Evaluation of CVI SiC/SiC Composites for High Temperature Applications

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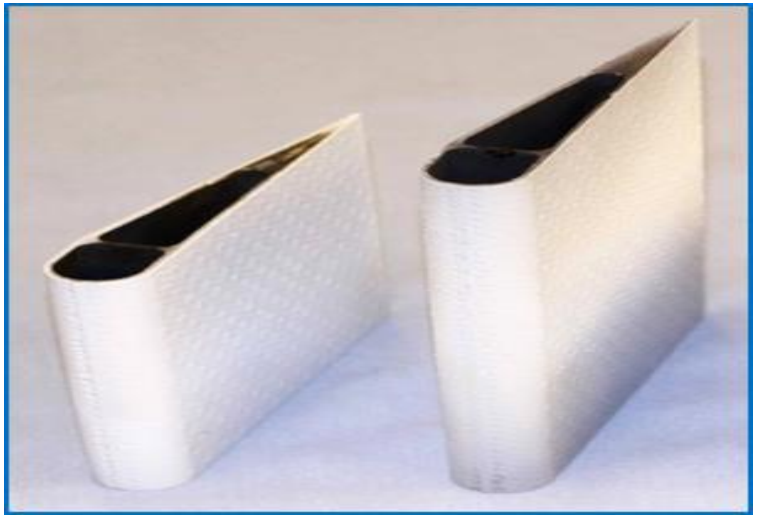
NASA Transformational Tools and Technology Project

Critical Aeronautics Technologies (CAT) Sub-Project

- High Temperature Engine Materials
- *Technical Challenge:* Develop high temperature materials for turbine engines that enable a 6% reduction in fuel burn for commercial aircraft, compared to current SOA materials



SiC/SiC Components for Gas Turbine Engines: Benefits

- **Reduced component weight (1/3 density of superalloys)**
- **Higher temperature capability/increased thermal margin**
- **Reduced cooling requirements**
- **Improved fuel efficiency** → **further increase with 2700°F CMC components**
- **Reduced emissions (NO_x and CO_2)**

Incentive to Increase Engine Operating Temperatures

Evaluation of CVI SiC/SiC Composites for High Temperature Applications

Objectives

- Establish stress-dependent and temperature-dependent parameters for modeling SiC/SiC composite creep behavior.

$$\varepsilon^{\cdot} = B\,\sigma^{n}$$

$$\mathrm{Ln}(\varepsilon^{\cdot}) = n.\mathrm{Ln}(\sigma) + \mathrm{Ln}\,(B)$$

$$B = A^{*}\,e^{\left(\frac{-Q}{RT}\right)}$$

Where $\boldsymbol{\varepsilon^{\cdot}}$ is creep strain rate, B and A are constants, $\boldsymbol{\sigma}$ is the applied stress, ***n*** is the stress exponent, **Q** is the apparent activation energy, R is the gas constant and T is the temperature in K.

- Determine damage mechanisms and failure modes under creep deformation from 2200°F (1200°C) to 2700°F (1482°C) in air.

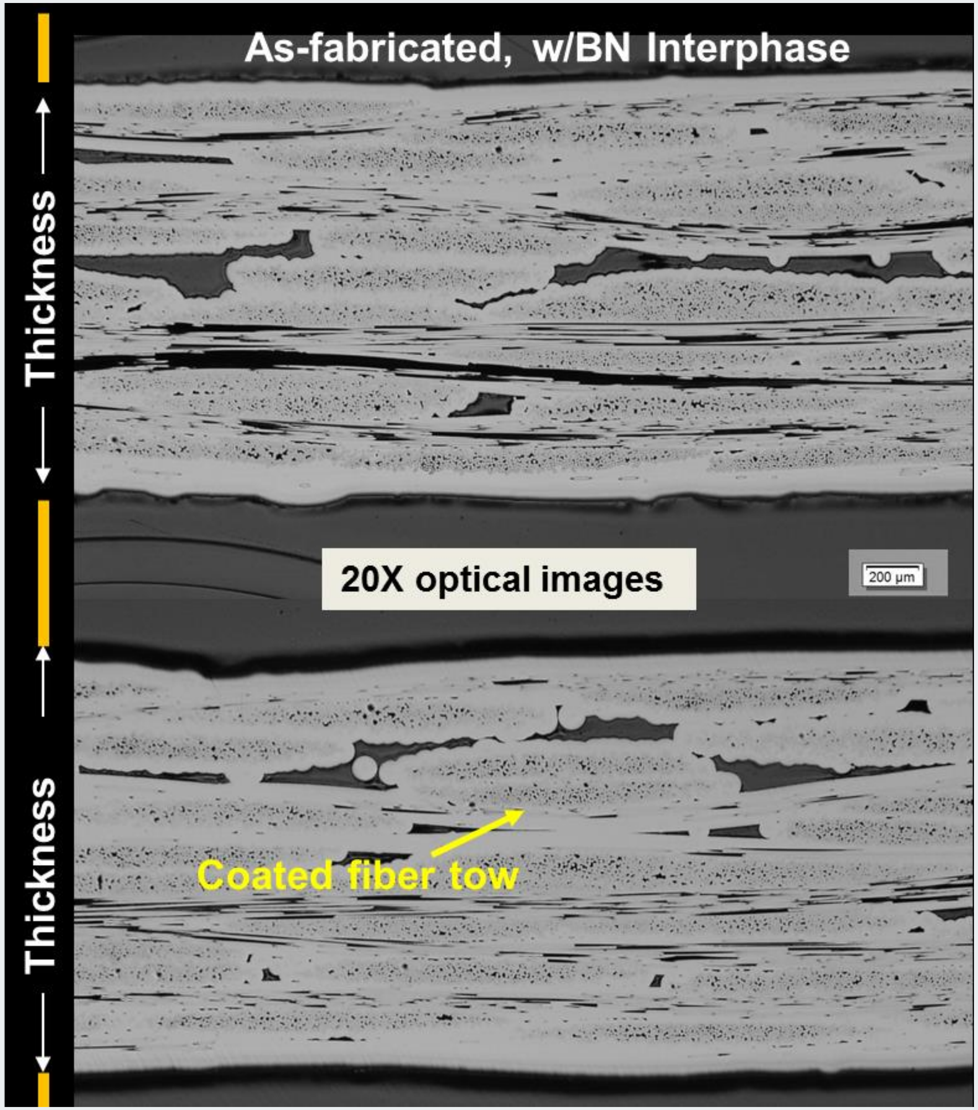
Evaluation of CVI SiC/SiC Composites for High Temperature Applications

Approach

- Building on a previous GRC study[1] of 2D CVI (chemical vapor infiltration) SiC/SiC reinforced with Sylramic™-iBN SiC fabric (manufactured by Hyper-Therm*)
- Building on previous SiC fiber and SiC/SiC CMC and minicomposites creep modeling (DiCarlo[2], Shinavski[3], Bhatt[4], and Almansour[5])
- Conduct CMC creep study at 2200°F (1200°C) to 2700°F (1482°C) —with a limited number of specimens
- Examine samples following 2700°F (1482°C) creep testing (run-out condition) and characterize their residual properties / integrity

* *Hyper-Therm HTC, Inc. became Rolls-Royce HTC*

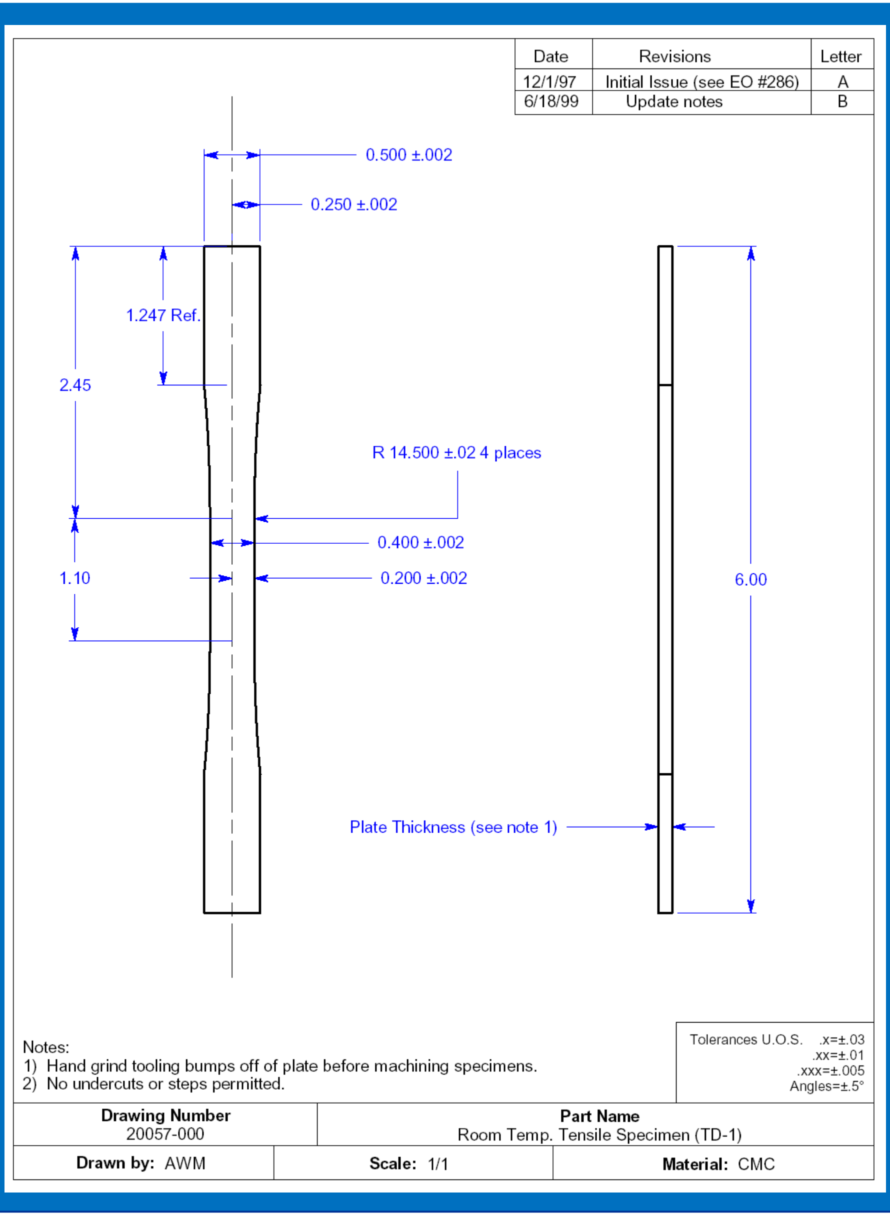
Previous Study[1]



- 2D CVI (chemical vapor infiltration) SiC/SiC reinforced with Sylramic™-iBN SiC fabric (manufactured by Hyper-Therm)

- Machined EPM geometry samples were CVI SiC seal-coated to seal the coupons' edges

EPM Tensile Geometry: 6” Dog-bone Sample



Date	Revisions	Letter
12/1/97	Initial Issue (see EO #286)	A
6/18/99	Update notes	B

Notes:
1) Hand grind tooling bumps off of plate before machining specimens.
2) No undercuts or steps permitted.

Tolerances U.O.S. .x=±.03
.xx=±.01
.xxx=±.005
Angles=±.5°

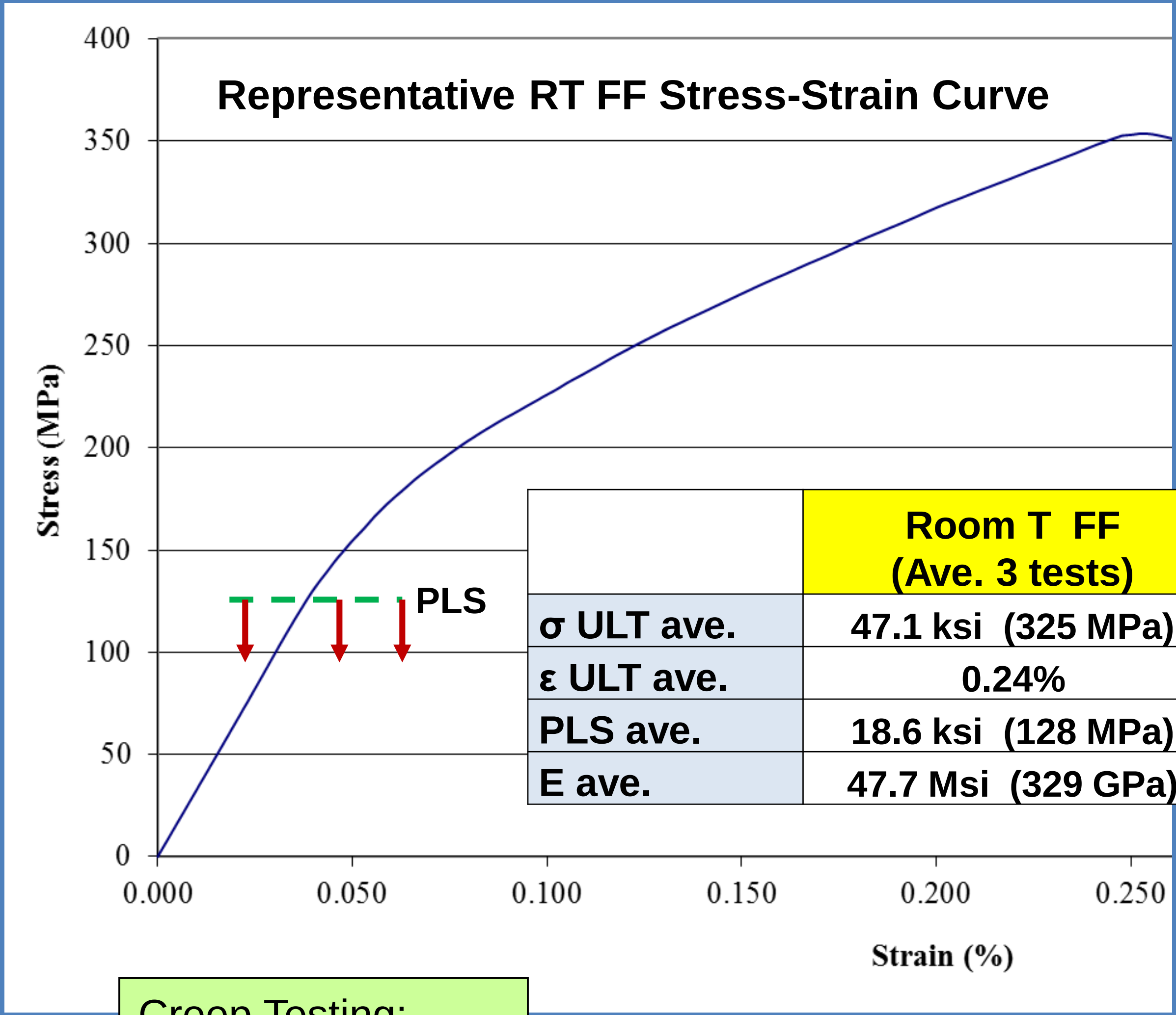
Drawing Number 20057-000	**Part Name** Room Temp. Tensile Specimen (TD-1)

Drawn by: AWM **Scale:** 1/1 **Material:** CMC

gage section:

20% reduction in width, with tapering from 0.5” (grip) to 0.4” (gage)

Sylramic^{TM}-iBN SiC Fiber-Reinforced CVI SiC Matrix CMC with BN Interphase — Fast Fracture Testing



For Comparison

	Room T FF (Ave. 3 tests)	2700°F FF (Ave. 2 tests)
σ ULT ave.	47.1 ksi (325 MPa)	31.9 ksi (220 MPa)
ε ULT ave.	0.24%	0.17%
PLS ave.	18.6 ksi (128 MPa)	
E ave.	47.7 Msi (329 GPa)	32.9 Msi (227 GPa)

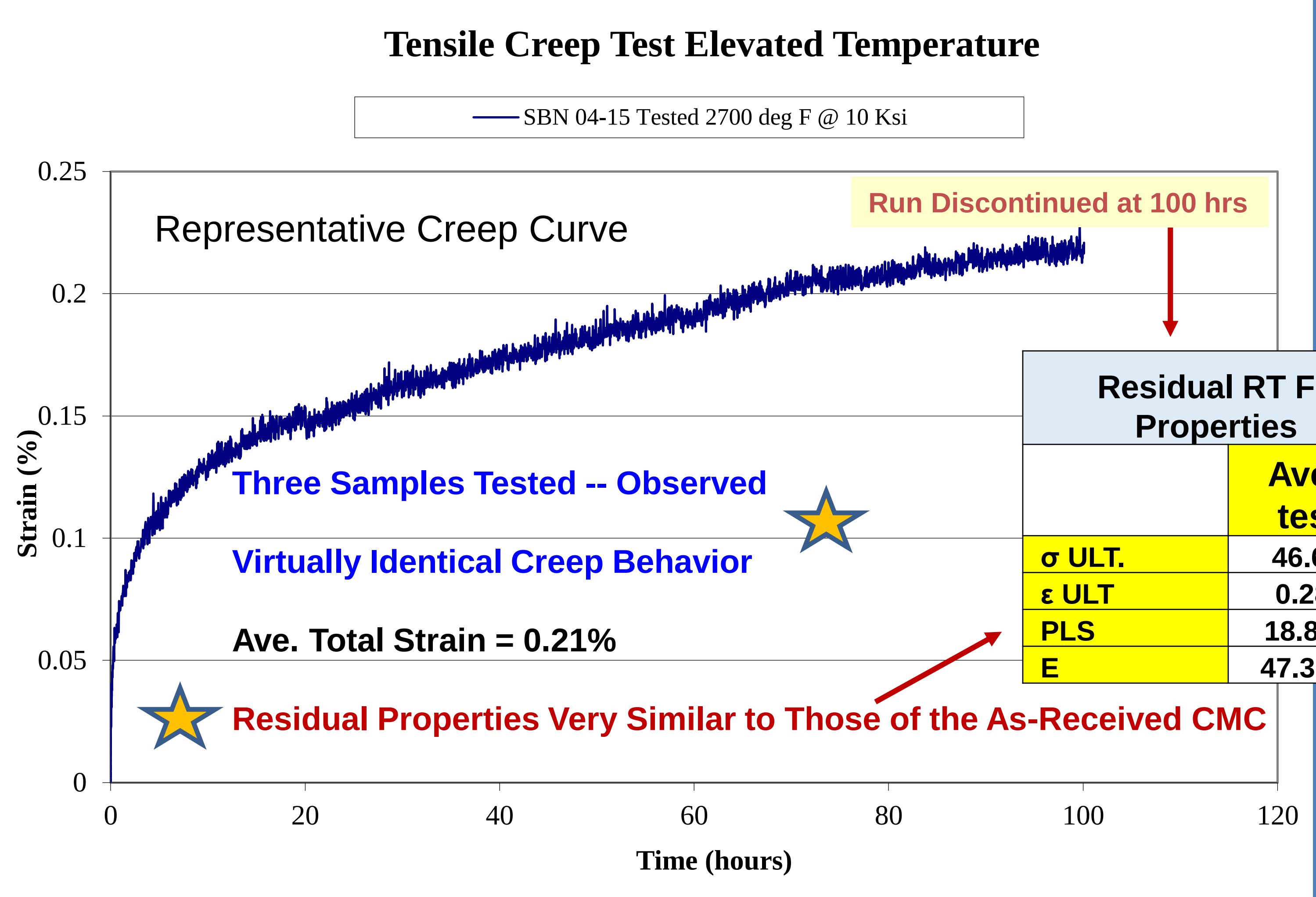
Creep Testing:
Stay below RT PLS

Ref. 1

Sylramic™-iBN SiC Fiber-Reinforced CVI SiC Matrix CMC with BN Interphase — 2700°F Tensile Creep, 10 ksi, Air

1482°C, 69 MPa

Ref. 1



Representative Creep Curve

Run Discontinued at 100 hrs

Three Samples Tested -- Observed Virtually Identical Creep Behavior

Ave. Total Strain = 0.21%

Residual Properties Very Similar to Those of the As-Received CMC

Residual RT FF Properties

	Ave. 3 tests
σ ULT.	**46.6 ksi**
ε ULT	**0.28%**
PLS	**18.8 ksi**
E	**47.3 Msi**

➡ Conclude that the matrix did not crack and that the fibers were not degraded during creep

Current Study

Material

- Similar to CMC material from previous GRC study

- 2D CVI (chemical vapor infiltration) SiC/SiC reinforced with Sylramic™-iBN SiC fabric
- Machined tensile samples were CVI SiC seal-coated to seal the coupons' edges
- Made by HTC (via NASA LaRC-funded SBIR Phase II Contract NNX11CB63C). Bequeathed by D. Brewer

- Relevant material system, especially for 2700°F applications

Current Study

Creep of CVI SiC/SiC CMC [3]

- When CMCs are loaded *below* the matrix cracking stress (PLS), fibers are not exposed to oxidation damage and they carry a fraction of the applied load.
- If the matrix is more creep resistant, the fiber unloads over time and matrix load increases, which increases the possibility of matrix damage.

2D CVI SiC/SiC Reinforced with Sylramic™-iBN:
Creep in Air— *5 Different Conditions, and RT FF of As-Received*

Specimen ID	Test Condition (Temperature: °F, Stress: ksi, Time: hrs)
1520-S2-1	2700°F, 10 ksi for 100 hrs
1520-S2-2	2700°F, SPLCF*, R=0.5, 5 / 10 ksi for 100 hrs
1520-S2-3	2700°F, 10 ksi for 100 hrs, 12.5 ksi for 100 hrs, 15 ksi for 100 hrs
1520-S2-4	RT FF Tensile Test
1520-S2-5	2200°F, 12.5 ksi for 100 hrs, 2460°F, 12.5 ksi for 100 hrs, 2700°F, 12.5 ksi for 100 hrs
1520-S2-6	2700°F, 12.5 ksi for 300 hrs

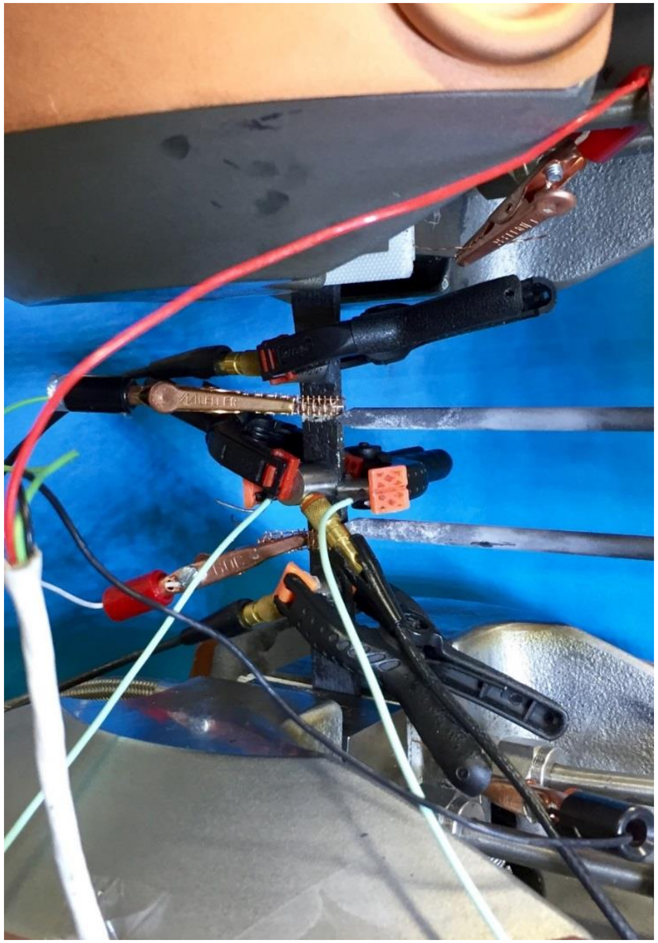
Creep Testing: Stay below RT PLS

* **Sustained Peak Low Cycle Fatigue**

2D CVI SiC/SiC Reinforced with Sylramic^{TM}-iBN:
Testing at Room Temp.: As-Rec. and Following Creep in Air

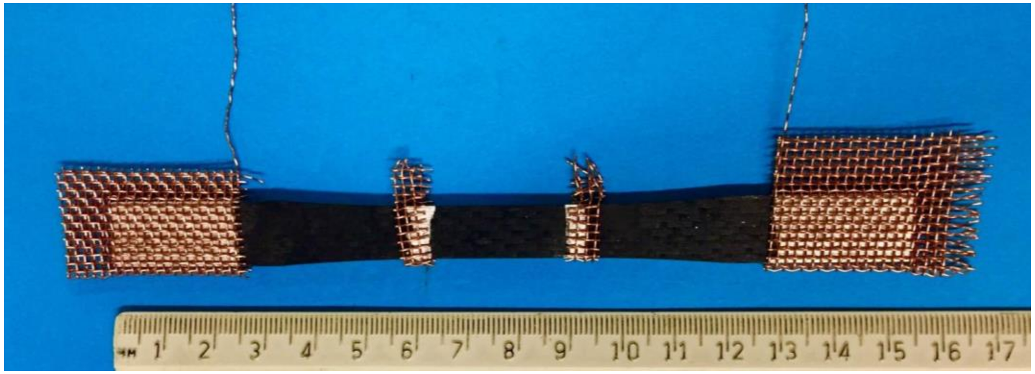
Used various characterization approaches (AE, resistivity, hysteresis testing, and fractography) to determine which ones provide the most useful post-test information.

[*In Progress*]



S2-6 (Post-creep)

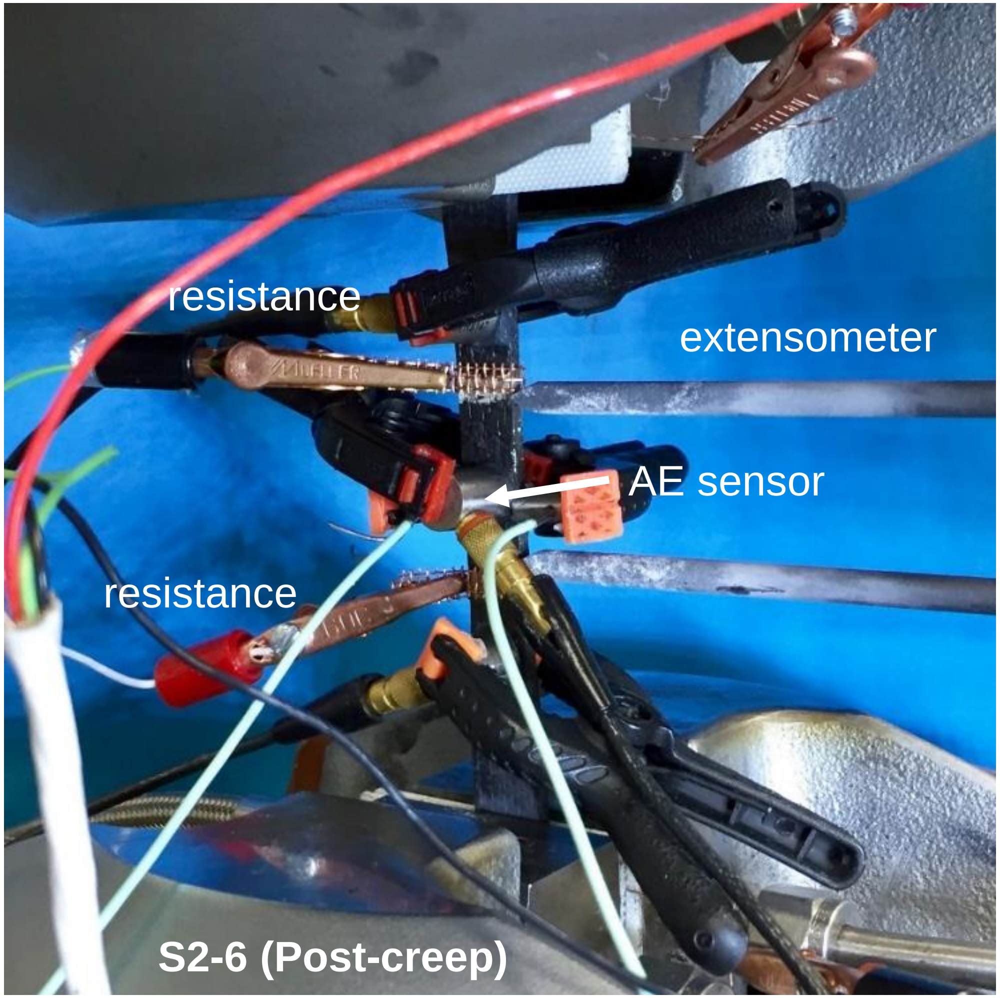
Unique Acoustic Emission (AE) Set-up

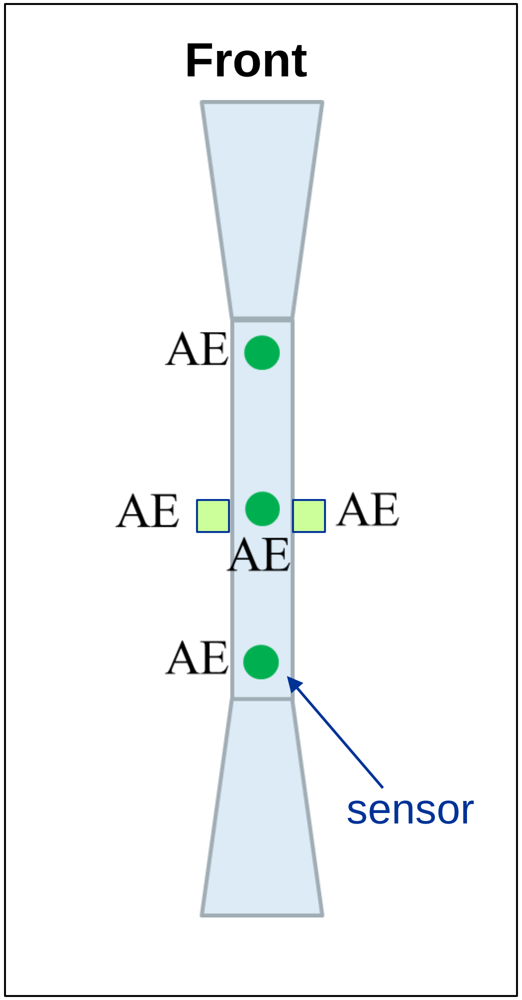


S2-4 (As-Fabricated Sample)

Prepped for Resistivity Measurement

2D CVI SiC/SiC Reinforced with Sylramic™-iBN:
Testing at Room Temp.: As-Rec. and Following Creep in Air

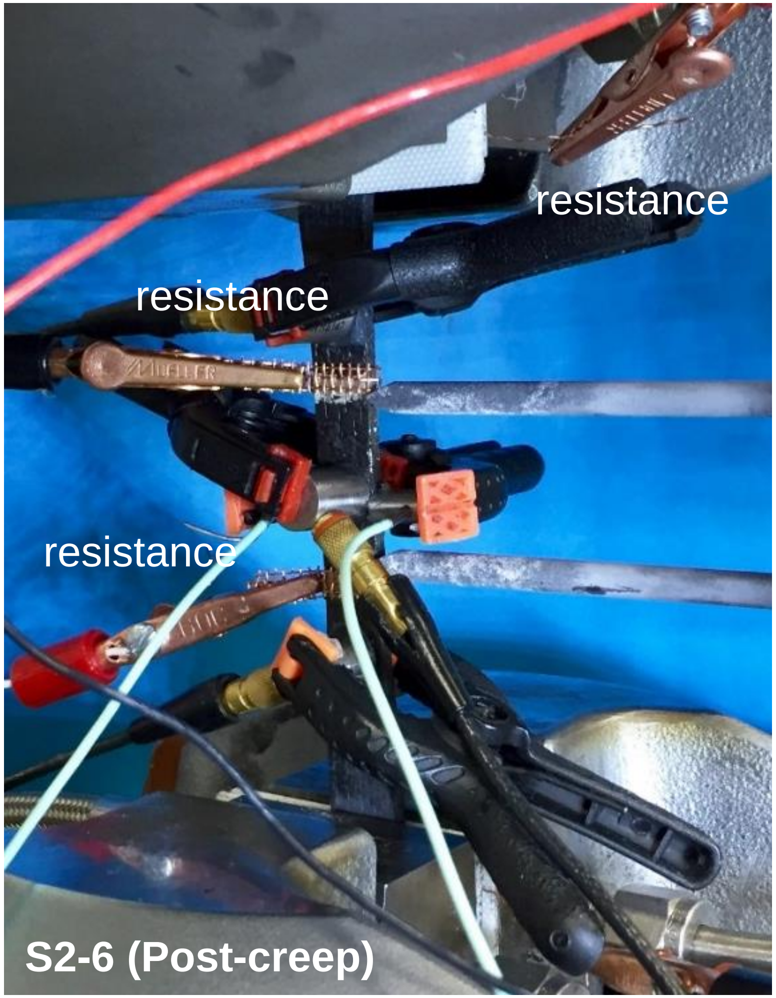


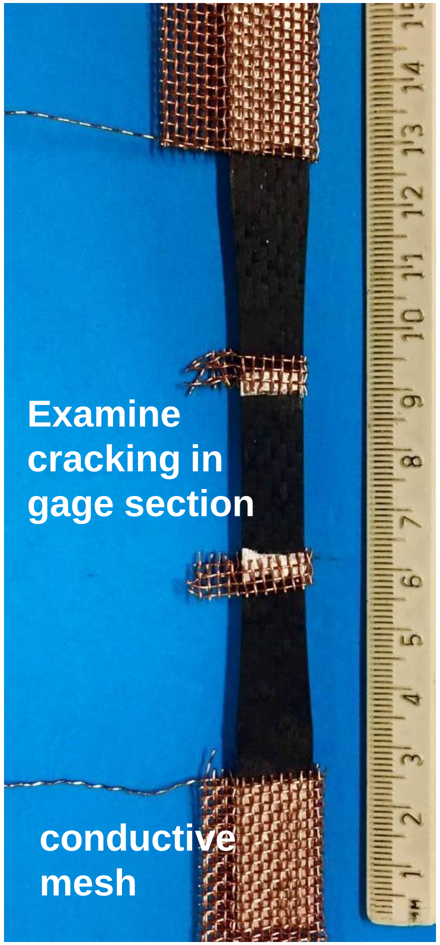


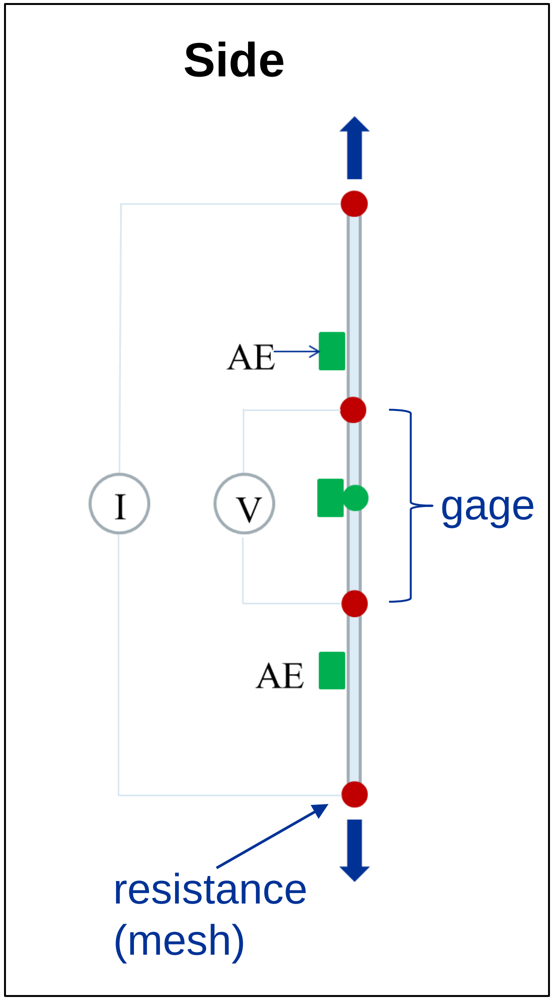
Unique Acoustic Emission (AE) Set-up for Characterizing Cracking

2D CVI SiC/SiC Reinforced with Sylramic™-iBN:

Testing at Room Temp.: As-Rec. and Following Creep in Air



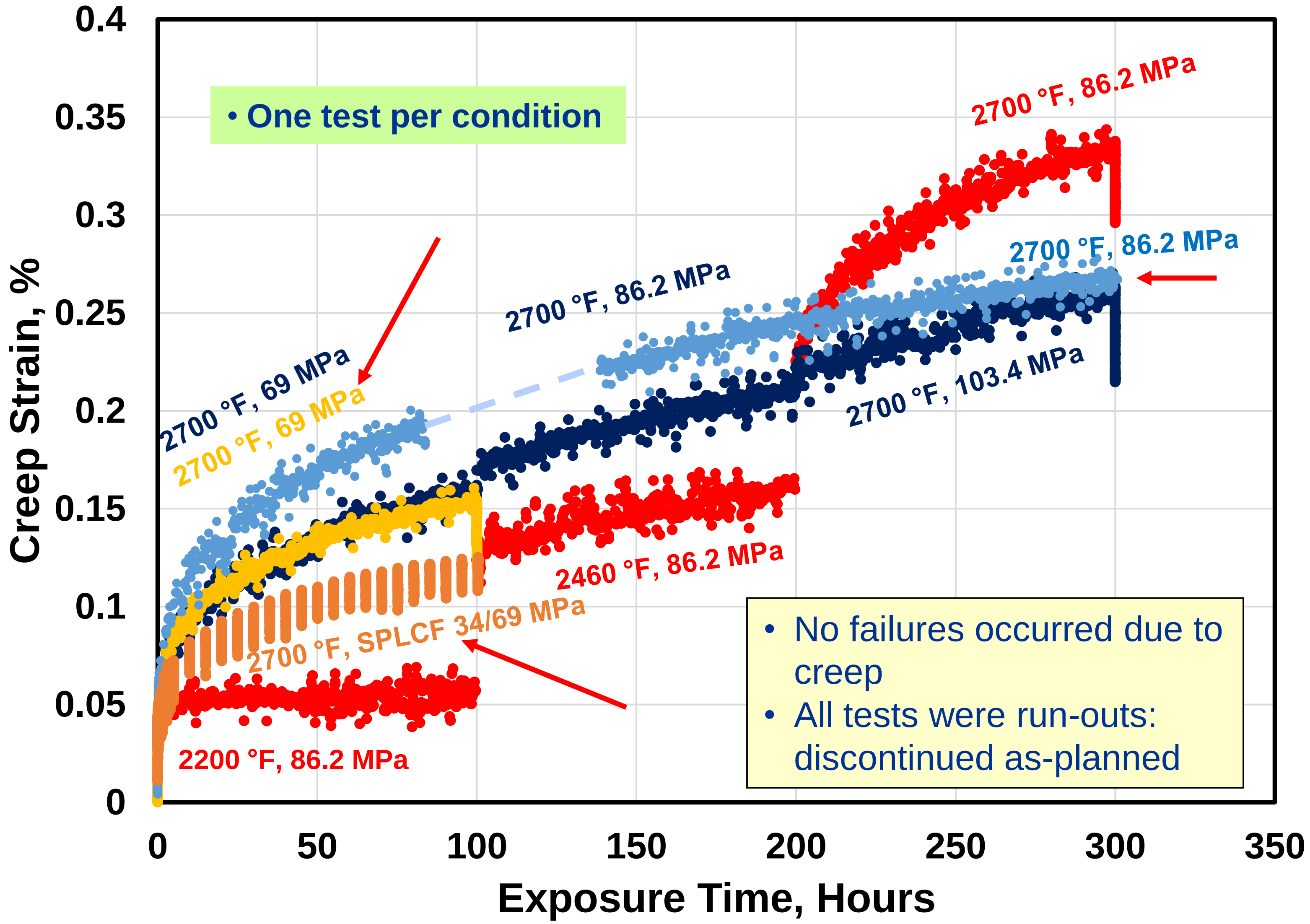




Also Collecting Resistivity Data for Characterizing Cracking

2D CVI SiC/SiC Reinforced with Sylramic^{TM}-iBN:

Creep in Air— *Results of 5 different testing conditions*

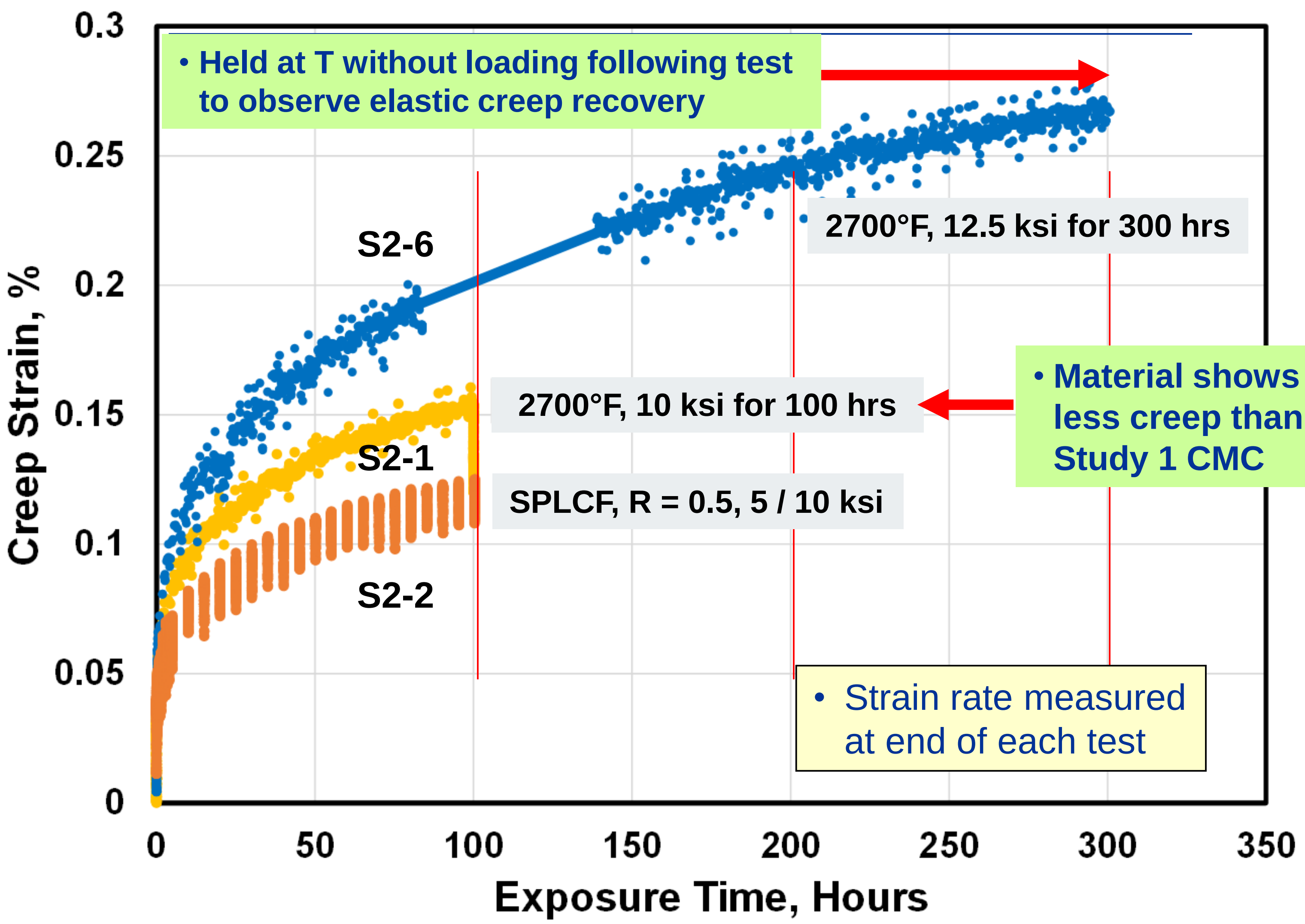


- One test per condition

- No failures occurred due to creep
- All tests were run-outs: discontinued as-planned

2D CVI SiC/SiC Reinforced with Sylramic™-iBN:

Creep in Air at 2700°F (1482°C)



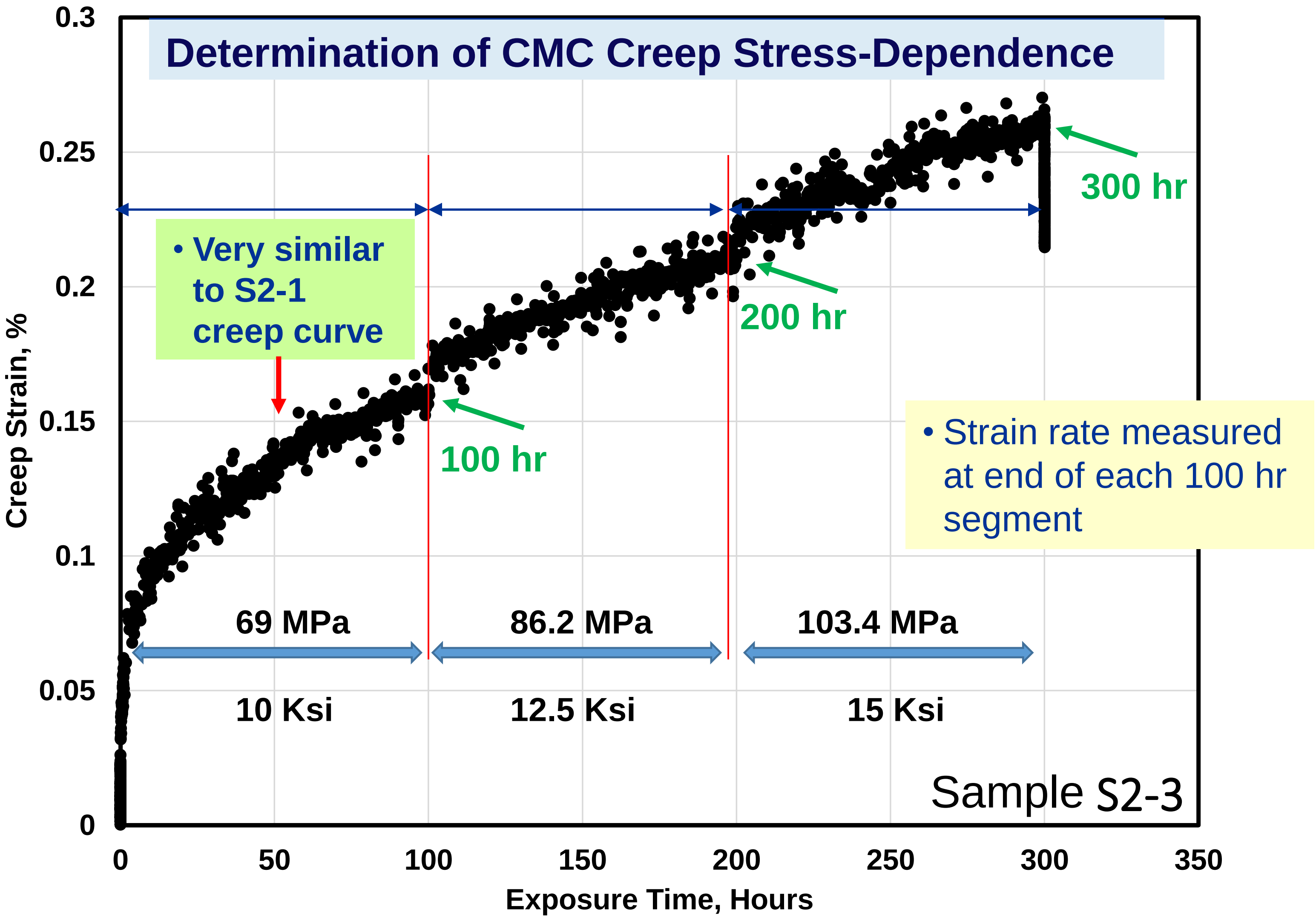
- Held at T without loading following test to observe elastic creep recovery

- Material shows less creep than Study 1 CMC

- Strain rate measured at end of each test

2D CVI SiC/SiC Reinforced with Sylramic™-iBN:

Creep in Air at 2700°F (1482°C)— *Exposed to 3 stresses*

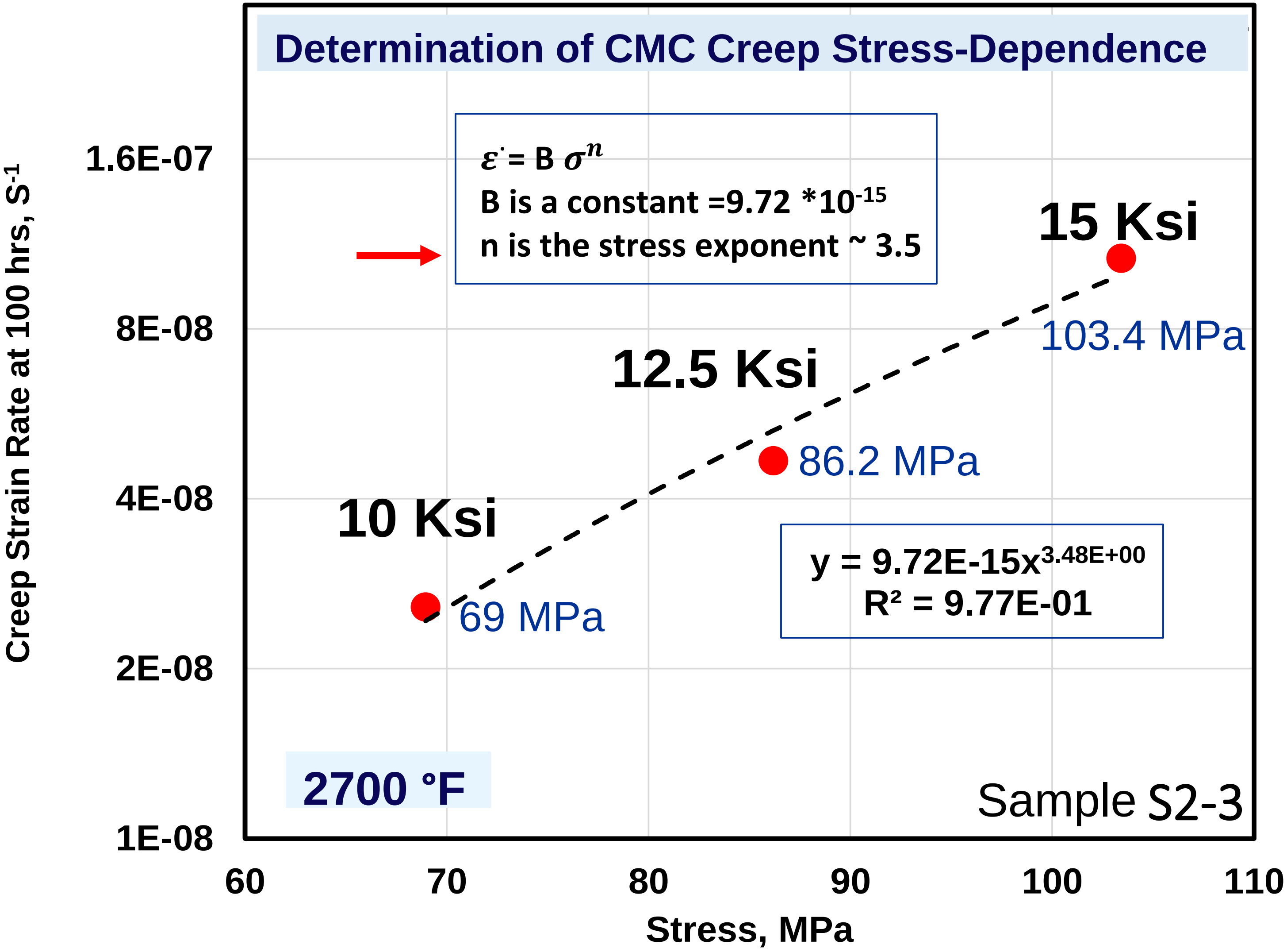


- Very similar to S2-1 creep curve

- Strain rate measured at end of each 100 hr segment

2D CVI SiC/SiC Reinforced with Sylramic™-iBN:

Creep in Air at 2700°F (1482°C)



$\dot{\varepsilon} = B\,\sigma^{n}$

B is a constant =9.72 $*10^{-15}$

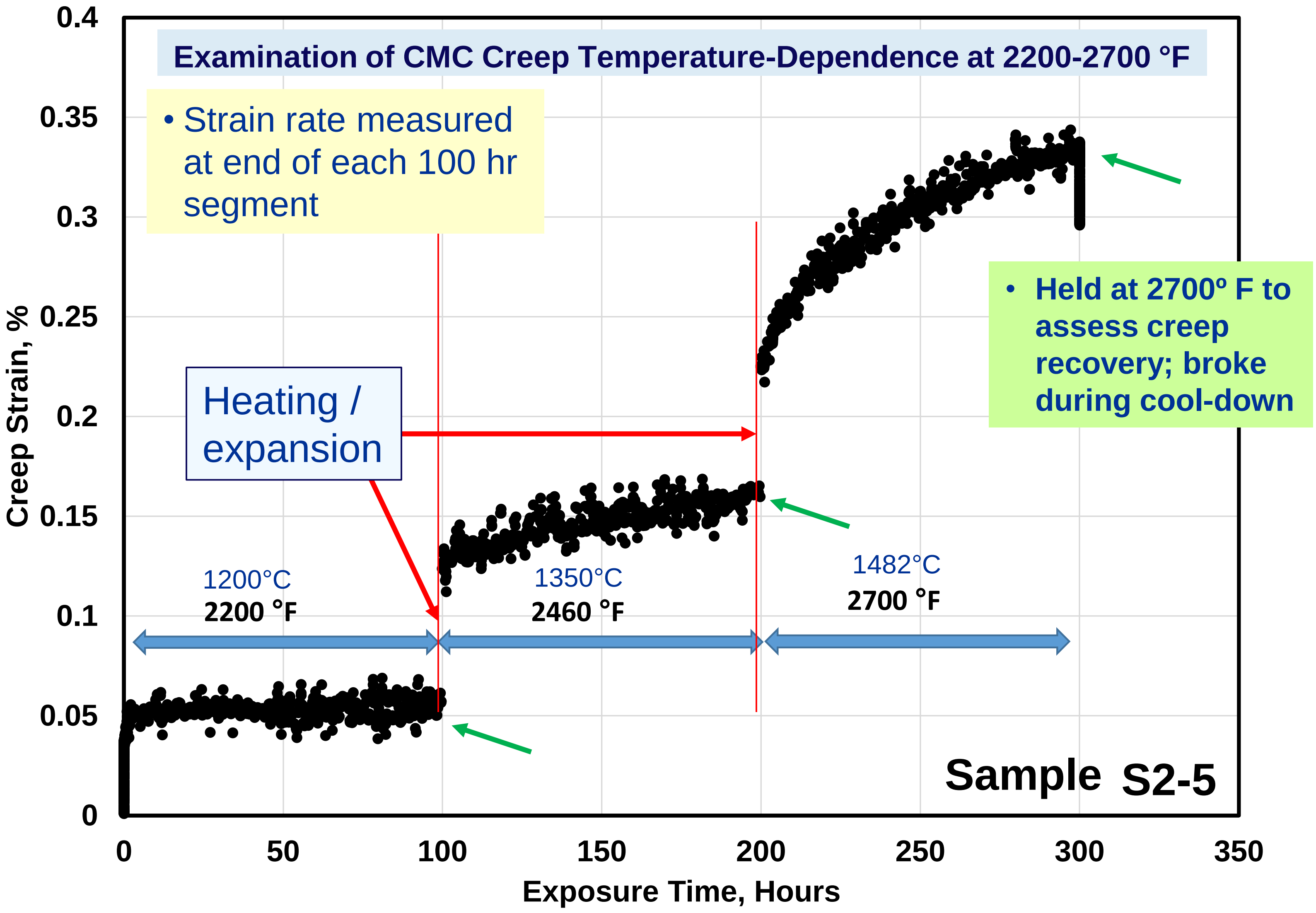
n is the stress exponent ~ 3.5

$y = 9.72E\text{-}15x^{3.48E+00}$

$R^2 = 9.77E\text{-}01$

2D CVI SiC/SiC Reinforced with Sylramic™-iBN:

Creep in Air, 12.5 ksi (86.2 MPa)—Exposed to 3 Temperatures



Examination of CMC Creep Temperature-Dependence at 2200-2700 °F

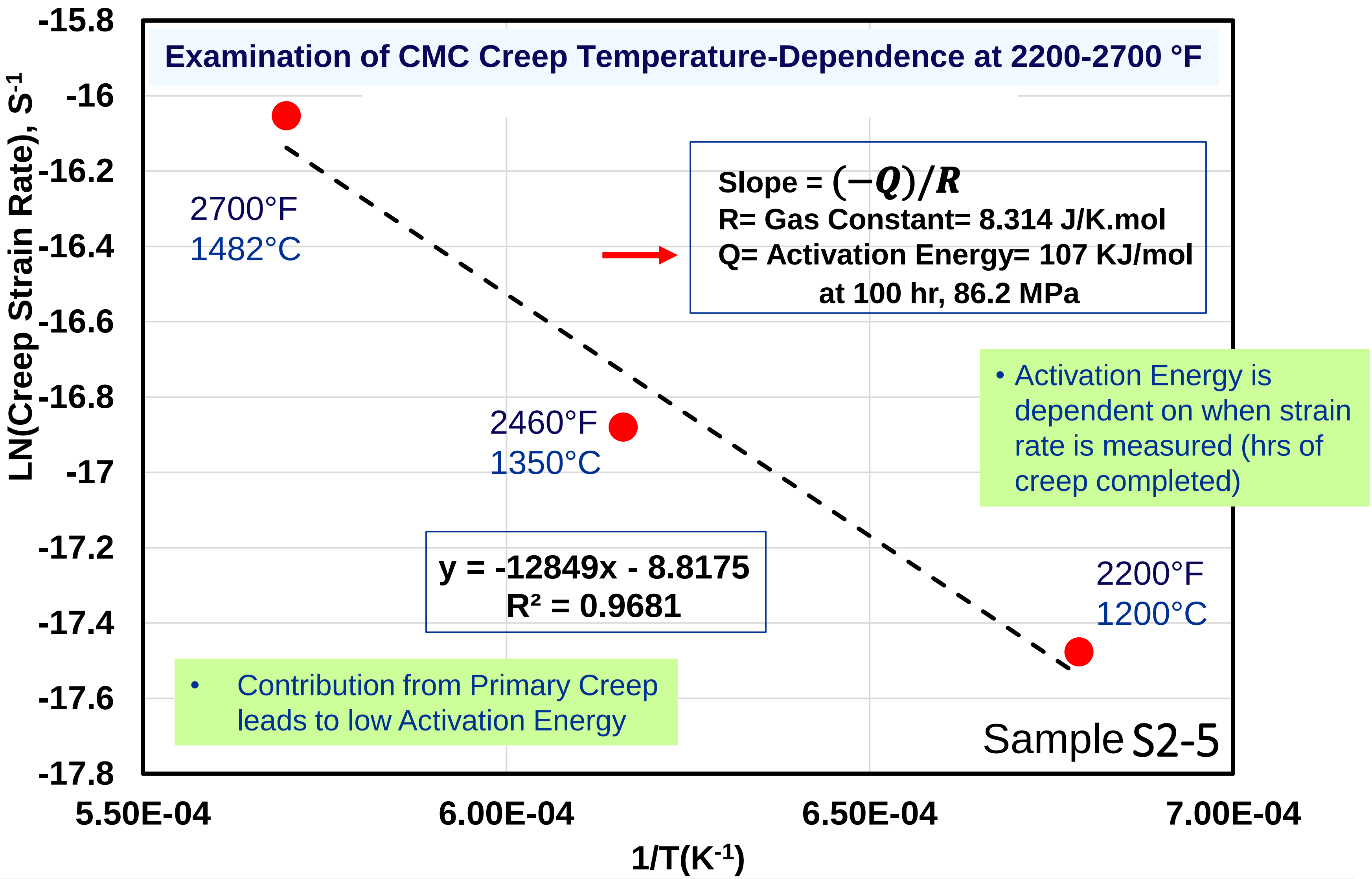
- Strain rate measured at end of each 100 hr segment

Heating / expansion

- **Held at 2700º F to assess creep recovery; broke during cool-down**

2D CVI SiC/SiC Reinforced with Sylramic^{TM}-iBN:

Creep in Air, 12.5 ksi (86.2 MPa)—Exposed to 3 Temperatures



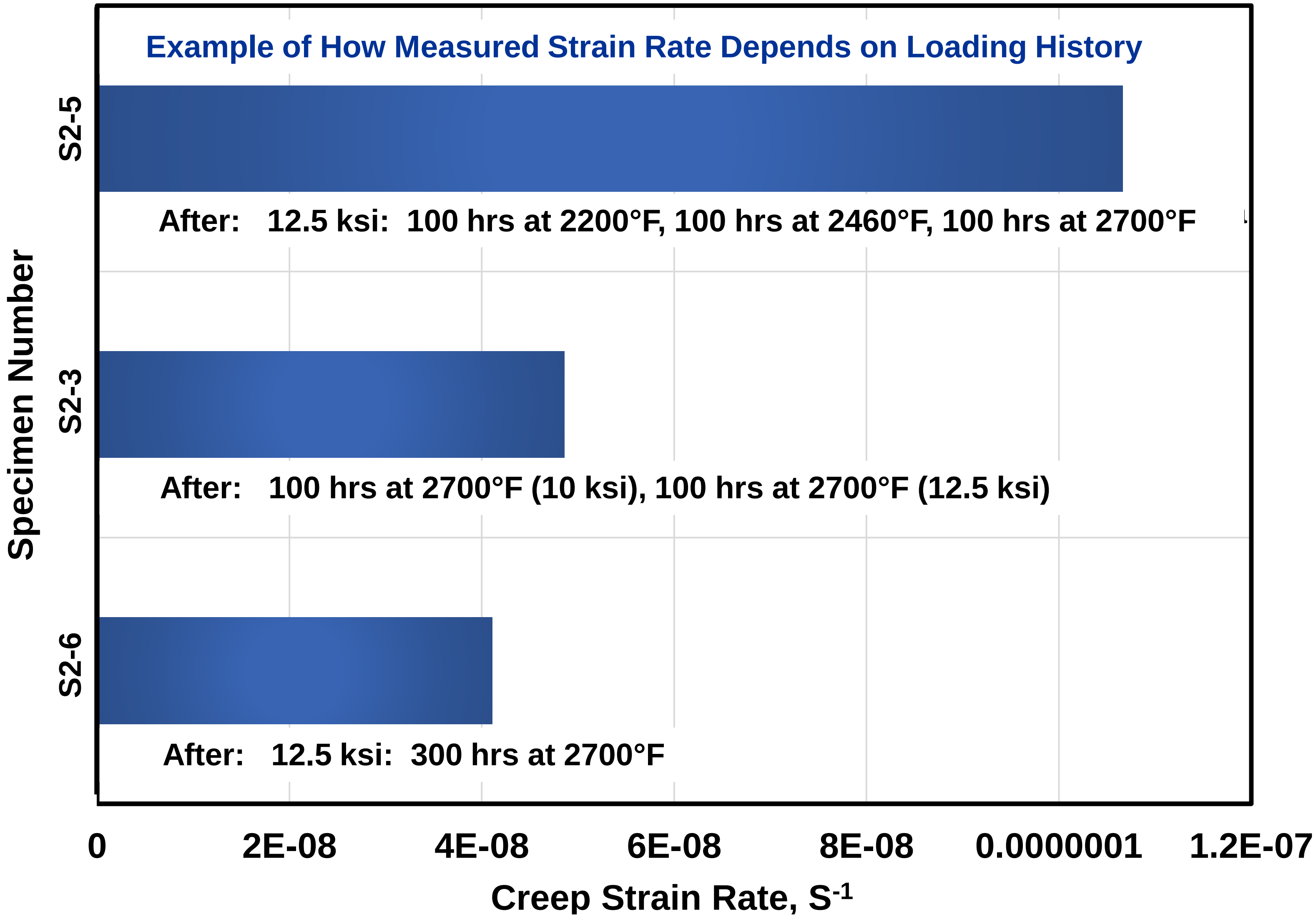
Slope = $(-Q)/R$
R= Gas Constant= 8.314 J/K.mol
Q= Activation Energy= 107 KJ/mol
at 100 hr, 86.2 MPa

y = -12849x - 8.8175
$R^2 = 0.9681$

- Activation Energy is dependent on when strain rate is measured (hrs of creep completed)

- Contribution from Primary Creep leads to low Activation Energy

CMC Creep Dependence on Mechanical and Thermal Loading Histories



2D CVI SiC/SiC Reinforced with Sylramic^{TM}-iBN:

Creep in Air— *5 Different Conditions, and* RT FF of As-Received

Specimen ID	Test Condition (Temperature: °F, Stress: ksi, Time: hrs)	RT Fast Fracture Residual UTS (ksi, MPa)	RT Fast Fracture Ultimate Strain (%)
1520-S2-1	2700°F, 10 ksi for 100 hrs	49.5, 341	0.26
1520-S2-2	2700°F, SPLCF, R=0.5, 5 / 10 ksi for 100 hrs	51.6, 356	Ext. Moved
1520-S2-3	2700°F, 10 ksi for 100 hrs, 12.5 ksi for 100 hrs, 15 ksi for 100 hrs	45.1, 311	0.31
1520-S2-4	RT Tensile test	49.5, 341	0.33
1520-S2-5	2200°F, 12.5 ksi for 100 hrs, 2460°F, 12.5 ksi for 100 hrs, 2700°F, 12.5 ksi for 100 hrs	Broke upon cooling	Not tested at RT
1520-S2-6	2700°F, 12.5 ksi for 300 hrs	47.1, 325	0.31

Work Remaining / Future Work

- Fractography and microstructural characterization.
- Analyze AE (acoustic emission) and resistivity data.
- Analyze hysteresis testing data.
- Compare fiber loadings: Study 1 (previous) and Study 2 (current) materials.
- Review SiC/SiC activation energy data in open literature.
- Examine crack spacing in gage section of tested samples.
- Examine data collected when specimens were held at T following the creep testing to see how much strain recovery occurred.
- ***Prepare updated presentation (A. Almansour presenting at Pac Rim Conf. in 2017).***
- Consider obtaining another panel of CVI SiC/SiC and conduct testing at 2700°F / 3 stresses and 12.5 ksi / 3 temperatures. Test minimum 2 samples per condition. Use selected post-test analysis techniques.

Summary and Conclusions

- CVI SiC/SiC CMCs incorporating Sylramic™-iBN SiC fiber are being evaluated via tensile creep testing to determine creep parameters for modeling.

- A stress exponent was determined at 2700°F, and an activation energy was calculated.

- As reported previously (Shinavski et al[3]), the activation energy measured depends on the time/strain at which strain rates are measured, and on loading history.

- All creep specimens achieved a run-out condition. Fractography conducted on those samples following RT FF residual strength measurement will help determine whether or not any samples cracked during creep testing.

- We are investigating various approaches to analyzing specimens following creep testing such as AE and resistivity to help us understand CMC damage mechanisms.

References

1. D. Kiser, J. DiCarlo, L. Evans, R. Bhatt, R. Phillips, and T. McCue, “Evaluation of CVI SiC/SiC Composites for High Temperature Turbine Engine Applications,” Proceedings of the 39th Annual Conf. on Composites, Materials, and Structures, Cape Canaveral, FL, January 2015.
2. J. DiCarlo, “Modeling Creep of SiC Fibers and Its Effects on High-Temperature SiC/SiC CMC,” Proceedings of the 38th Annual Conf. on Composites, Materials, and Structures, Cape Canaveral, FL, January 2014.
3. R. Shinavski, S. Harris, and W. Thibault, “Creep Response of SiC/SiC Composites at 2700-3000°F,” Proceedings of the 39th Annual Conf. on Composites, Materials, and Structures, Cape Canaveral, FL, January 2015.
4. R. T. Bhatt, “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,” Proceedings of the 41st Annual Conf. on Composites, Materials, and Structures, Cape Canaveral, FL, January 2017.
5. A. Almansour and G. Morscher, “Modeling of Different Fiber Type and Content SiC_f/SiC Minicomposites Creep Behavior,” presented at the 41st International Conference and Expo on Advanced Ceramics and Composites, Daytona Beach, FL, January 2017.