



Creep and Cyclic Fatigue Durability of 3D Woven SiC/SiC Composites with (CVI+PIP) Hybrid Matrix

**R.T. Bhatt
Ohio Aerospace Institute
NASA Glenn Research
Center
Cleveland, OH**

Support from NASA Fundamental Aeronautics Program

**Prepared for Presentation at the ECI Conference, Santa Fe, New Mexico,
November, 2017**



Objectives and Approach

Objective:

- Determine influence of fiber types on creep behavior/creep rupture life of 3D woven SiC/SiC composites with (CVI+PIP) hybrid matrix.
- Determine creep parameters of the composites to model creep behavior and creep rupture life.
- Evaluate dwell and low cycle fatigue resistance of the composites.

Experimental Approach:

- Focus on 3D-woven SiC/SiC CMC with (CVI+PIP) hybrid matrix and Sylramic-iBN/Super Sylramic-iBN fibers.
- Measure on-axis tensile creep behavior of CMC at stresses below the matrix cracking stress and at 1315⁰C and 1482⁰C for up to 1000hrs in air.
- Measure residual on-axis tensile properties of composites after 300hr creep tests
- Conduct sustained peel low cycle fatigue (SPLCF) and low cycle fatigue(LCF) tests at 1482⁰C at 138MPa in air.

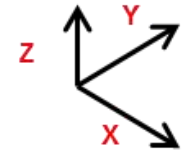
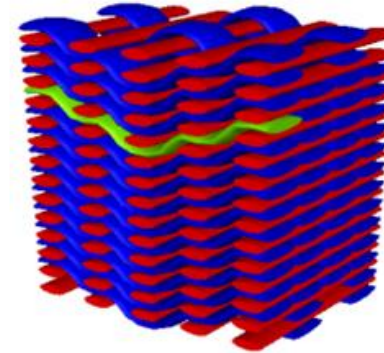
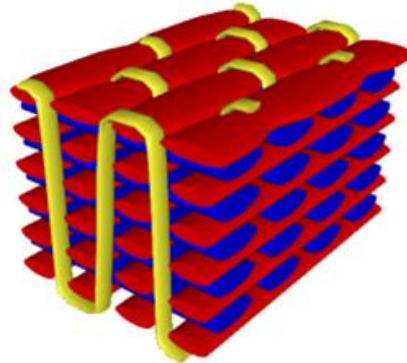
CMC Material

Fiber Architecture:

Orthogonal and Angle interlock

Orthogonal

Angle Interlock



Composite Characteristics:

Fibers: Sylramic™-iBN and Super Sylramic™-iBN SiC fibers

Interface: Chemical Vapor Infiltrated CVI BN

Matrix: (CVI SiC+ PIP SiC)

Composite Vendors:

Fiber weaving: TEAM, Inc

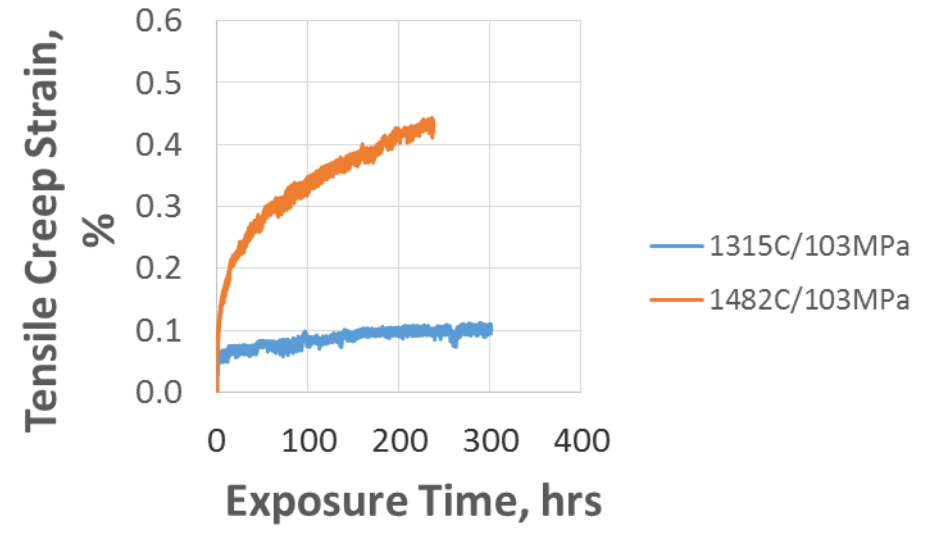
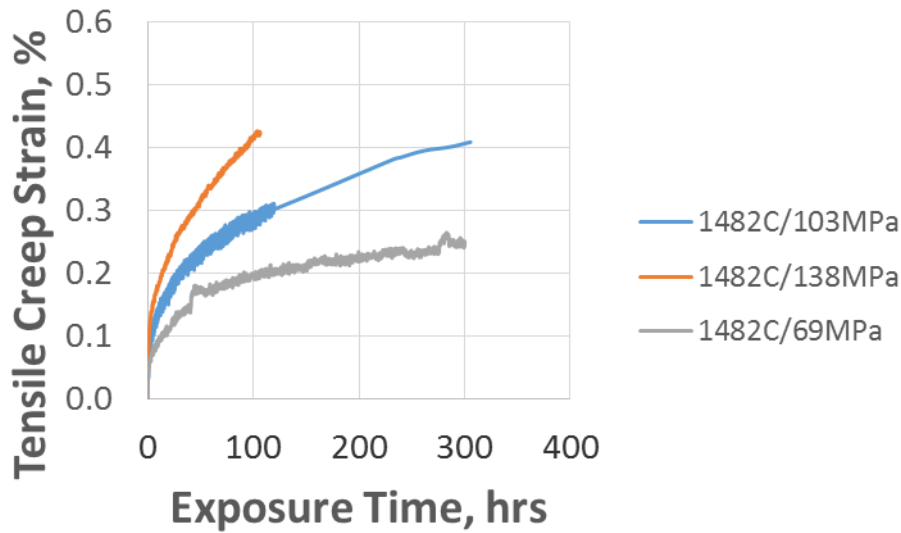
Composite preform with CVI BN and SiC coating: Rolls Royce HTC Inc,
(Long Beach CA)

PIP processing: Teledyne Scientific, Thousand Oaks CA

Note: Sylramic™ fibers fabricated by Dow Corning(currently by COIC-ATK).
The i-BN and Super-iBN treatment conducted at NASA

Influence of Stress on Creep Behavior of 3D Woven SiC/SiC Composites at 1315°C and 1482°C in air

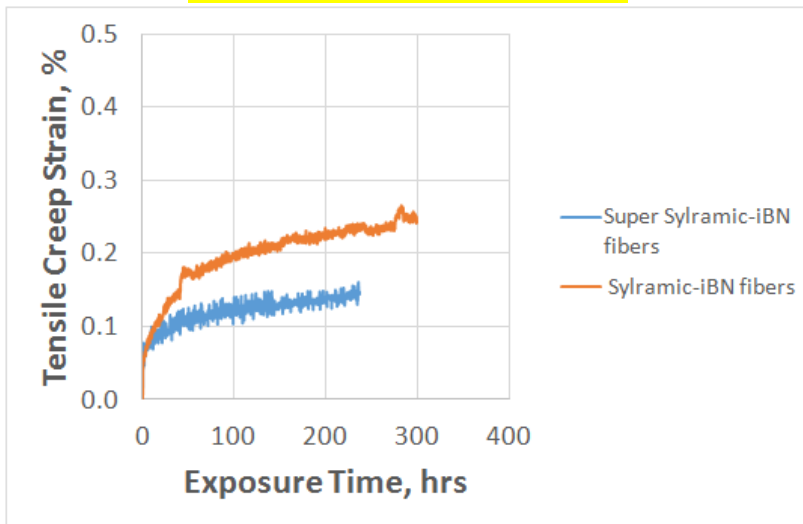
Fiber Architecture: Orthogonal-2 floater ($V_{fx} \sim 0.07$, $V_{fy} \sim 0.22$, $V_{fz} \sim 0.02$, $V_{CVSiC} \sim 0.28$)



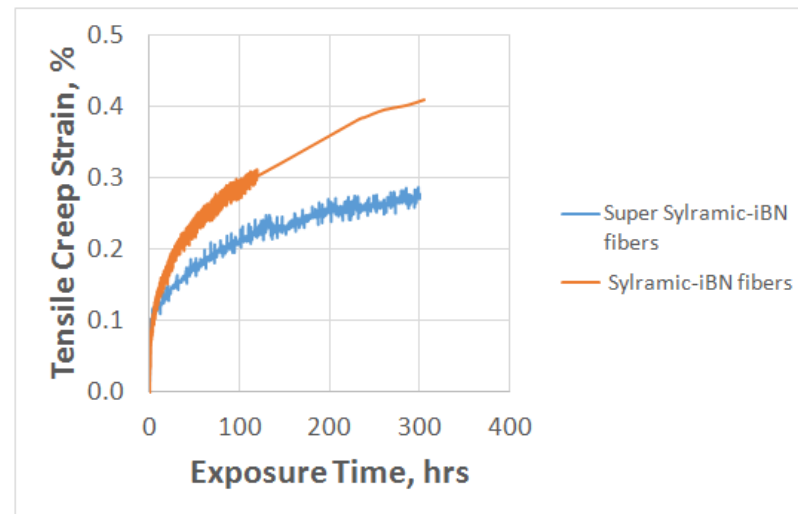
Influence of Fiber Types on Tensile Creep Behavior of 3D Woven SiC/SiC Composites with (CVI+PIP) Hybrid Matrix

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

1482°C/69MPa/Air



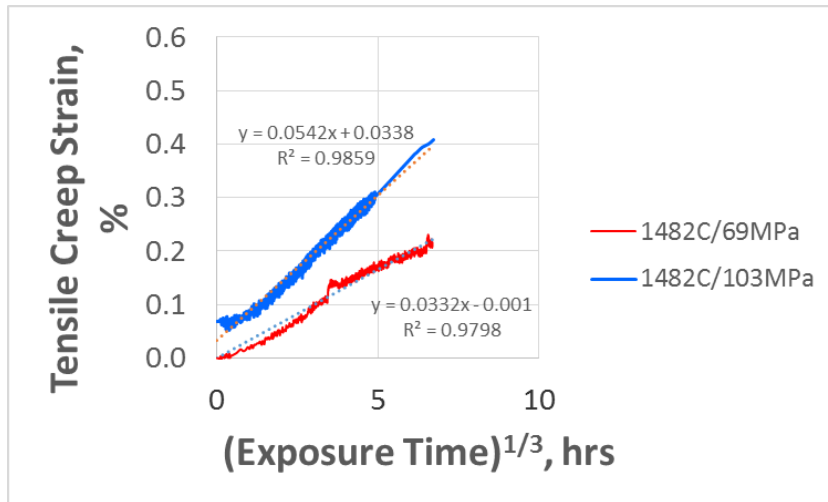
1482°C/103MPa/Air



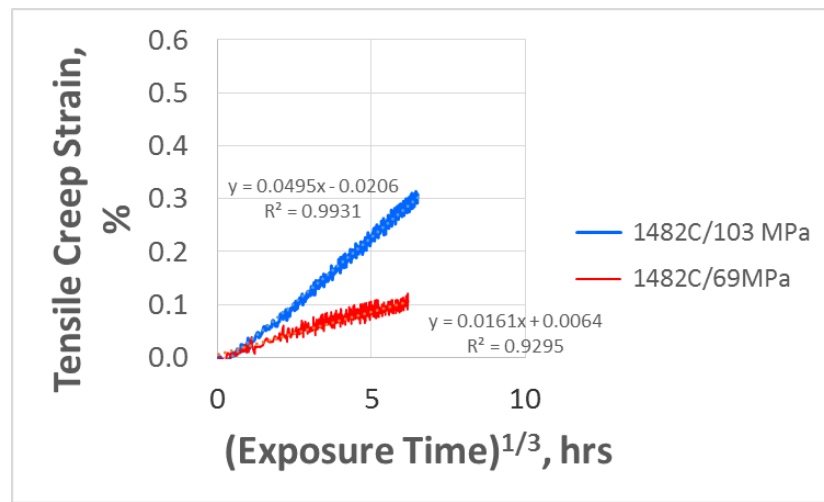
Note: 3D woven (Orthogonal) SiC/SiC composites with Super Sylramic-iBN fibers show slightly better creep resistance than those containing Sylramic-iBN fibers

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

Sylramic-iBN fibers

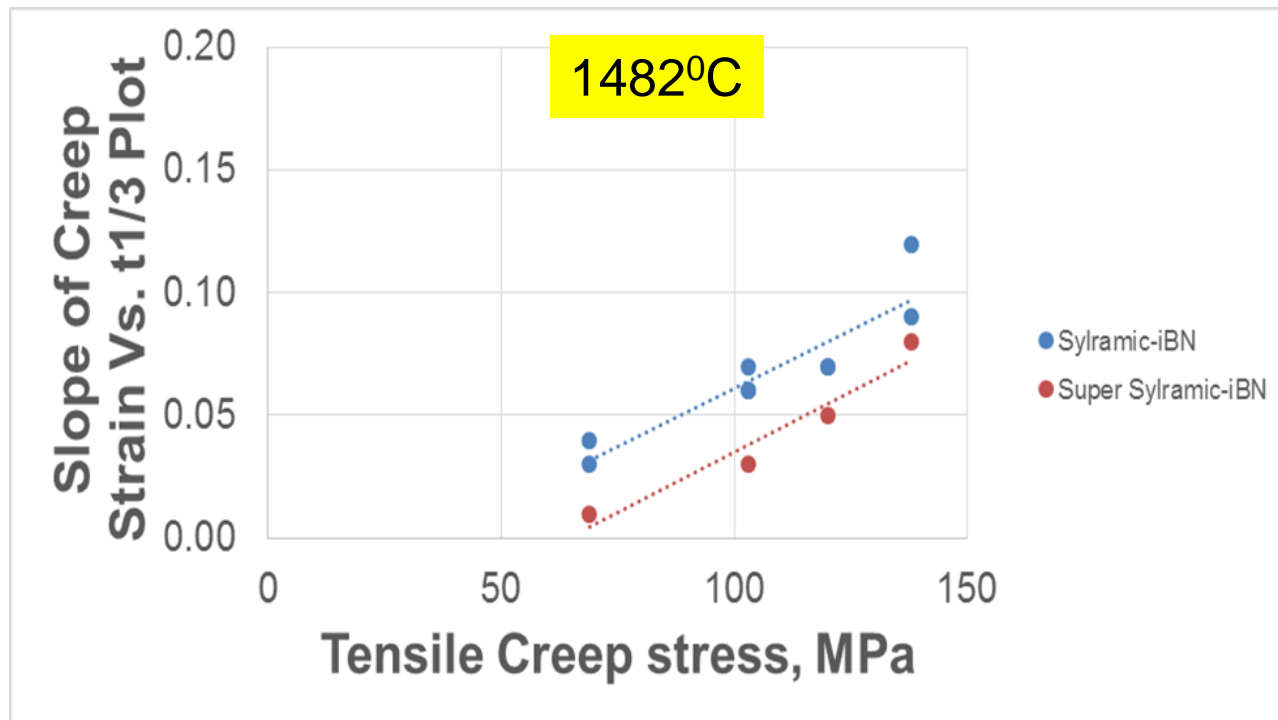


Super Sylramic-iBN fibers

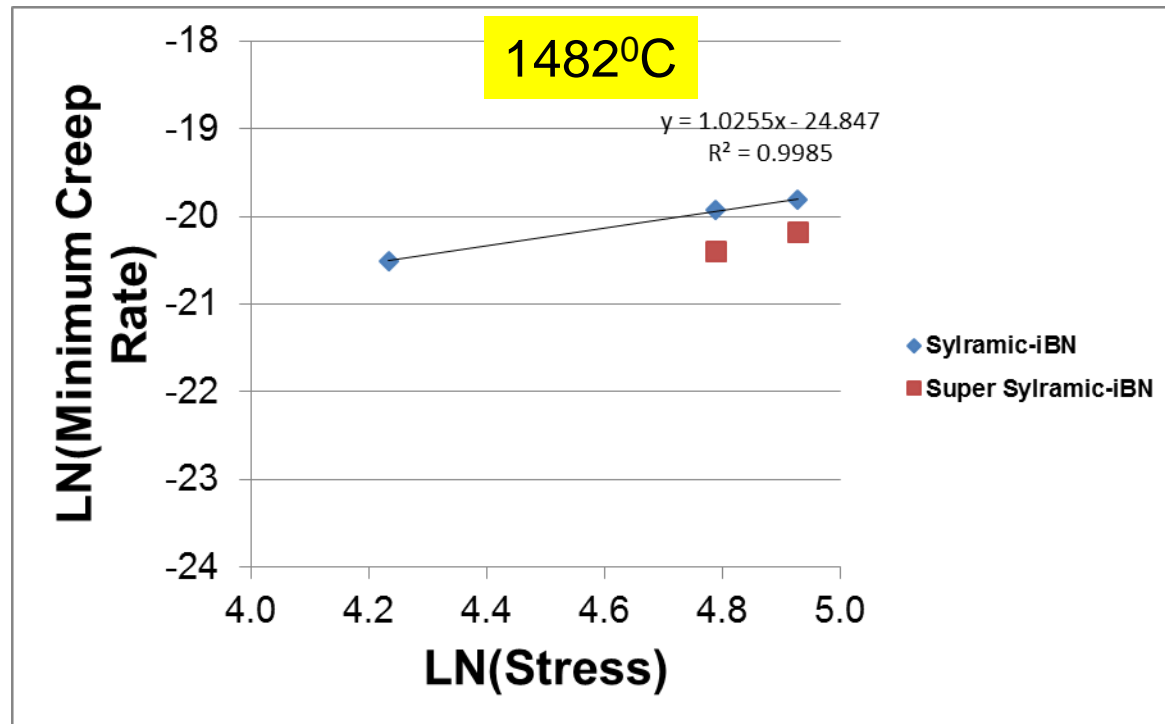


- At 1482^oC, the accumulated creep strain in 3D woven SiC/SiC composites with (CVI+PIP) hybrid matrix and with Sylramic-iBN or Super Sylramic-iBN fibers linearly varies with $t^{1/3}$ and shows linear stress dependency. A behavior similar to CVD-derived ultra SCS monofilament SiC fiber and CVD SiC matrix.

Variation of Slope of Creep Strain vs. $t^{1/3}$ plot with Creep Stress For 3D Woven SiC/SiC Composites With (CVI+PIP) Hybrid Matrix



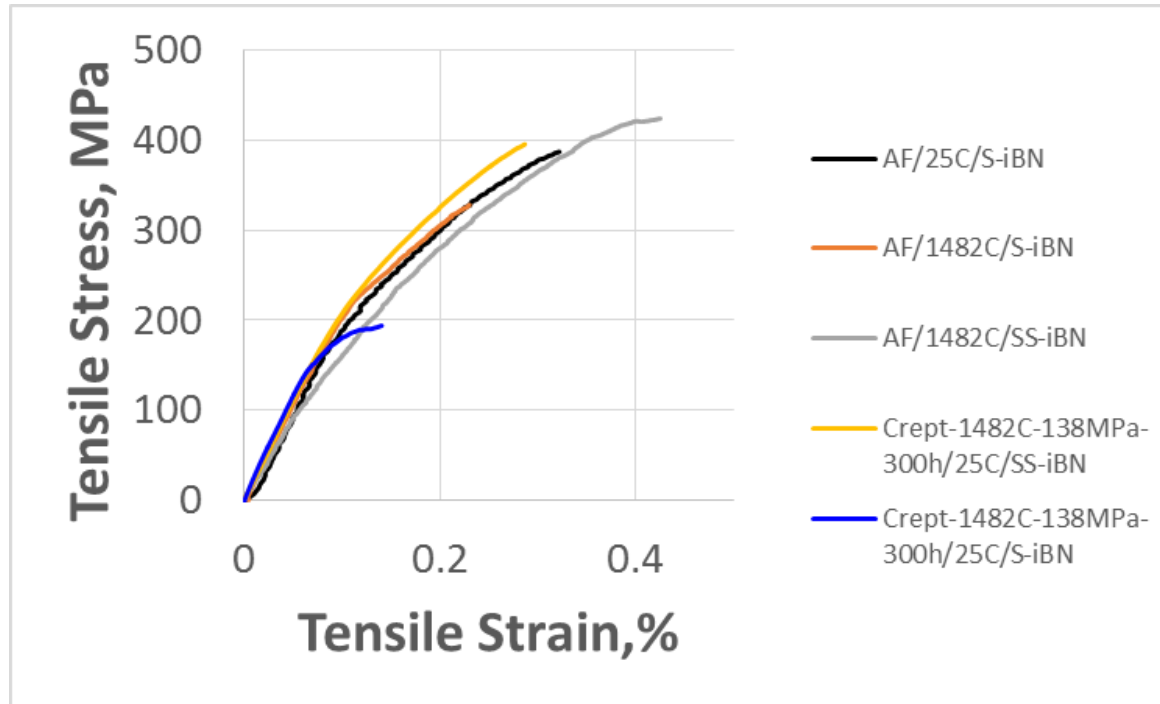
Variation of Minimum Creep Rate with Creep Stress For 3D Woven SiC/SiC Composites With (CVI+PIP) Hybrid Matrix



Note: 3D woven SiC/SiC composites with (CVI+PIP) matrix and Sylramic-iBN fibers or Super Sylramic-iBN fibers show stress exponent of ~1

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

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

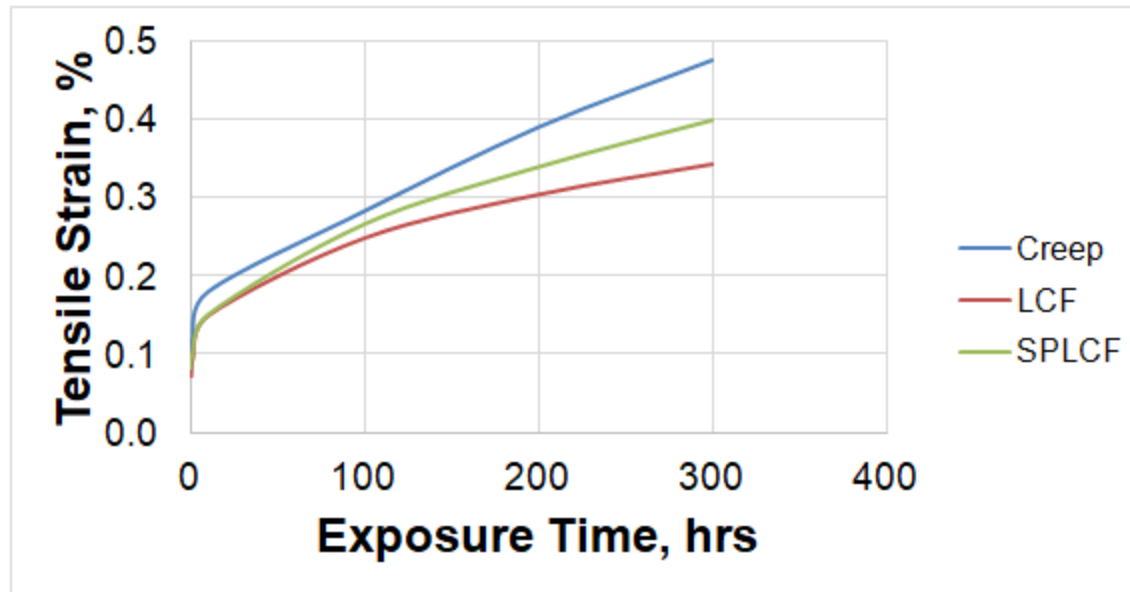


AF: As-Fabricated, S-iBN: Sylramic™-iBN, SS-iBN: Super Sylramic™-iBN

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

Comparison of Creep, LCF and SPLCF Behaviors of 3D Woven SiC/SiC Composites With (CVI+PIP) Hybrid Matrix Tested at 138 MPa at 1482°C in Air

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



Note: Accumulated strain with time in 3D woven SiC/SiC composites depends on hold time at the maximum stress.

Creep Data Analysis

Conventional Creep Equations used to Analytically Model Tensile Creep of CMC

Total Strain (%): $\varepsilon = \varepsilon_e + \varepsilon_p + \varepsilon_s$

Elastic Strain: $\varepsilon_e = \sigma/E$

Primary Stage Strain: $\varepsilon_p = A \varepsilon_e [1 - \exp(-t/\tau)^k]$

Secondary Stage Strain: $\varepsilon_s = (C/T) (\sigma^n / d^m) \Theta$ (power law creep)

Where E = temperature-dependent elastic modulus

σ = Composite tensile stress; n = stress exponent

τ = primary stage time constant

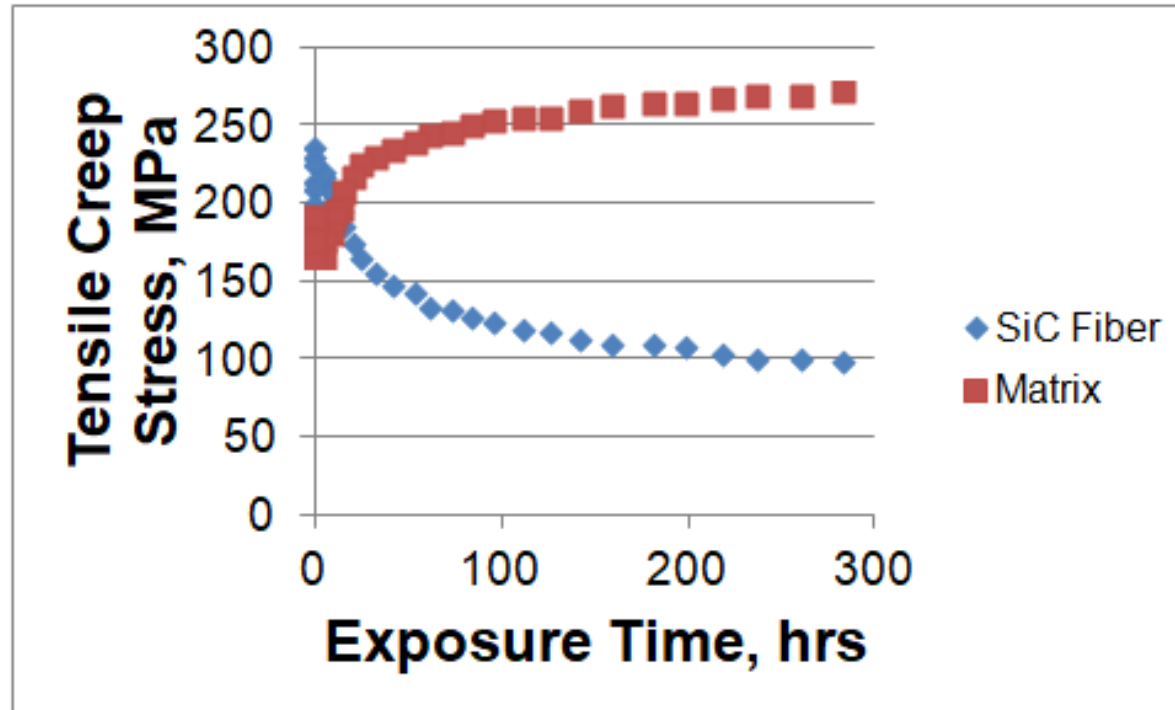
$\Theta = t \exp(-D/T)$; t = time, T = Kelvin

D = Q/R ; Q = controlling energy, R = Boltzmann constant

d = average grain size (nm); m = grain size exponent

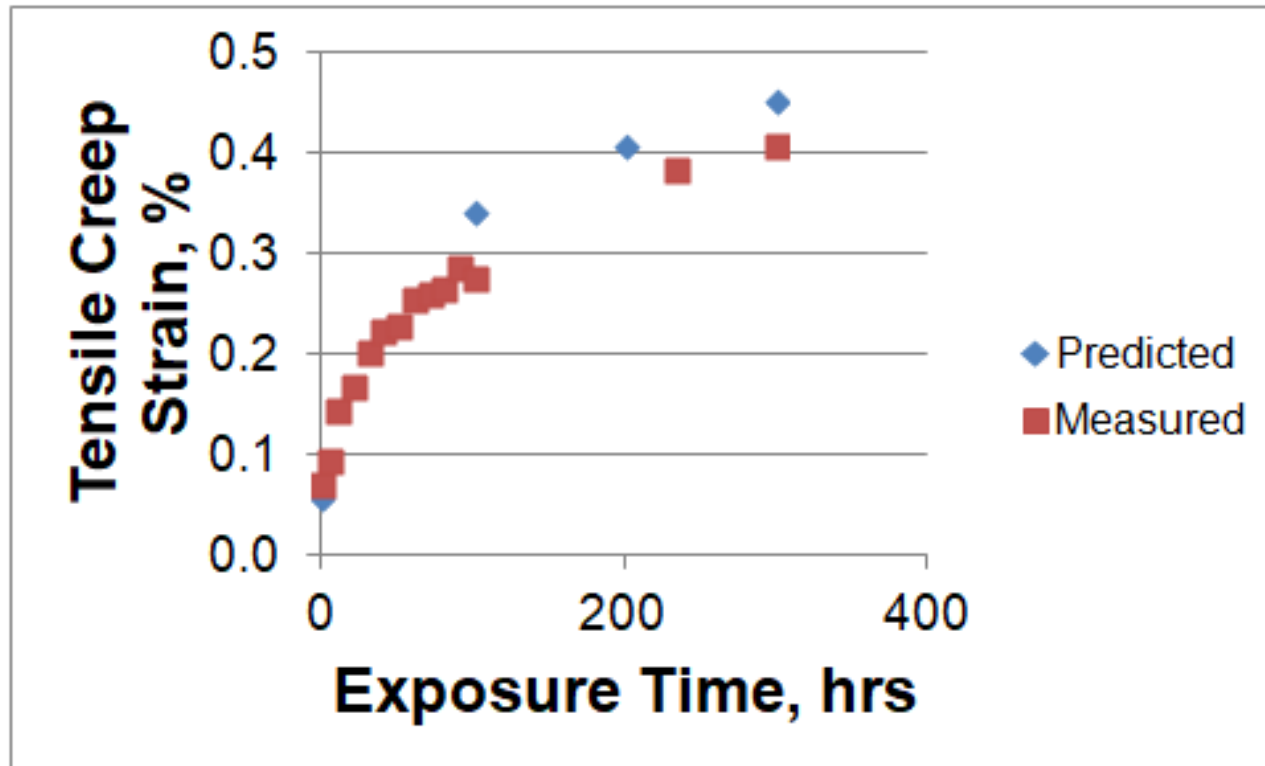
τ, k, n, m, A, C, D = empirically determined best-fit parameters from creep data

Predicted Variation of Fiber and Matrix Stresses with Time During On-axis CMC Creep: NASA 3D Hybrid CMC at 1482°C and 103 MPa

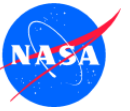


Note: Since creep resistance of CVI SiC matrix is greater than that of polymer derived SiC fibers, stress relaxation occurs in the fibers during creep test. As a consequence, the stress on CVI SiC matrix continuously increases and the stress on fibers continuously decreases with increasing exposure time. This also suggests that long term creep durability of the composite at high temperatures is controlled predominantly by the CVI matrix and current commercially available polymer derived SiC fibers do not have adequate creep resistance to achieve 500 to 1000hr durability at high temperatures.

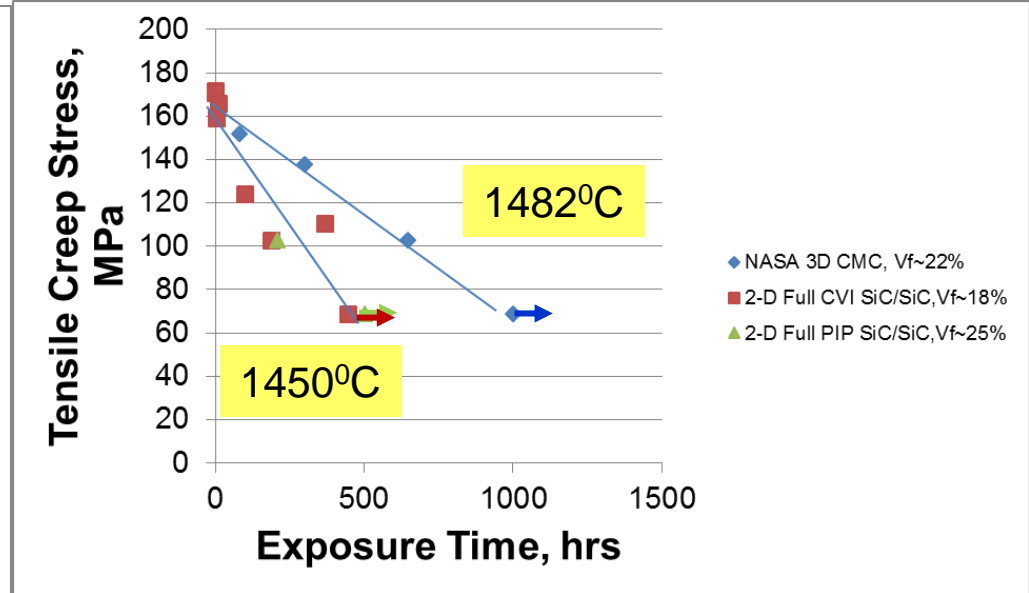
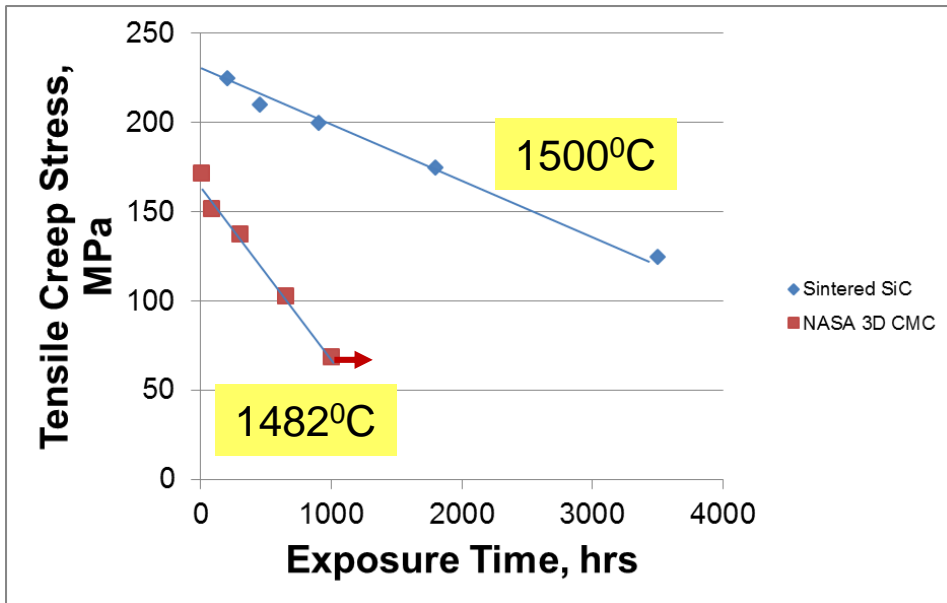
Comparison of Predicted and Measured Tensile Creep Strain with Time During On-axis CMC Creep: NASA 3D Hybrid CMC at 1482°C and 103 MPa



The creep strain can be represented by the equation: $\epsilon_p (\%) = (C)[\sigma_m] [t \times \exp (-D/T)]^{1/3}$ where σ_m is the stress in CMC, T is temperature, D 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.

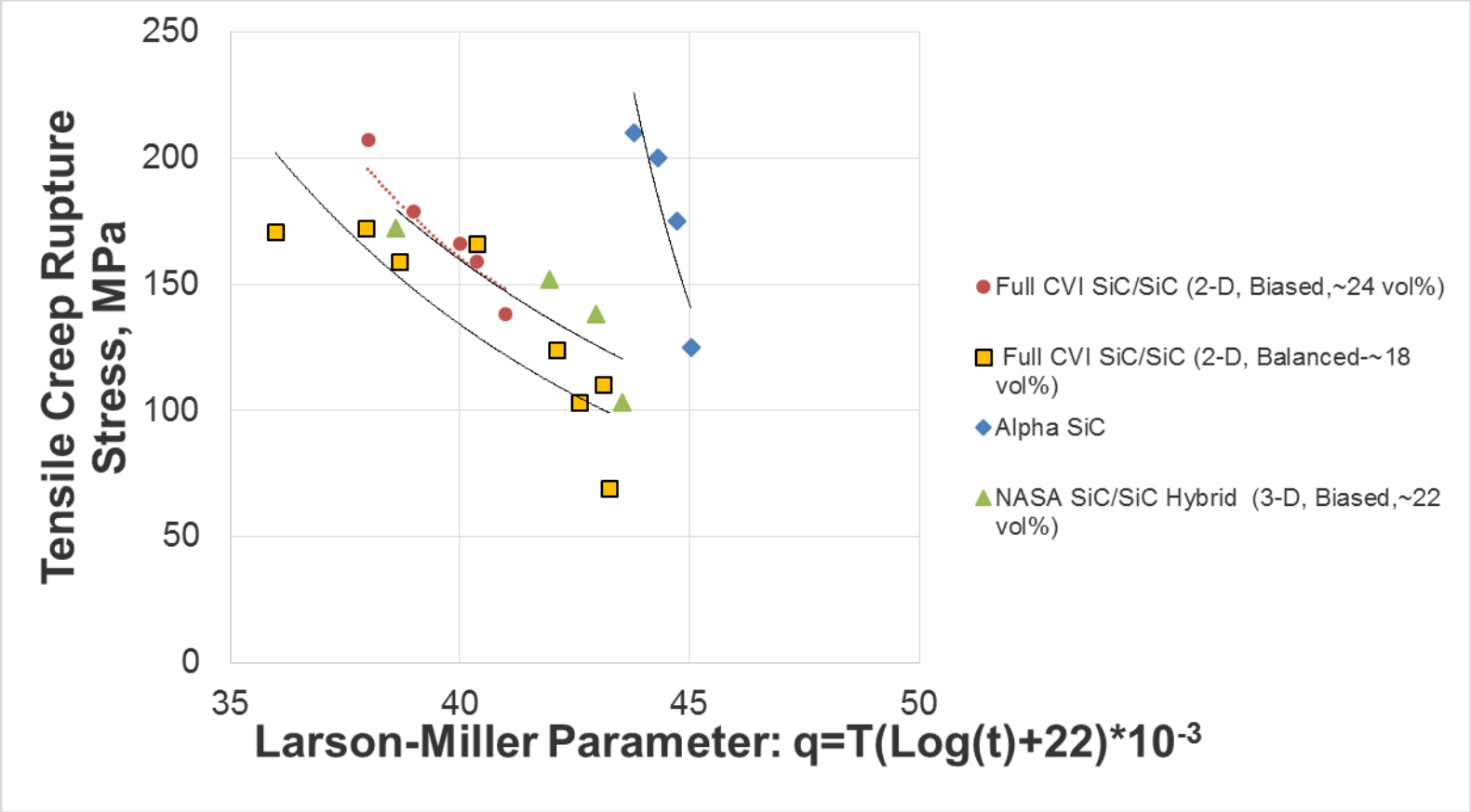


Comparison of Creep Durability for Sintered Alpha SiC, NASA 3D SiC/SiC with (CVI+PIP) Hybrid Matrix, 2D Full CVI and Full PIP SiC/SiC Composites



Note: NASA 3D CMCs with Sylramic-iBN fibers exhibit 500 to 1000 hrs creep durability between 138 and 69 MPa at 1482°C. Further improvement in creep durability of composites in the same stress range can be achieved by using Super Sylramic-iBN fibers, optimizing CVI content and reducing composite porosity. However, achieving 500-1000hr durability at creep stress 150 MPa and above at 1482°C is difficult. The 2D full CVI and PIP SiC/SiC composites do not show 500hr creep durability at stresses > 69MPa.

Comparison of Larson-Miller Parameter for Sintered Alpha SiC, NASA 3D SiC/SiC with (CVI+PIP) Hybrid Matrix, 2D Full CVI and Full PIP SiC/SiC Composites



Summary and Conclusions



- **NASA 3D CMCs with Sylramic-iBN fibers exhibit 500 to 1000 hrs creep durability between 138 and 69 MPa at 1482°C. Further improvement in creep durability of composites can be achieved by using Super Sylramic-iBN fibers, optimizing CVI content and reducing composite porosity. However, achieving 500-1000hr durability at creep stress 150 MPa and above at 1482°C is difficult.**
- **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. Using these creep parameters, the creep behavior of the composites can be predicted. However, creep rupture life is difficult to predict and is influenced by other factors such as slow crack growth resistance of CVI matrix, stress redistribution around cracked fiber tows and local stresses**
- **NASA 3D CMCs also exhibit good dwell and LCF resistance.**
- **For 1482°C capable SiC/SiC CMCs discussed in this study, it was determined that at high temperatures no close creep match between the fiber and matrix existed in order that both could carry the on-axis CMC load for long times. The current study also support the need for development of small-diameter CVI-derived SiC fibers to match the creep behavior of CVI-derived matrices, and thus obtain SiC/SiC CMC with highest creep resistance.**