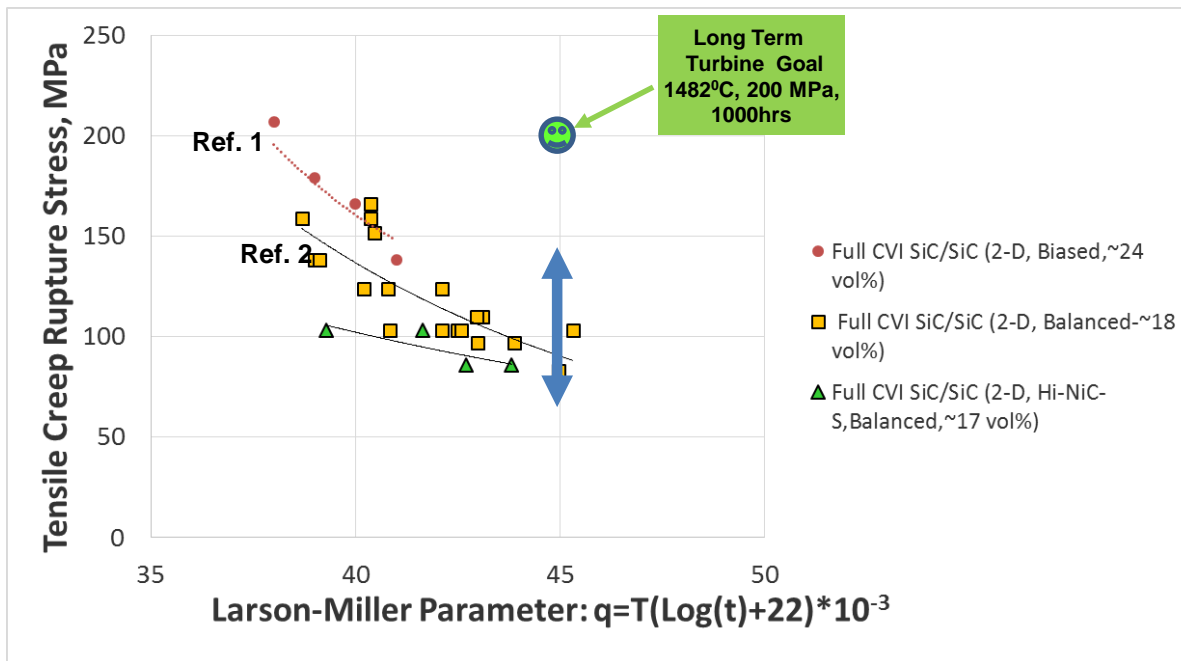
1482<sup>o</sup>C



**AF: As-Fabricated**

**Note: The 3D woven SiC/SiC composites with Hi-Nicalon-S fibers after 400-600 hr creep at 1482<sup>o</sup>C/69MPa retain significant % of as-fabricated room temperature and 1482<sup>o</sup>C in-plane properties**

# Comparison of On-Axis SiC/SiC Rupture Data of Balanced and Biased Full CVI SiC/SiC, and NASA 3D SiC/SiC with (CVI+PIP) Hybrid Matrix



*The CMC matrix in 2-D woven balanced or biased full CVI SiC/SiC composites with Sylramic-iBN fibers cracked during initial creep loading or during creep and exhibit fiber dominated creep.*

1. “Development of the Next Generation of Ceramic Matrix Composites for Turbine Engines – 2700°F SiC/SiC Composites” A. Chamberlain and R. Shinavski, 2012 and 2013 Cocoa Beach Conference Proceedings.
2. “SiC Matrix Composites for High Temperature Hypersonic Vehicle Structural Thermal Protection Systems” R. Shinavski, W. Thibault, and S. Harris, NASA SBIR Phase II Final Report , 2014.

**Blue arrow:** NASA 3D woven SiC/SiC composites with (CVI+PIP) hybrid matrix and with Sylramic-iBN/super Sylramic-iBN fibers tested at 1482°C for up to 700hrs under isothermal tensile creep, low cycle fatigue, sustained low cycle fatigue(SPLCF) and SPCLF under thermal gradient conditions without failure.



# Summary and Conclusions

- The influence of fiber types and fiber vol% on creep/stress rupture properties of 3D woven SiC/SiC composites with ( CVI+PIP) hybrid matrix were studied at 2700F at 69, 103 and 138MPa in air. The fiber types investigated were Sylramic-iBN, super Sylramic-iBN and Hi-Nicalon-S fibers. The results indicate the following.
- At high temperatures no close creep match between commercially available SiC fibers and SiC matrix exists in order that both could carry the on-axis CMC load for long times.  
If creep mismatch ratio is  $>1$ , the matrix will control and if this mismatch ratio is  $<1$ , the fiber will control the long term creep durability of an un-cracked CMC.
- Under the same tested conditions, the creep resistance of composites with super Sylramic-iBN fibers and ~28 vol% CVI SiC is similar or slightly better than those with Sylramic-iBN fibers,
- The long term creep/creep rupture properties of 3D composites with Sylramic-iBN or Super Sylramic-iBN fibers are controlled by the (CVI+PIP) matrix.
- The composites crept for 300hr between 69-138MPa at 2700F retained significant % of their in-plane tensile properties at room as well as at 2700F.

# Summary and Conclusions (continued)

- In 3D SiC/SiC composites with Hi-Nicalon-S fibers containing high vol% of fiber and low vol% of CVI SiC and creep tested at 2700F showed the matrix cracks during initial creep loading or during creep deformation depending on the applied stress, causing fiber dominated creep and short creep rupture life.
- The long term creep durability in these can be achieved composites can be achieved at low stresses if these composites are fabricated with fiber vol% <36 and CVI SiC vol% between 25-30 similar to 2D woven SiC/SiC composites.
- In 2D or 3D woven composites containing partial or full CVI SiC and exhibiting creep mismatch ratio of >1, increasing fiber vol% may not necessarily improve their long term creep durability, as in the case of 3D SiC/SiC composites with Hi-Nicalon-S fibers. Other factors such as vol% of CVI SiC, interfacial shear strength between the fiber and the matrix will also affect creep durability.