National Aeronautics and Space Administration

SiC FIBERS AND SiC_f /SiC CERAMIC MATRIX MINICOMPOSITES DAMAGE BEHAVIOR

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Advanced Ceramic Matrix Composites: Science and Technology of Materials, Design, Applications, Performance and Integration

Nov. 7th 2017

Santa Fe, NM

Research Supported by: NASA Transformational Tools and Technologies Project (TTT)

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- MINICOMPOSITES TESTING & CHARACTERIZATION
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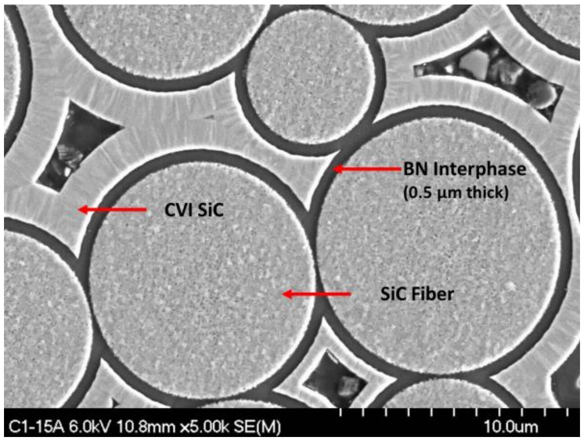
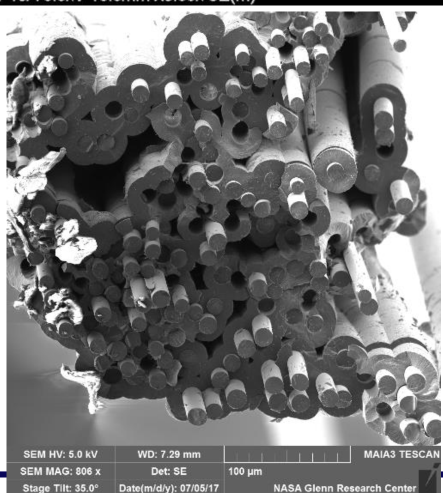
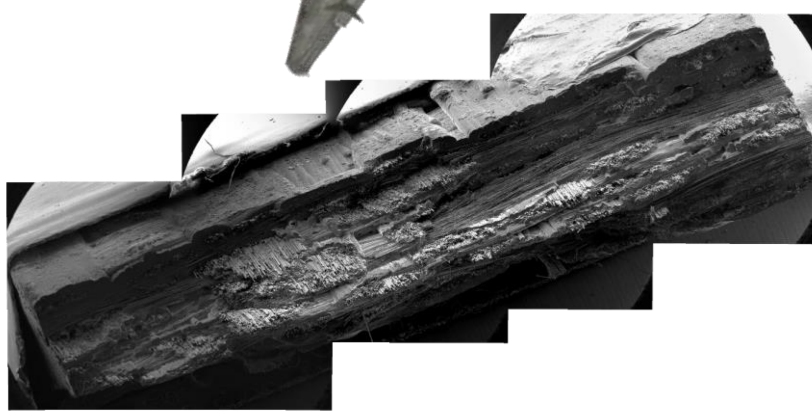
Introduction

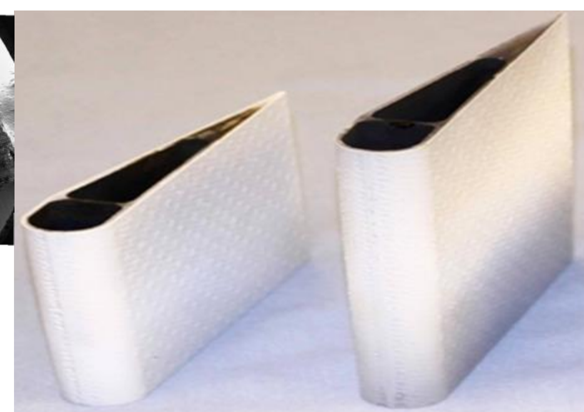
SiC_f /SiC Ceramic Matrix Composites (CMCs) are candidates for high-temperature applications such as the new generations of aircraft engines and hypersonic vehicles due to:

- Improved engine efficiency by using high-temperature capable CMC components to increase engine operating temperatures.
- Reduced component weight (1/3 density of superalloys) and fuel consumption.
- Reduced cooling requirements & emissions (NO_x and CO_2).









Objectives

To Understand:

- Environment's effect on the thermo-mechanical behavior of SiC_f /SiC ceramic matrix composites' constituents.
- Stress dependent matrix crack initiation and propagation.
- Creep behavior of the different constituents of the composite.
- Creep behavior of different SiC_f /SiC minicomposites.
- Characterize the behavior of hybrid matrices and EBCs.
- Support SiC_f /SiC CMC & SiC fiber environmental durability.

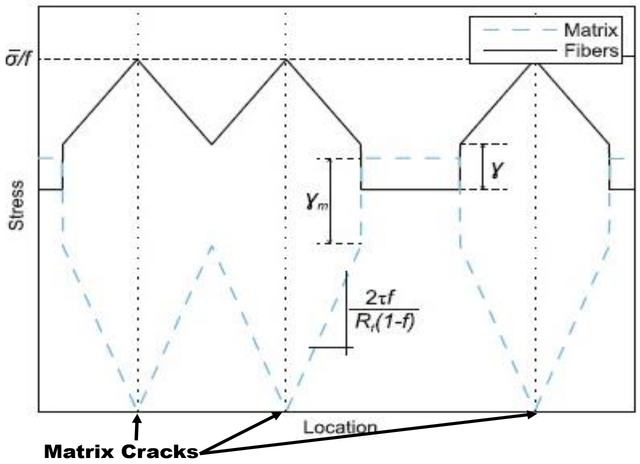
SiC_f /SiC Minicomposites Damage Mechanisms

➢ Case A: CMCs are loaded below the matrix cracking stress. (Static or Dynamic Loading)

- Fibers carry a fraction of the applied load.
- Fibers are not exposed to oxidation damage and the most dominant damage mechanism is creep of fibers.

➢ Case B: CMCs are loaded above the matrix cracking stress.

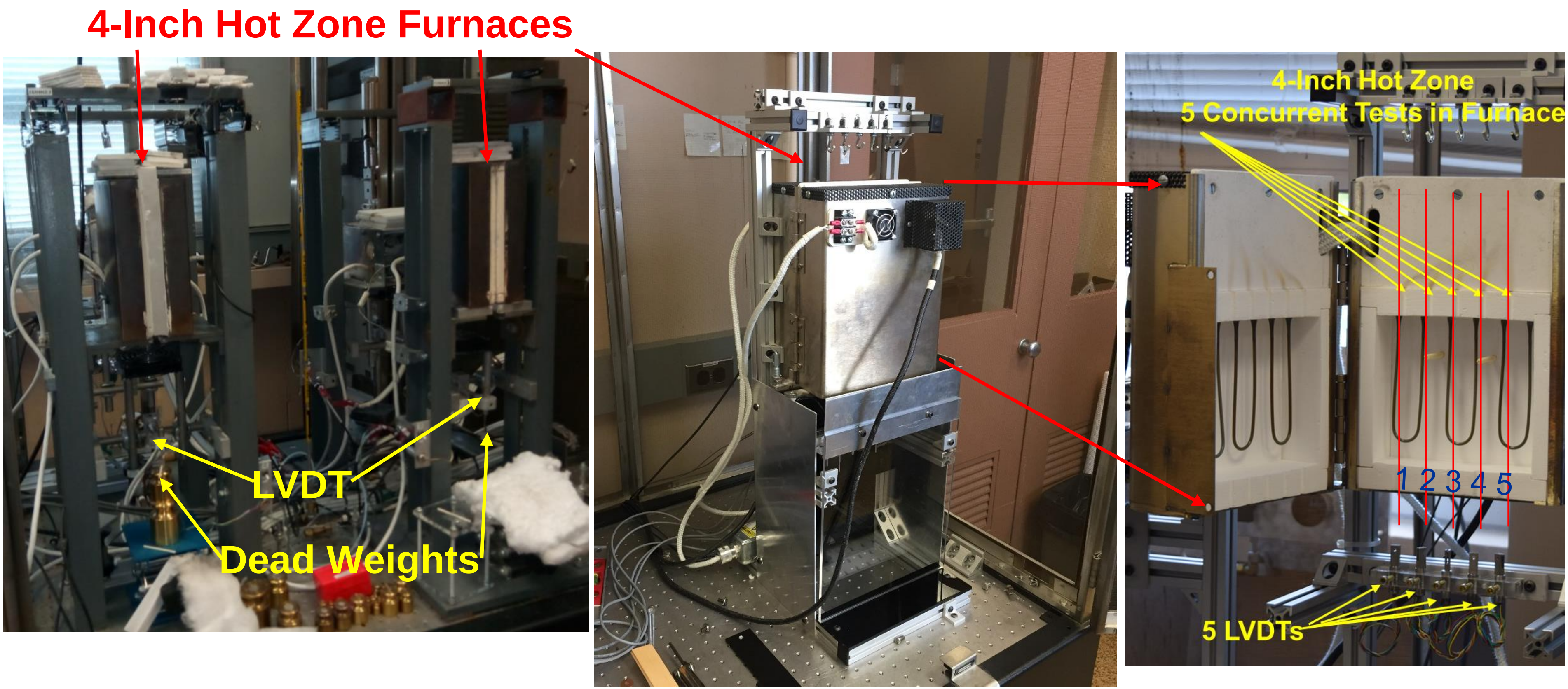
- Fibers carry all of the applied load in the vicinity of through-thickness matrix crack.
- Fibers are exposed to oxidation and creep damage mechanisms.
- Load transfer from oxide layer to the core of the fibers.
- Long lengths of fibers at high stress due to multiple matrix cracks and the reduction of fibers' cross-sectional area due to oxidation.



➢ Hutchinson, J. W., Jensen, H. M., Sep. 1990. Models of fiber debonding and pullout in brittle composites with friction. Mech. Mater. 9 (2), 139–163.

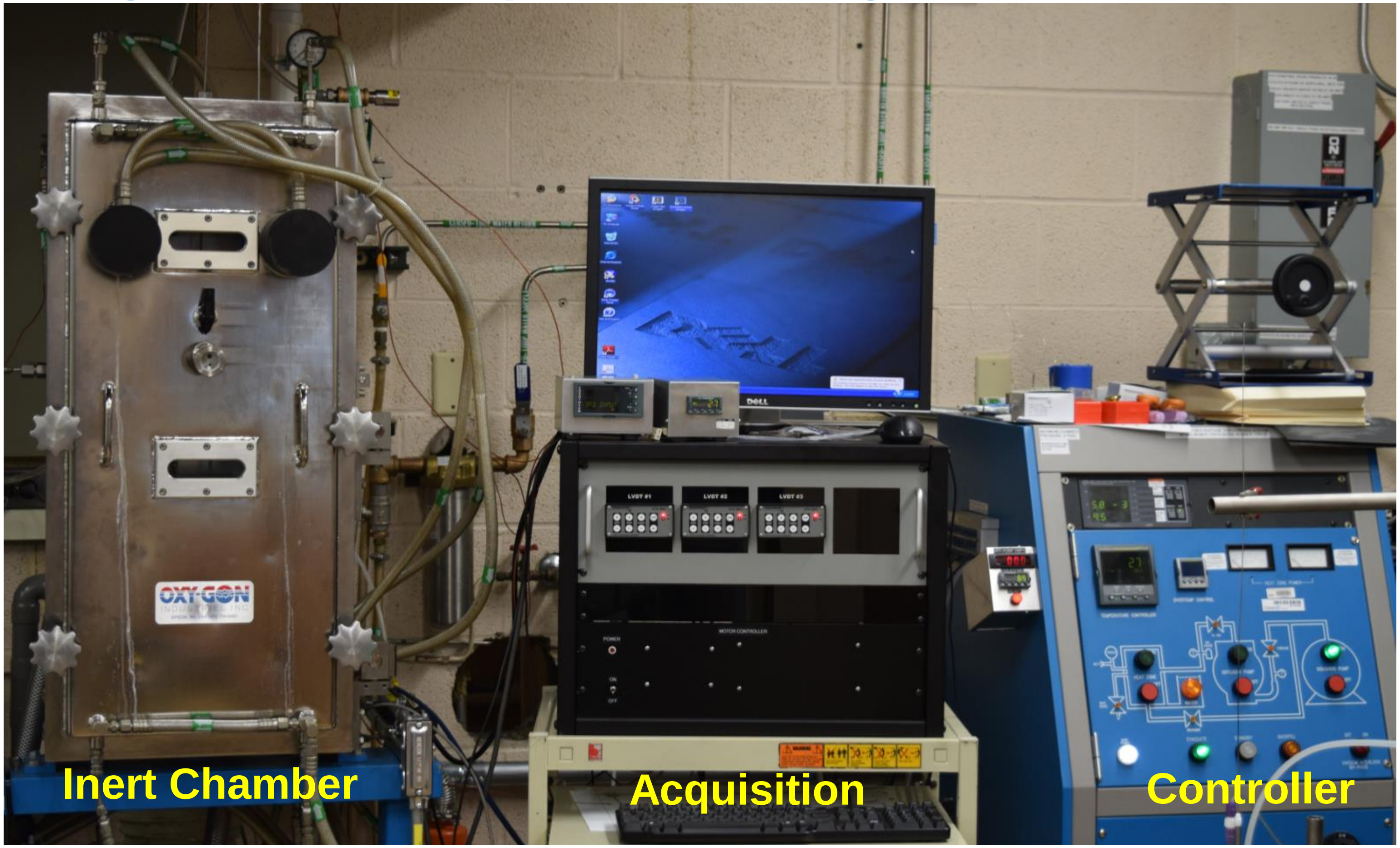
FIBER CREEP TESTING & CHARACTERIZATION

Single Fiber Creep Tests in Air



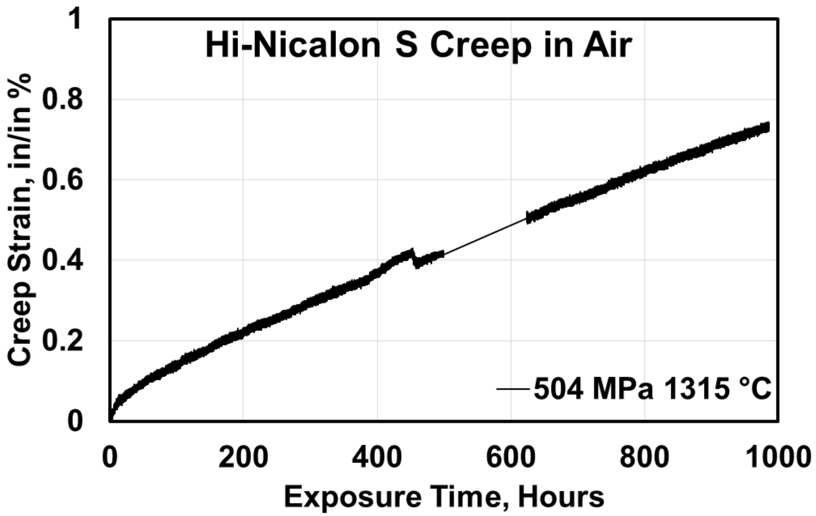
- All tests are conducted on a vibration free table.
- Availability of 1 inch or 4 inch hot zone furnaces.
- "Shields" have been installed beneath the furnace to minimize air flow/currents around fibers.
- In-house software to acquire displacement, temperature and time data.
- Results are used to understand fiber creep, creep rupture and slow crack growth.

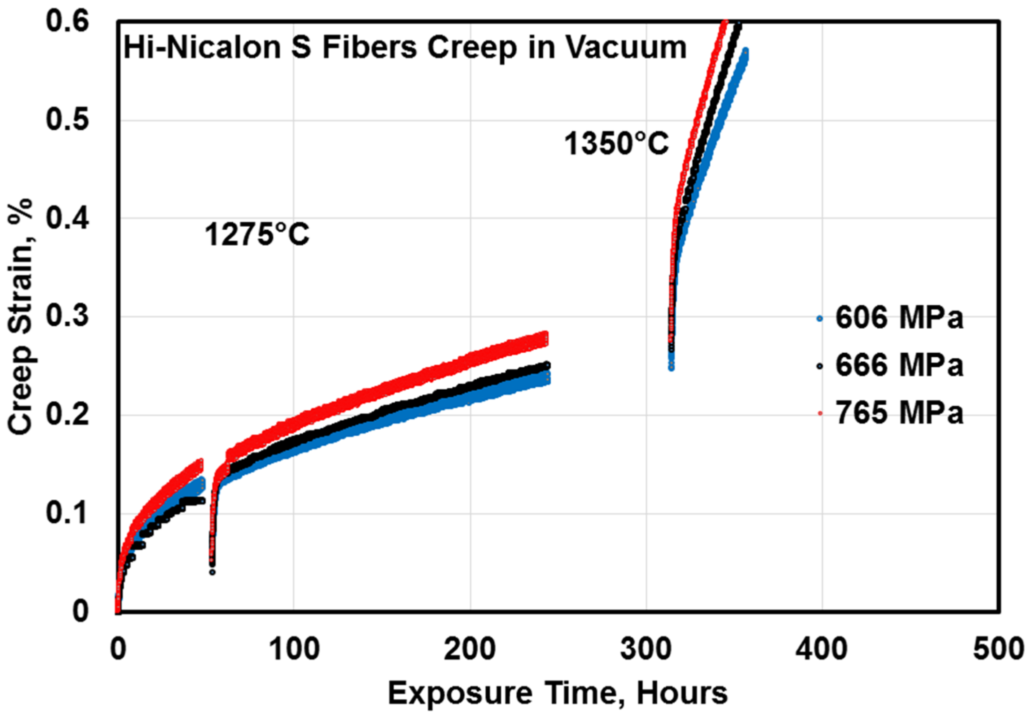
Single Fiber Creep Tests in Argon or Vacuum

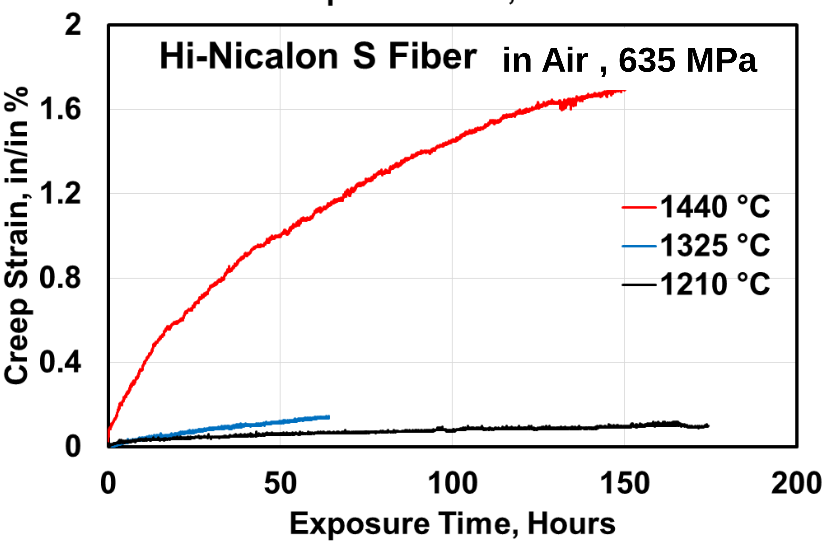


- Can run 3 concurrent single fiber or fiber tow creep tests in Argon or vacuum up to 3000 ˚F.
- 4 inch hot zone furnace with graphite heating elements.
- Potentially, it can run 5 or more concurrent creep tests with minor modifications.
- Vacuum or Argon environments simulate fiber conditions in un-cracked composites.
- Results are used in modelling creep behavior of composite coupons and sub-elements.

Single Fiber Creep Tests Results

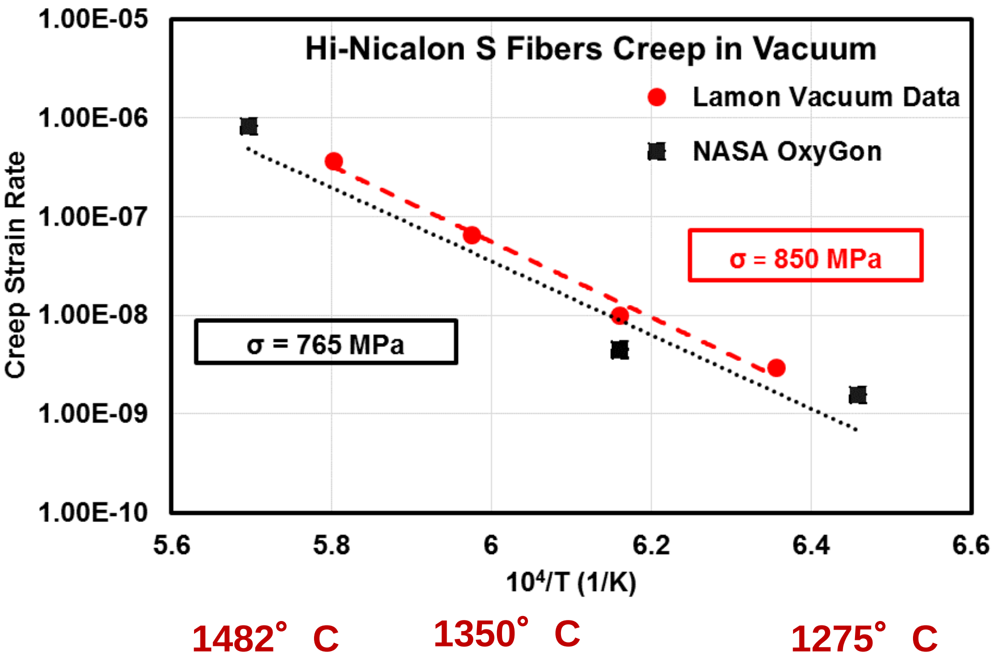


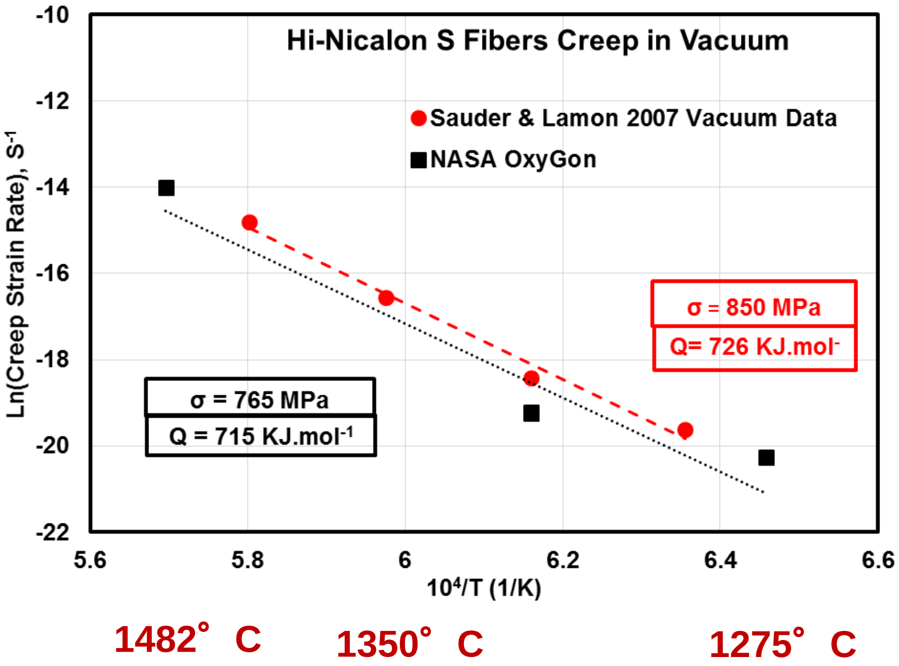




- Creep tests in air were performed to simulate fiber environment in the vicinity of matrix cracks and obtain necessary parameters for modelling.
- Creep tests in vacuum were performed to simulate fiber environment in uncracked composites and obtain parameters for modelling.

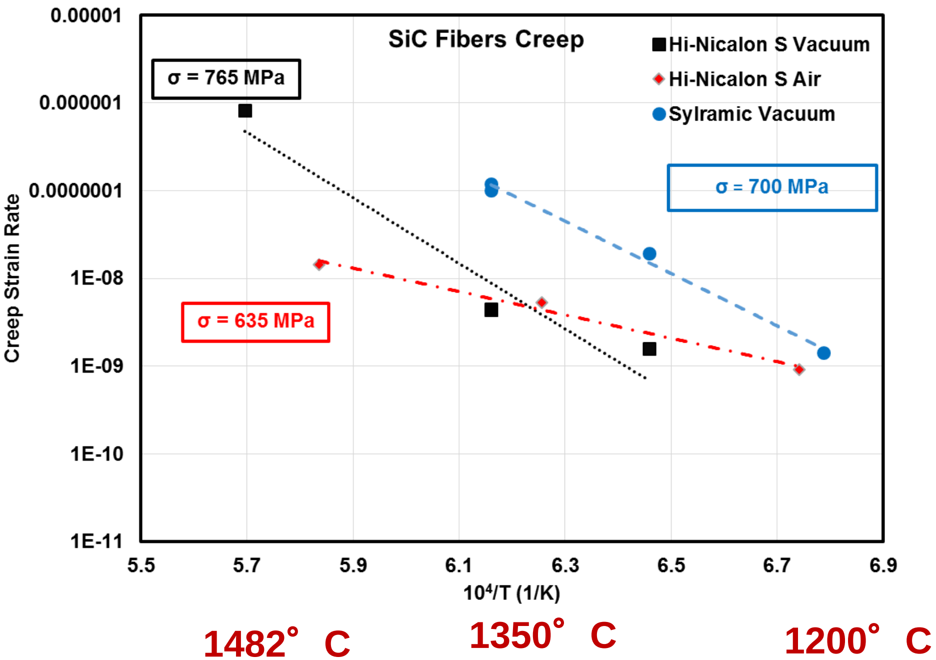
Single Fiber Creep Tests in Vacuum Results

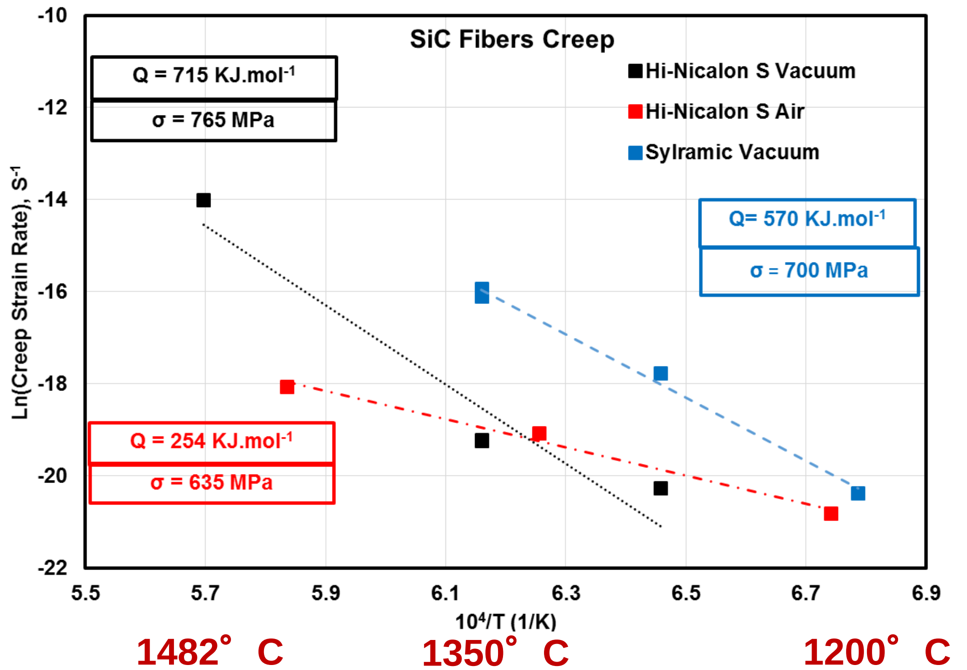




- Reproduced Sauder and Lamon 2007 Hi-Nicalon S single fiber creep tests in vacuum.
- There is good agreement in Hi-Nicalon S fiber creep temperature dependence and activation energy.

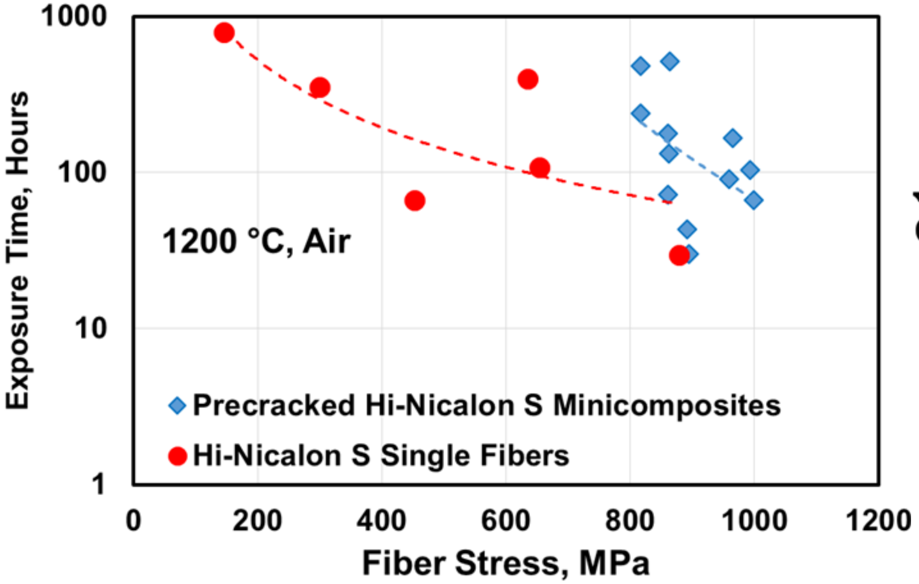
Single SiC Fiber Creep Tests in Air and Vacuum

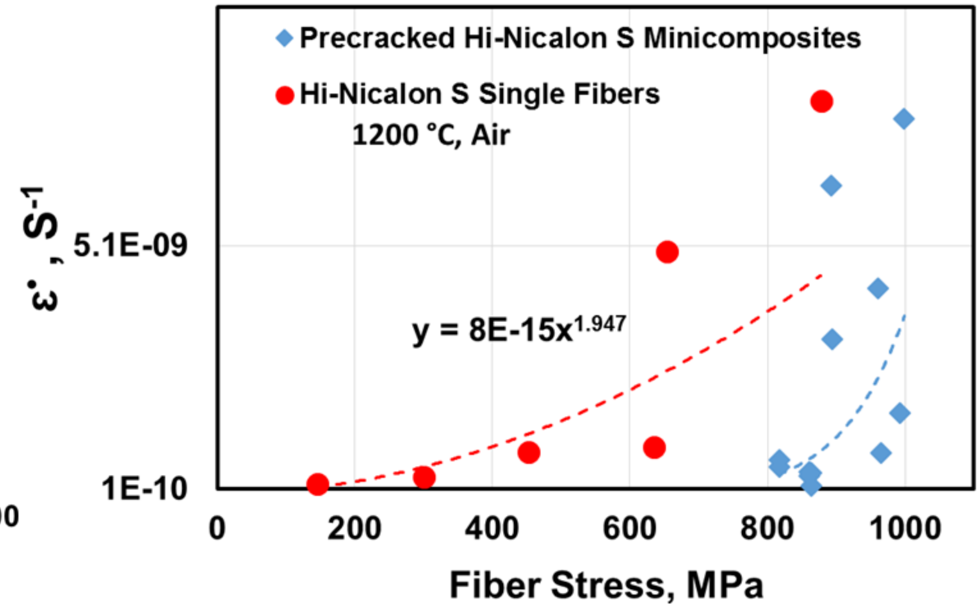




- Single Hi-Nicalon S fiber creep temperature dependence in air was compared to that of Hi-Nicalon S fibers in vacuum. Also, It was compared to creep temperature dependence and activation energy of as-received Sylramic fibers
- Hi-Nicalon S is more creep resistant in vacuum than air.
- Hi-Nicalon S is more creep resistant than as-received Sylramic in vacuum.

Correlating Single Fibers Creep to Minicomposites



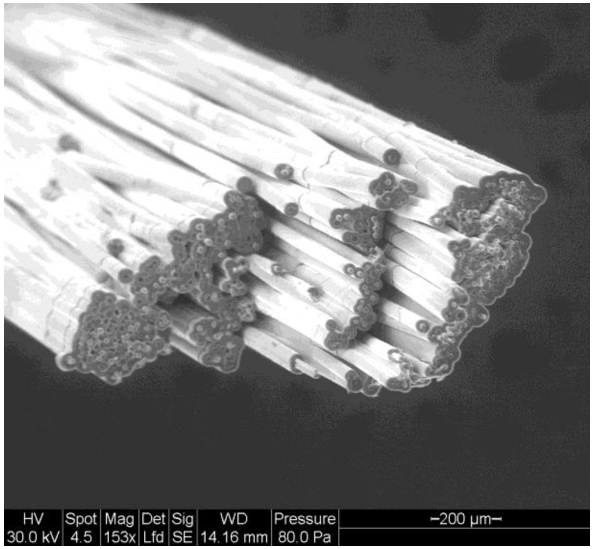


- Creep of single Hi-Nicalon S fiber tests in air were compared to that of precracked Hi-Nicalon S minicomposites.
- There is an agreement in creep-rupture data. However, more Hi-Nicalon S fiber creep tests are needed at a higher stress of 1 GPa for further confirmation.

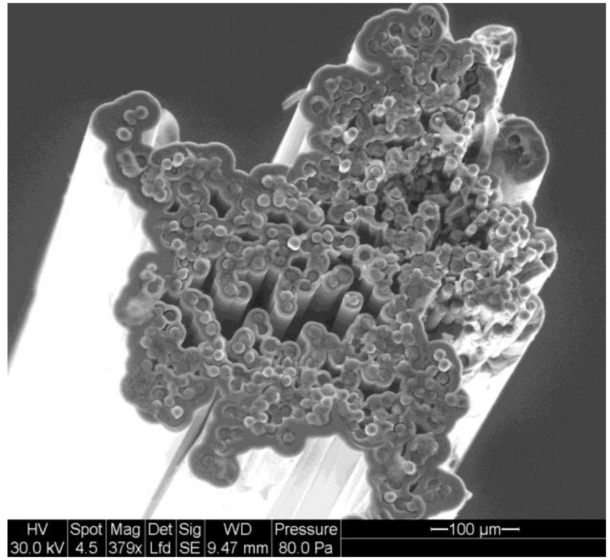
MINICOMPOSITES TESTING & CHARACTERIZATION

Materials and Properties

RR Hi-Nicalon Minicomposite

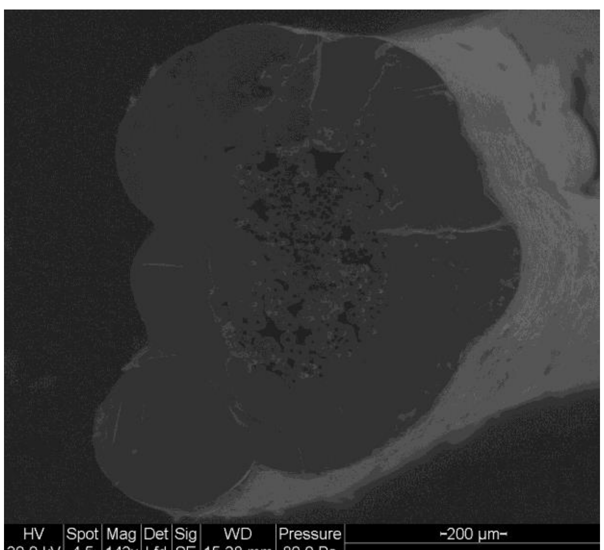


RR Hi-Nicalon S Minicomposite

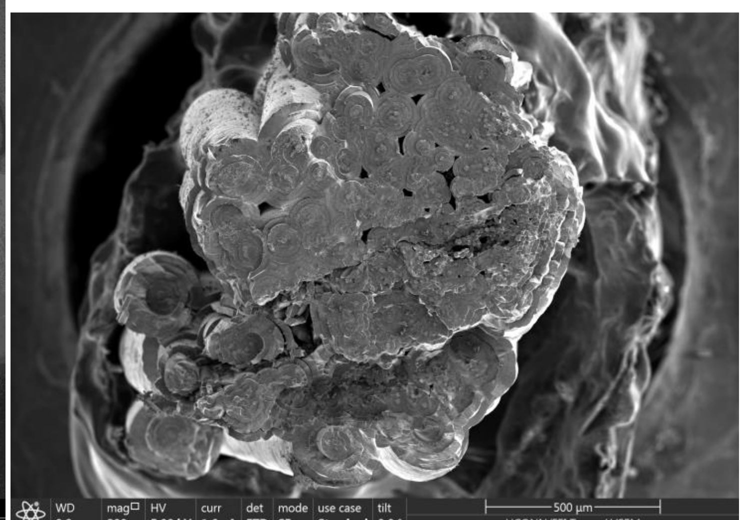


UCONN Hi-Nicalon S Minicomposite

RR 97%CVI-SiC Minicomposite



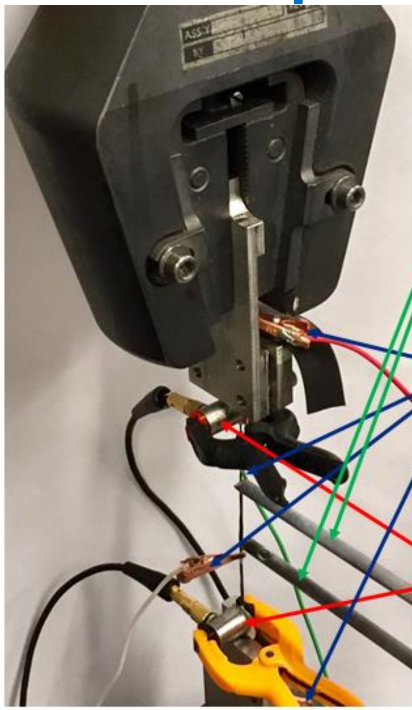
UCONN 95%CVI-SiC Minicomposite



- **The volume and mass of the fiber tow were estimated based on the average fiber diameter, number of fibers per tow, specimen length and density.**
- **The volume and mass of the interphase were estimated considering a constant thickness of 1 µm for RR minis and 0.2 µm for UCONN minis on each fiber using SEM.**
- **Then backed out the volume and mass of the matrix from ROM.**

1. Amjad Almansour, Emmanuel Maillet, Sivakumar Ramasamy and Gregory N. Morscher. "Effect of Fiber Content on Single Tow SiC Minicomposite Mechanical and Damage Properties Using Acoustic Emission": *Journal of the European Ceramic Society, 2015,* 10.1016/j.jeurceramsoc.2015.06.001.

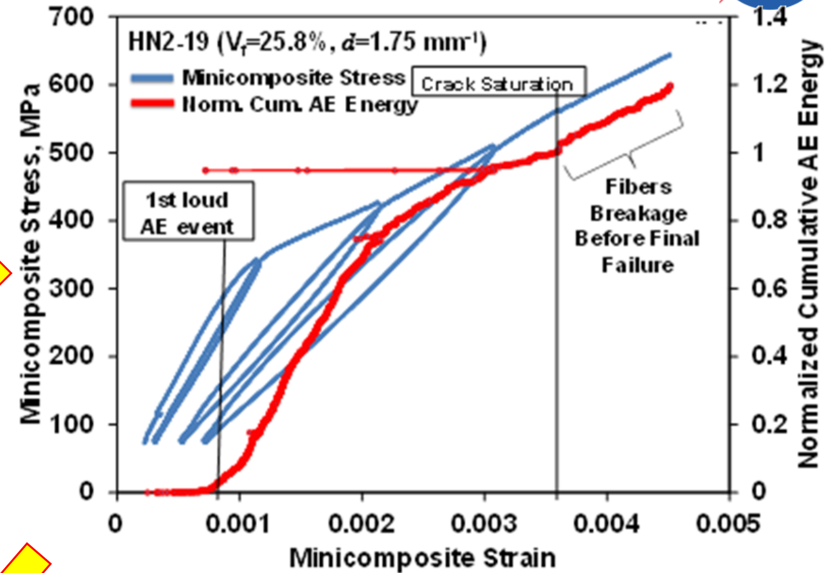
Minicomposite Crack Density at Room Temperature

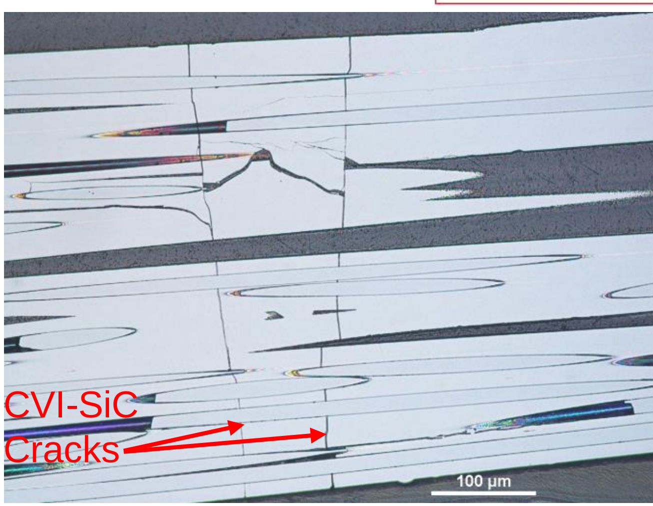


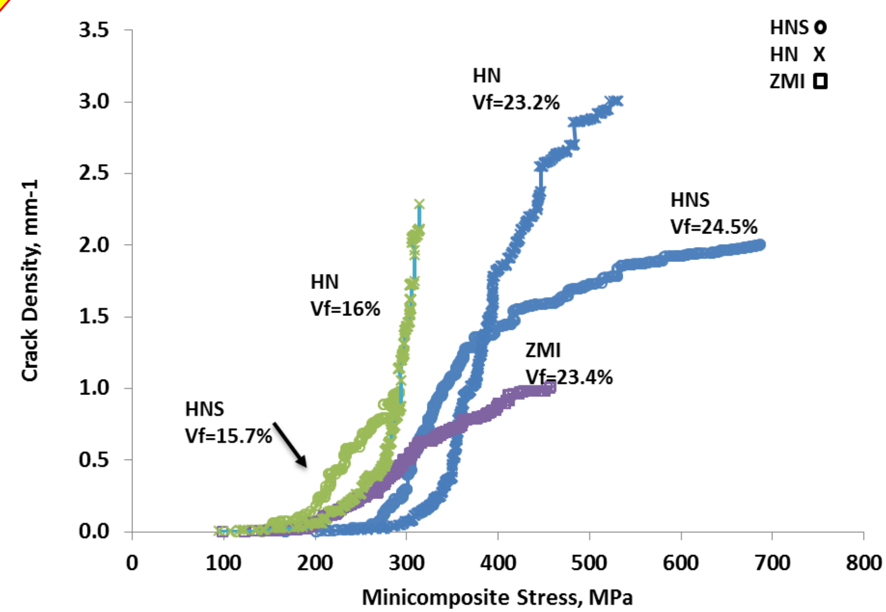
Extensometer probes for displacement measurements

Electrical Resistance: 4-probes~25 mm between inner probes

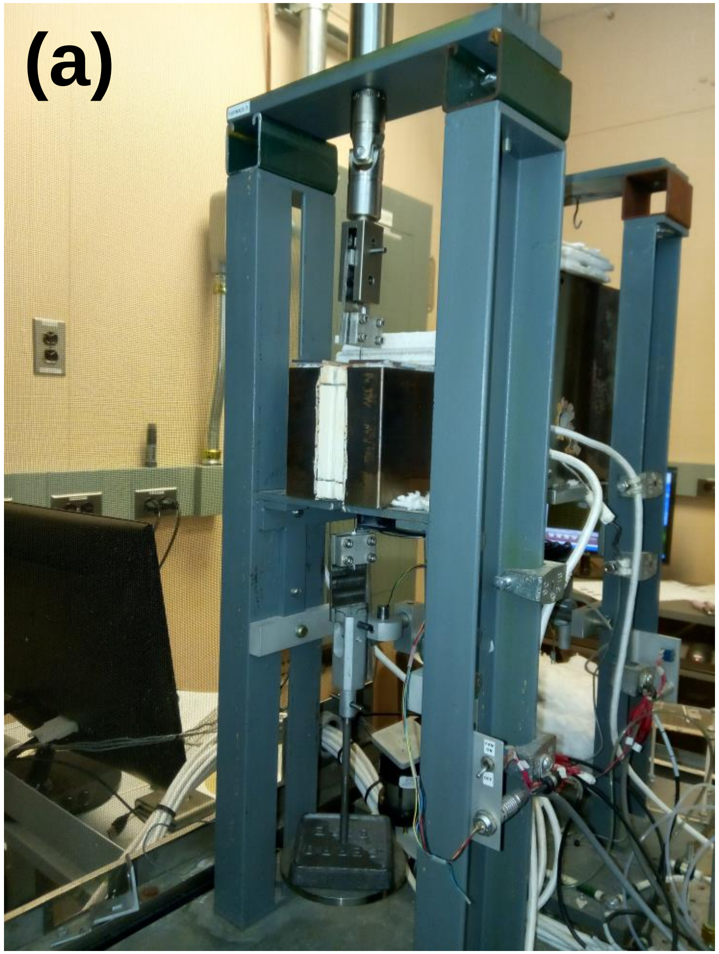
Acoustic Emission Sensors~87 mm apart

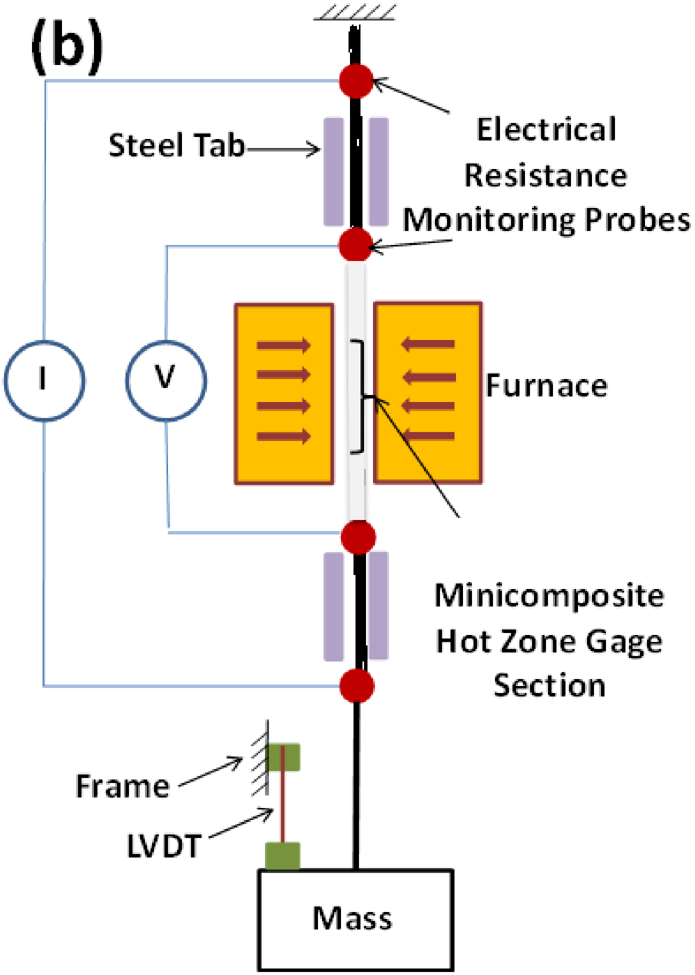






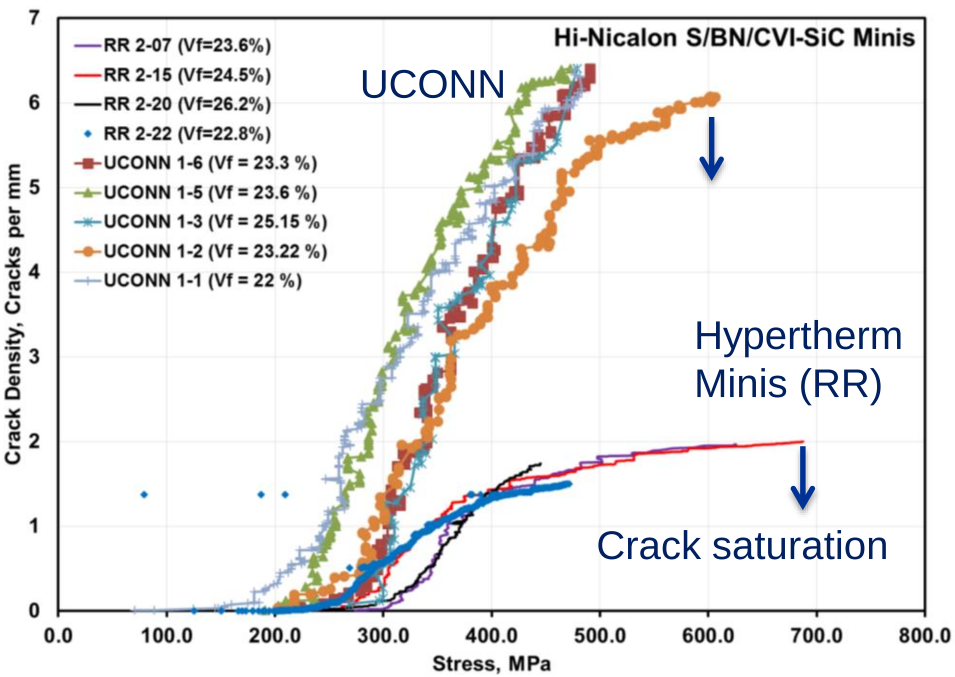
NASA Minicomposite Creep Test in Air

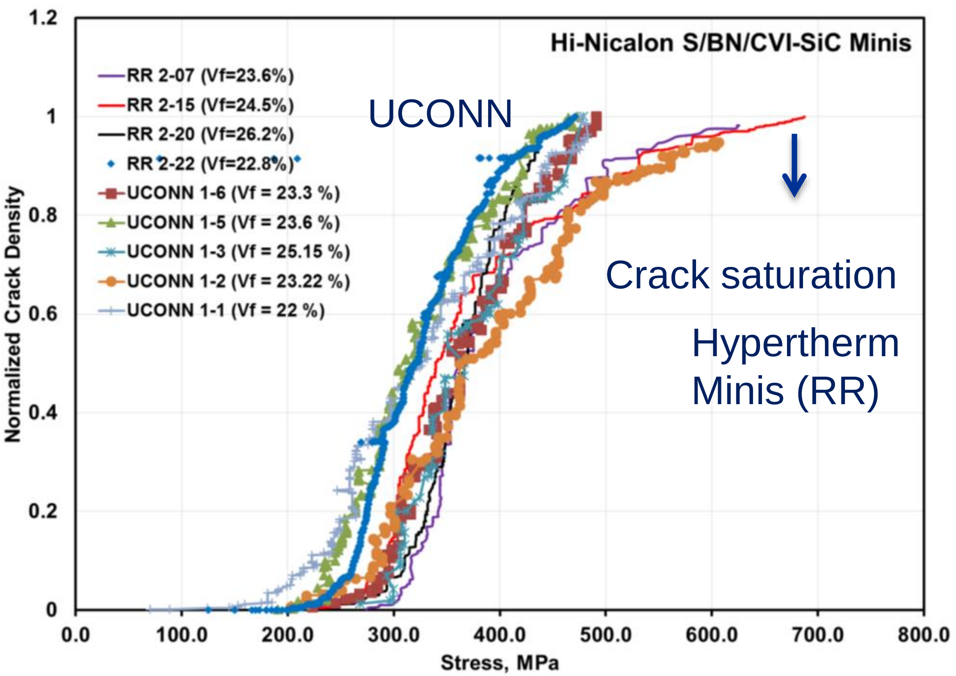




- Minicomposite creep tests in Air up to 2820 ˚F.
- 1 inch hot zone furnace

CVI-SiC Cracking Onset & Evolution



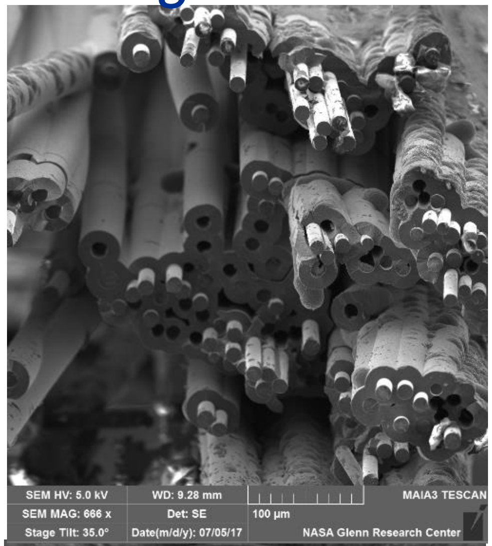


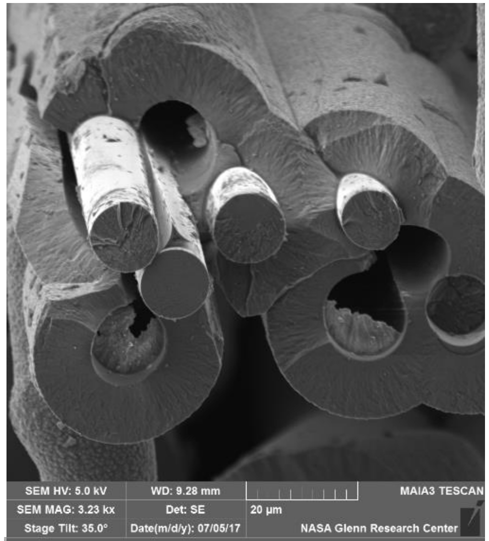
- Crack density evolution was estimated from the crack density at failure and the cumulative acoustic emission energy for the same fiber type and fiber volume fraction minicomposites.
- Minicomposite crack density evolution seems to be consistent where a good representative curve can be taken for future studies where controlled precracking is required.
- Slight difference in matrix cracking onset and evolution which might be due to fiber tow tension and/or 360° twist while processed.
- Difference in # of cracks at saturation is due to difference in interfacial shear strength and whether there is 20-30 nm Carbon layer on the fibers.

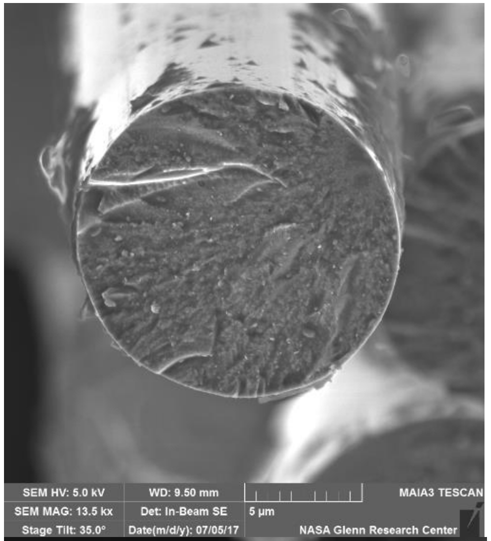
Minicomposite Fracture Surface

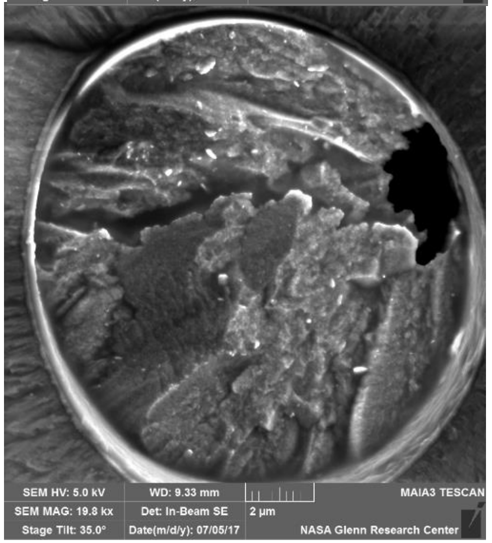
HNSUC1-2 $V_f = 23.2\%$ $\sigma_f = 2626$ MPa

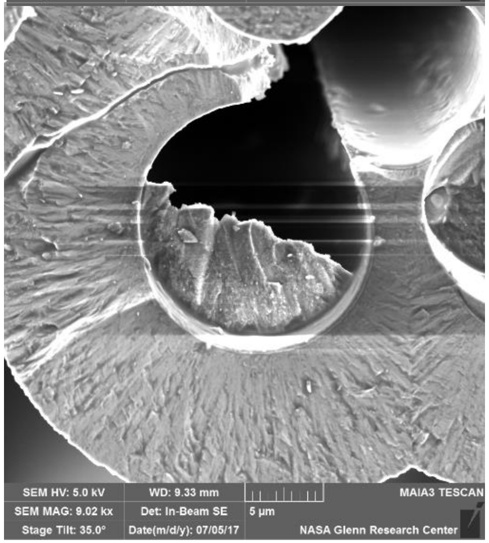
High UTS associated with fiber's small flaw size

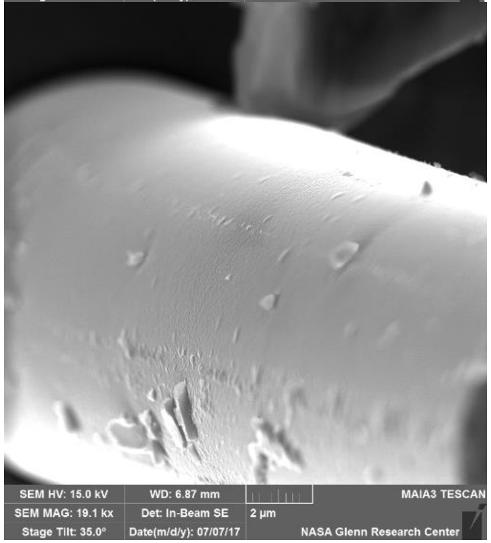




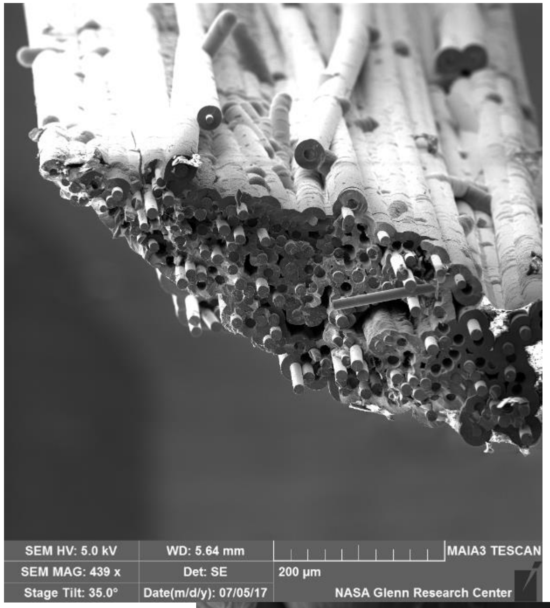








Minicomposite Fracture Surface

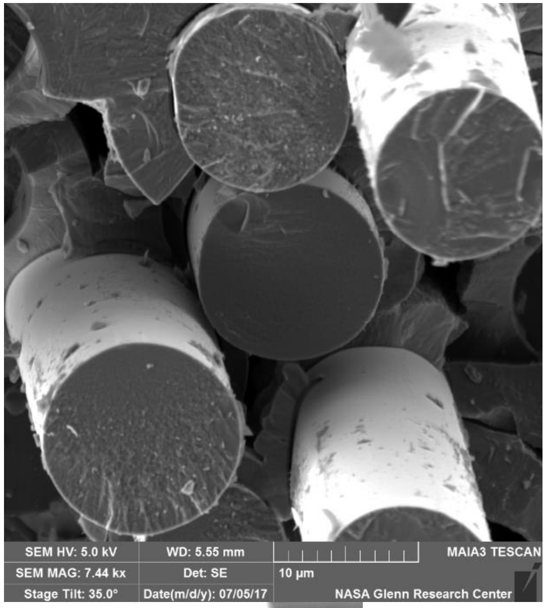


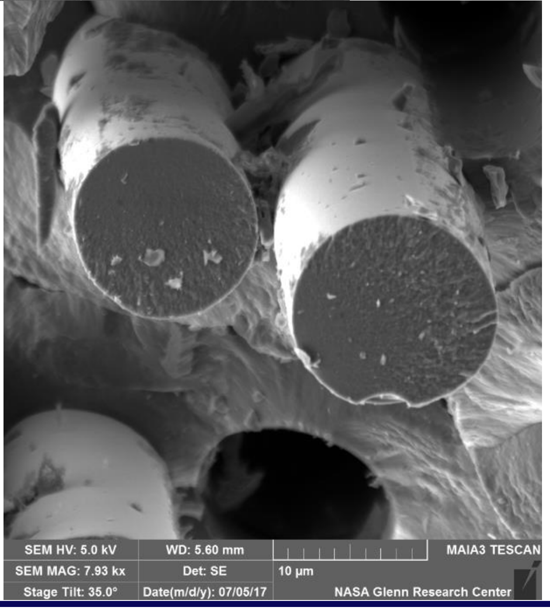
HNSUC1-6

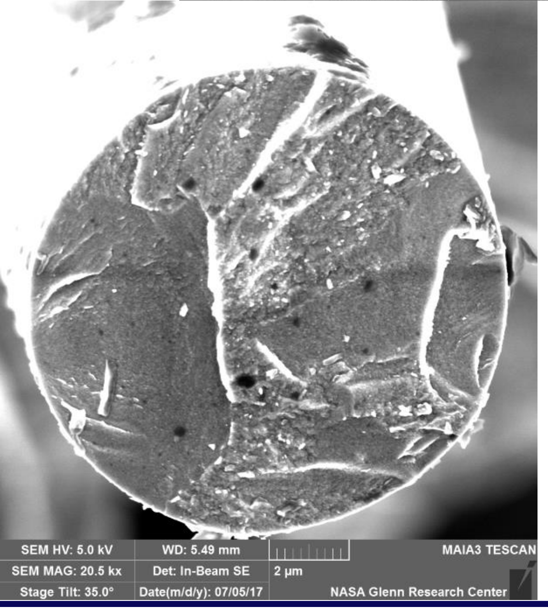
$V_f = 23.3\%$

$\sigma_f = 2093$ MPa

Low UTS associated with fiber's large flaw size

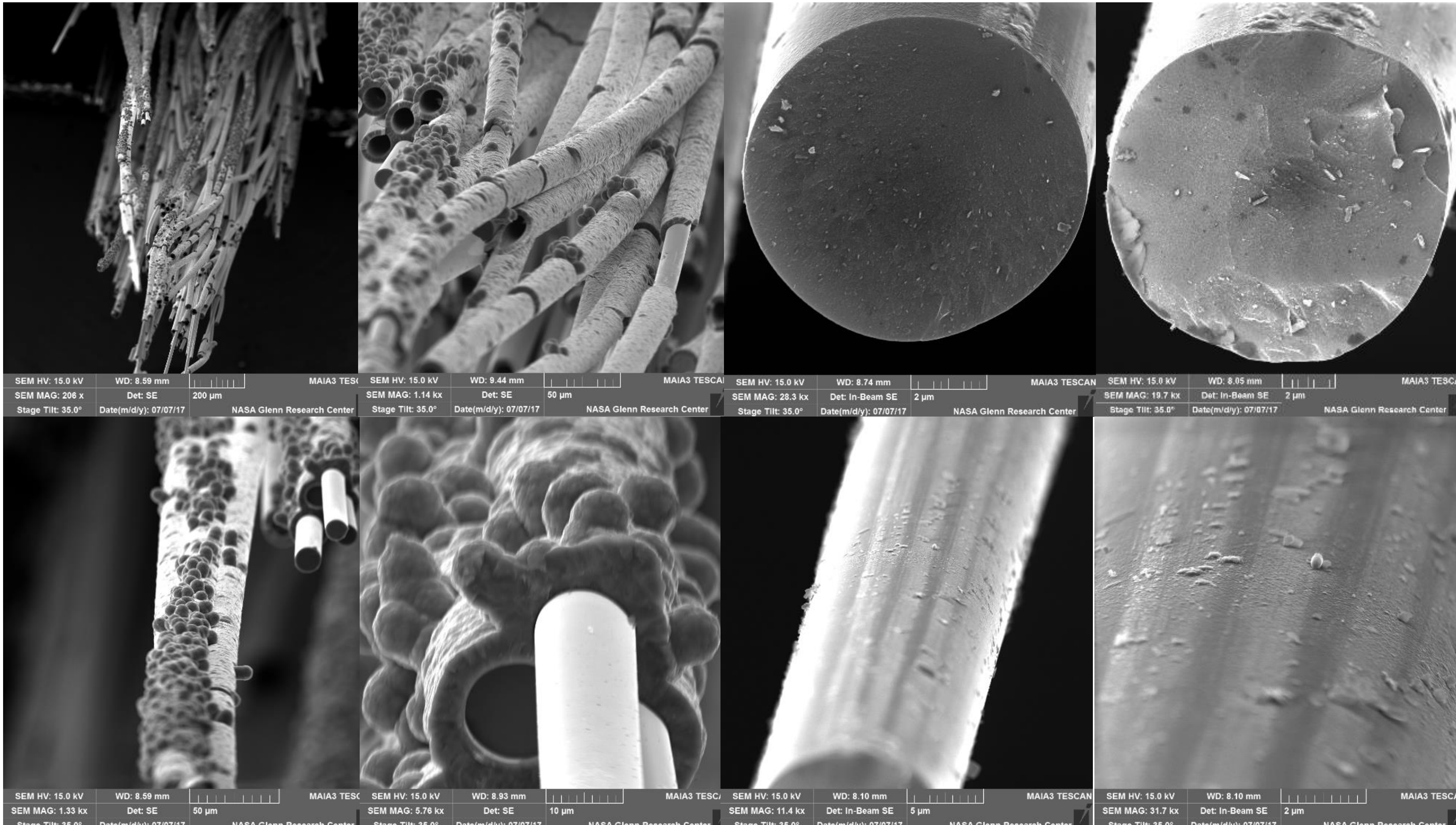






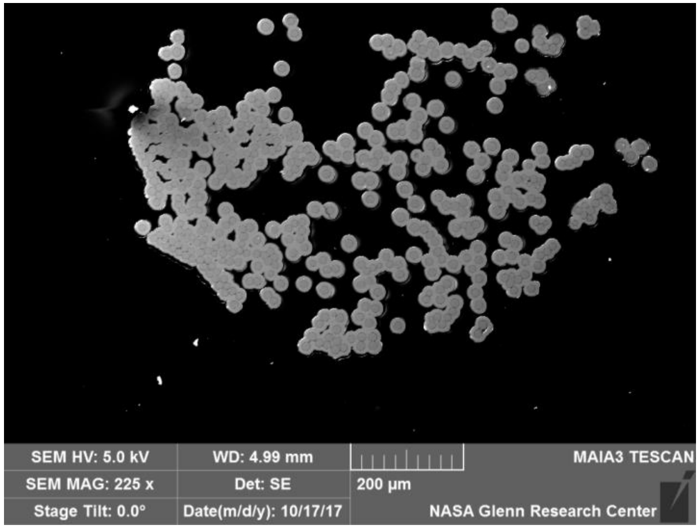
Minicomposite Fracture Surface

HNSUC2-4 $V_f = 41.4\%$ $\sigma_f = 1403$ MPa

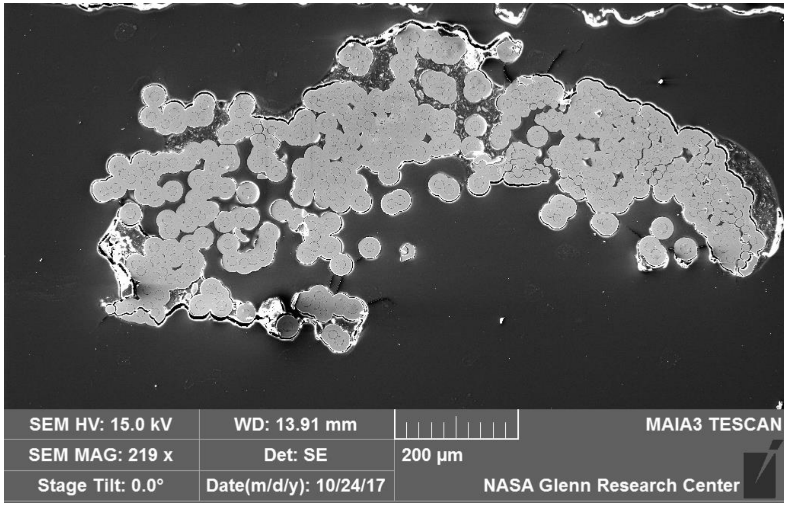


Minicomposite Cross-sections

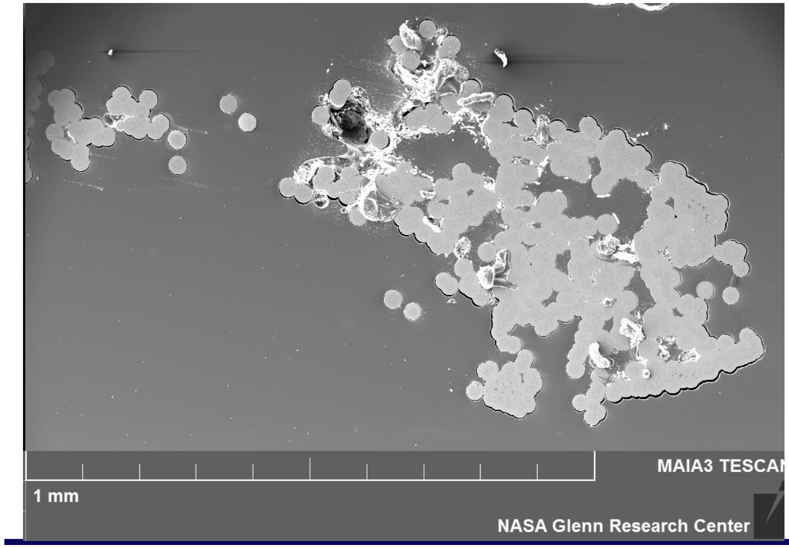
σ_{UTS} = 470 MPa, Vf = 25.15%



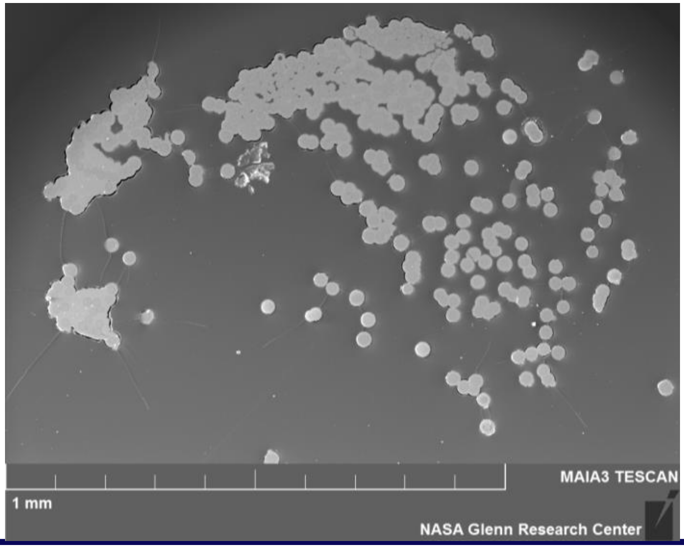
σ_{UTS} = 721 MPa, Vf = 25.64%



σ_{UTS} = 471 MPa, Vf = 23.6%



σ_{UTS} = 487 MPa, Vf = 23.3%



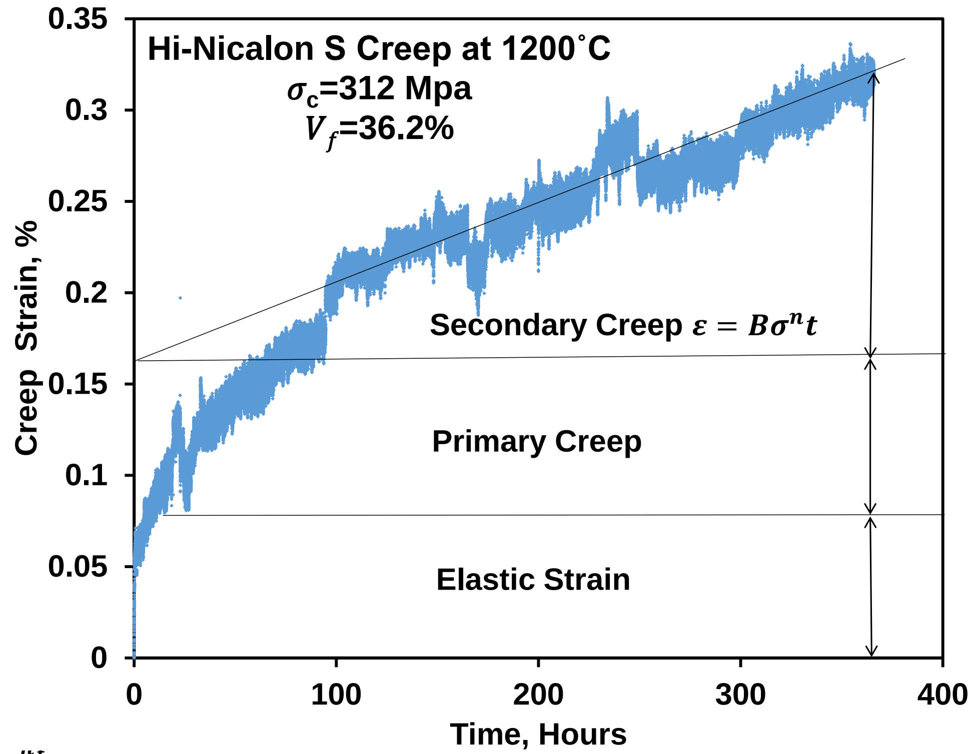
Creep Stress Transfer Model

$$\varepsilon_f = \frac{\sigma_f}{E_f} + \sigma_f A_f\left[1 - e^{-P_f t}\right] + B_f \sigma_f^{n_f} t$$

$$\varepsilon_m = \frac{\sigma_m}{E_m} + \sigma_m A_m\left[1 - e^{-P_m t}\right] + B_m \sigma_m^{n_m} t$$

$$\dot{\varepsilon}_f = \frac{\dot{\sigma}_f}{E_f} + \dot{\sigma}_f A_f - \dot{\sigma}_f A_f e^{(-P_f t)} + \sigma_f A_f P_f\left[e^{(-P_f t)}\right] + B_f \sigma_f^{n_f}$$

$$\dot{\varepsilon}_m = \frac{\dot{\sigma}_m}{E_m} + \dot{\sigma}_m A_m - \dot{\sigma}_m A_m e^{(-P_m t)} + \sigma_m A_m P_m\left[e^{(-P_m t)}\right] + B_m \sigma_m^{n_m}$$



$$\dot{\sigma}_m = \frac{\dot{\sigma} - \dot{\sigma}_f V_f}{V_m}$$

$$\dot{\sigma} = 0$$

$$\varepsilon_f = \varepsilon_m$$

$$\dot{\varepsilon}_f = \dot{\varepsilon}_m$$

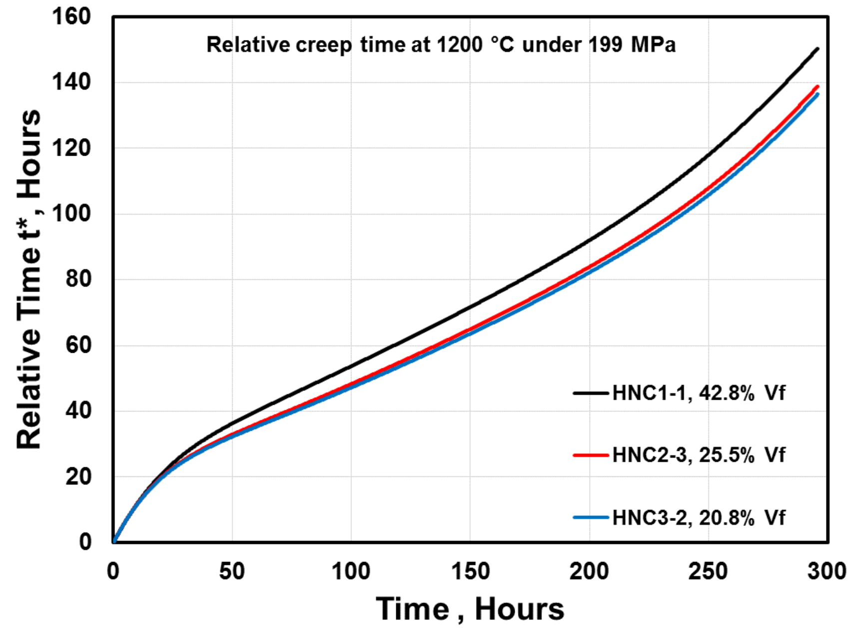
$$\dot{\sigma}_f = \frac{\sigma_m A_m P_m\left[e^{(-P_m t)}\right] - \sigma_f A_f P_f\left[e^{(-P_f t)}\right] + B_m \sigma_m^{n_m} - B_f \sigma_f^{n_f}}{\left[\frac{1}{E_f} + A_f - A_f e^{(-P_f t)} + \frac{V_f}{V_m E_m} + \frac{A_m V_f}{V_m} - \frac{A_m V_f e^{(-P_m t)}}{V_m} - B_f n_f \sigma_f^{n_f - 1} t - \frac{B_m n_m V_f \sigma_m^{n_m - 1} t}{V_m}\right]}$$

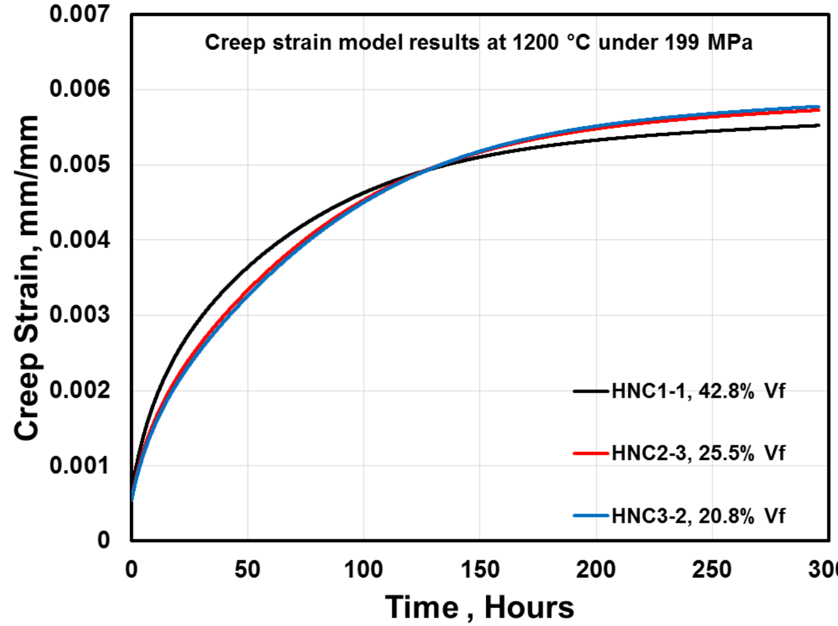
$$\sigma_{f(i)} = \sigma_{f(i-1)} + \Delta t\, \dot{\sigma}_{f(i-1)};\ \Delta t = t_i - t_{i-1}$$

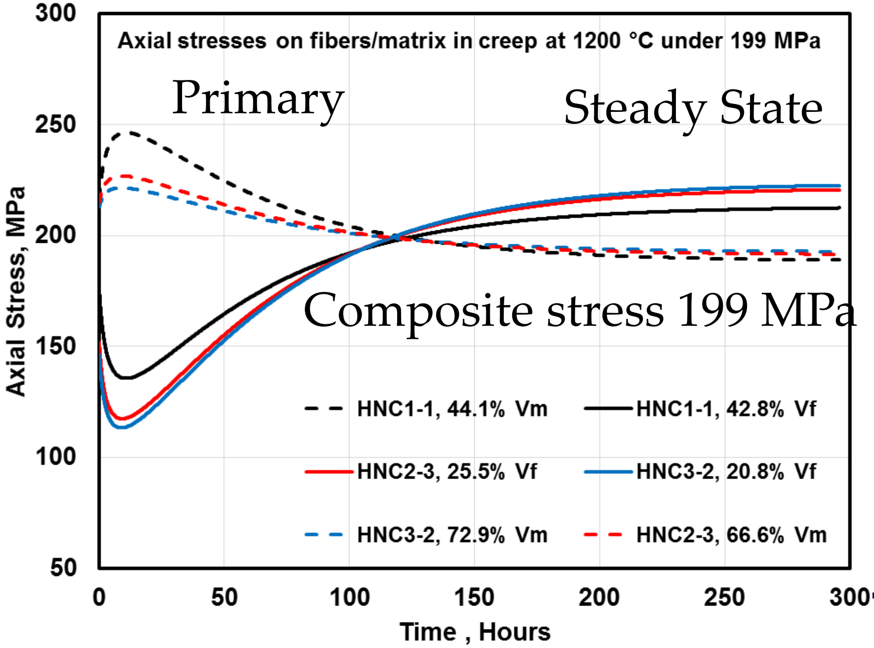
$$\sigma_{m(i)} = \sigma_{m(i-1)} + \Delta t\, \dot{\sigma}_{m(i-1)};\ \Delta t = t_i - t_{i-1}$$

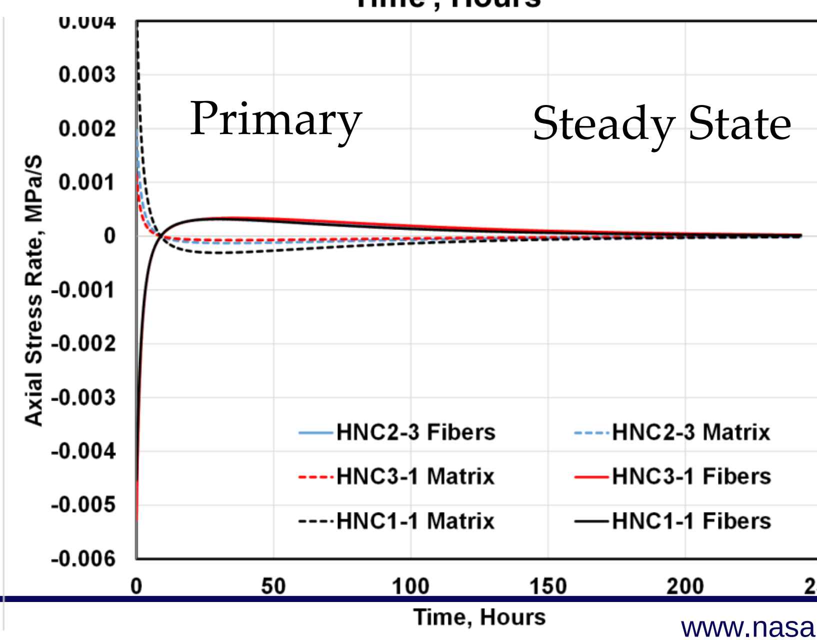
Δt is 10 seconds in the model

Creep Model Results for Constant Composite Stress









SUMMARY & CONCLUSIONS

- Testing and characterization of fiber creep in different environments has been studied.

- Minicomposite testing and characterization techniques were reviewed.

- Hi-Nicalon S fiber creep tests in air were compared to that of precracked Hi-Nicalon S minicomposites.

- University of Connecticut minicomposites were compared with Rolls-Royce minicomposites. UCONN minis have slight difference in matrix cracking onset and evolution which might be due to fiber tow tension and/or 360° twist while processed. Difference in crack densities at saturation is due to the different interfacial shear strength, BN thickness and Carbon layer on fibers. Moreover, traction and sliding marks were observed on pulled fibers.

- Fibers' flaw sizes observed on minicomposite fracture surfaces were correlated with fibers' ultimate tensile strength. Also, minicomposite cross-sections uniformity were correlated with ultimate tensile strength.

Acknowledgement

Ram Bhatt

Joseph Grady

Greg Morscher

Doug Kiser

Dan Gorican

Ron Philips

Terry McCue

NASA