



# High Temperature Lightweight Self-Healing Ceramic Composites for Aircraft Engine Applications

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## Acknowledgements

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# Introduction

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- **Advanced aircraft engines require the use of reliable, lightweight, creep-resistant and environmentally durable materials.**
- **Silicon carbide-based ceramic matrix composite (CMC) technology is being developed to replace nickel-based superalloy blades and vanes.**
  - **Near term 1589 K (2400 °F) (cooled).**
  - **Medium term 1755 K (2700 °F) (cooled).**
  - **Long term 1922 K (3000 °F) (uncooled).**



# Rule of Mixtures (ROM) Composite Theory

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$$P = P_{\text{fiber}} V_{\text{fiber}} + P_{\text{matrix}} V_{\text{matrix}}$$

$$V_{\text{fiber}} + V_{\text{matrix}} = 1$$

$P_i$  = Property of the  $i^{\text{th}}$  component (e.g. strength).

$V_i$  = Volume fraction of the  $i^{\text{th}}$  component .

- **Properties of the composite are determined by the properties of the fiber and the matrix and their relative volume fractions.**



# Objectives

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- **Develop a new class of ceramic composites –  
**Engineered Matrix Ceramics (EMCs).****
  - **Design different engineered matrices.**
  - **Demonstrate thermal strain compatibility with SiC.**
  - **Evaluate oxidation and mechanical properties.**
- **Fabricate engineered matrix composites.**
  - **Evaluate self-healing properties.**



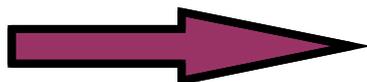
# Current SiC/SiC CMC Fabrication Processes

(courtesy R. Bhatt)

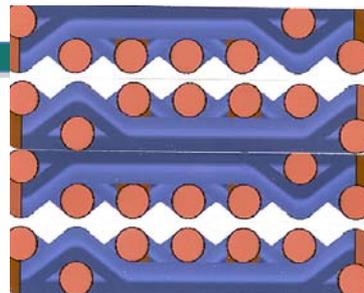
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SiC Fiber



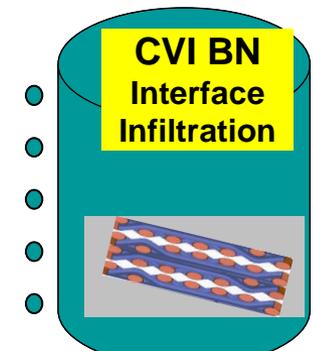
0/90 Fabric Weaving



Preform

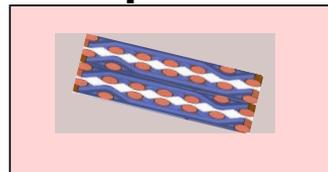


Reactor



Reactor

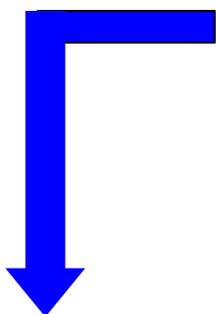
Preform Compression



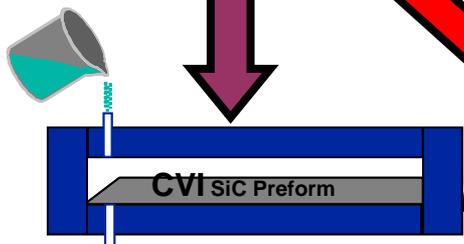
PIP

PIP or CVI-PIP CMC

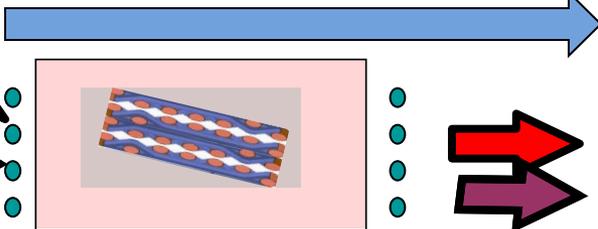
CVI-MI CMC



Full CVI CMC



Slurry Infiltration



Si Melt Infiltration



# Current Generation of CMCs: Matrix Microstructure

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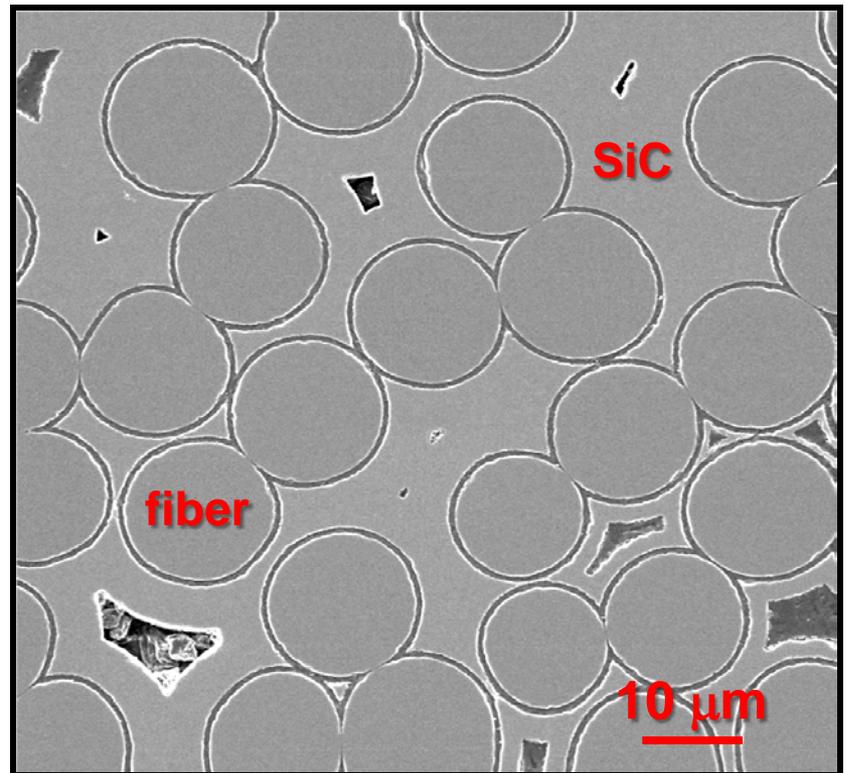
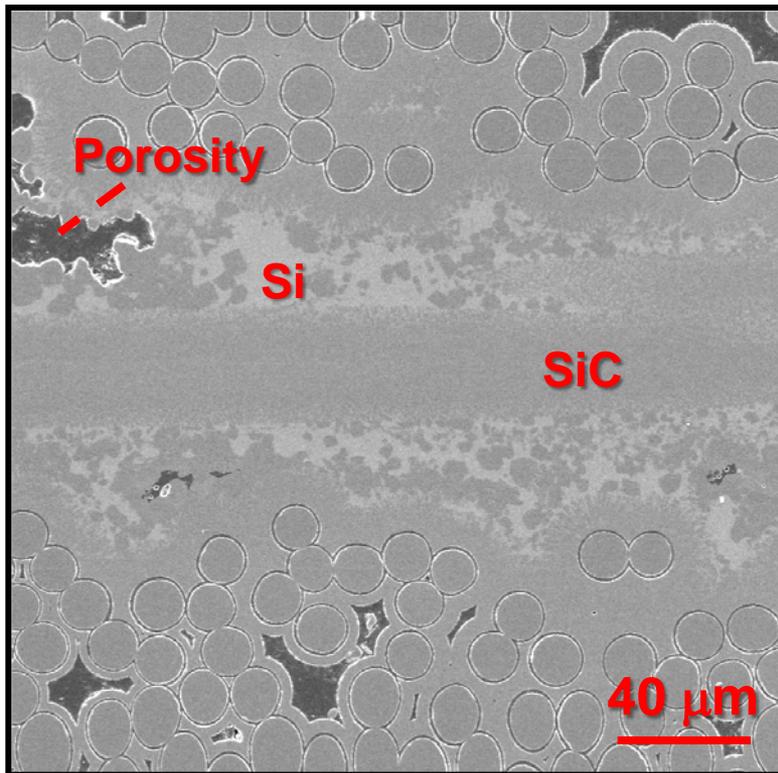
- **Silicon carbide (SiC).**
- **Unreacted or free carbon and silicon.**
- **Porosity:**
  - ~10-25 vol.% for chemical vapor infiltration (CVI).
  - ~10-25 vol.% for polymer infiltration and pyrolysis (PIP).
  - ~3-10 vol.% for Melt Infiltration (MI).



# Typical Microstructure of As-Processed BN-Coated Hi-Nicalon MI SiC Composites

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(Courtesy M. Singh)

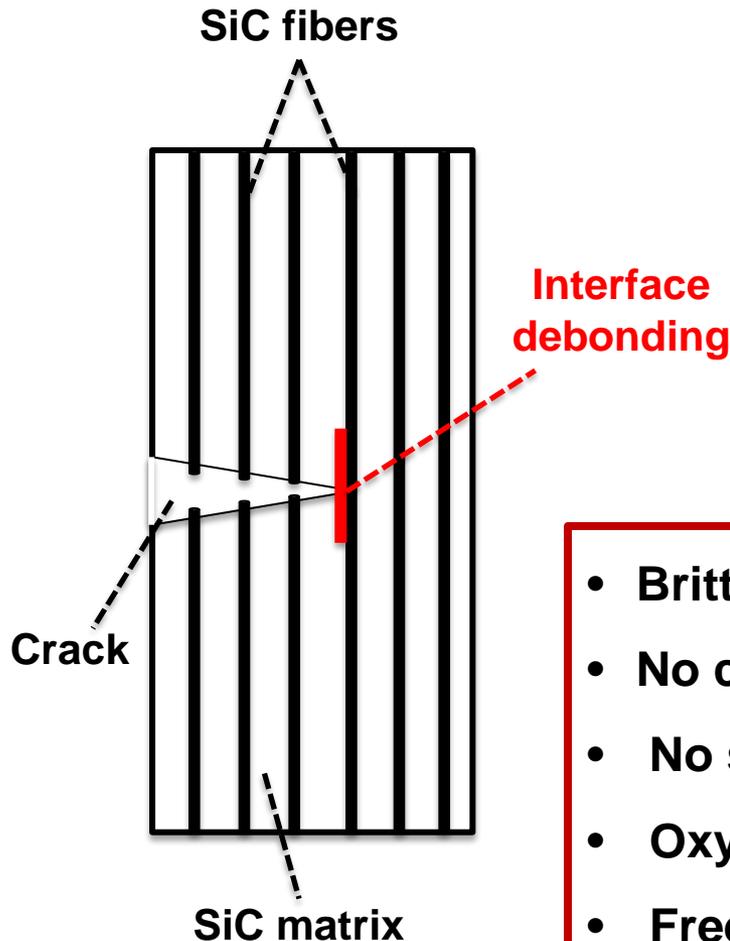


Density ~ 96-97 %



# Current SiC/SiC CMC Matrix Capabilities

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- Low matrix cracking strength (proportional limit - 69 MPa/10 ksi)
- Matrix fills space and provides a thermally conductive path.
- Fracture toughness due to crack bridging and interface debonding.

- Brittle at all temperatures.
- No crack tip blunting – fast crack propagation.
- No self-healing.
- Oxygen ingress to fibers shortens fiber life.
- Free Si in the matrix limits temperature usage to below 1588 K (2400 °F).

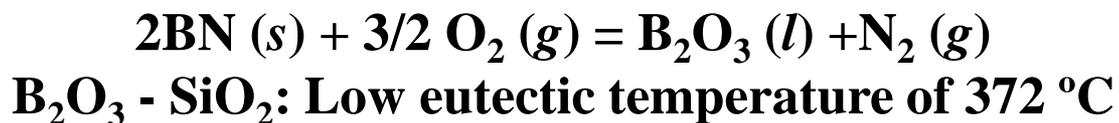
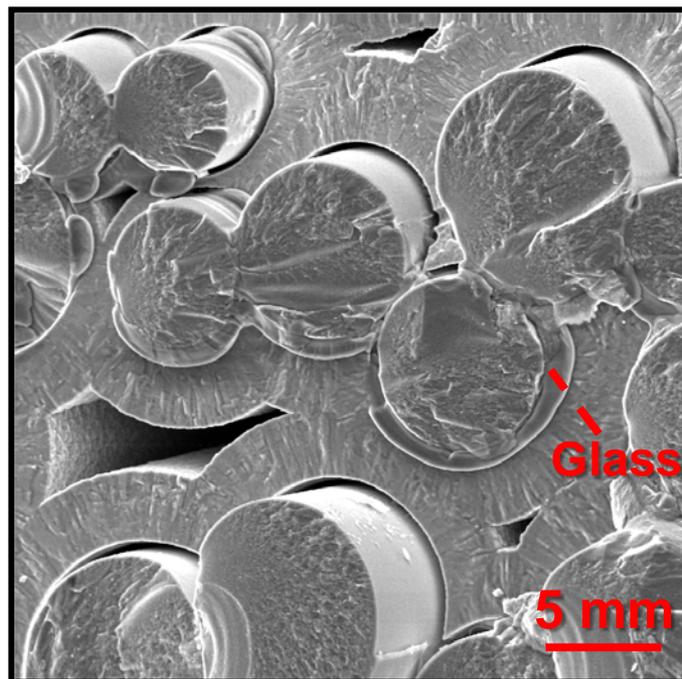
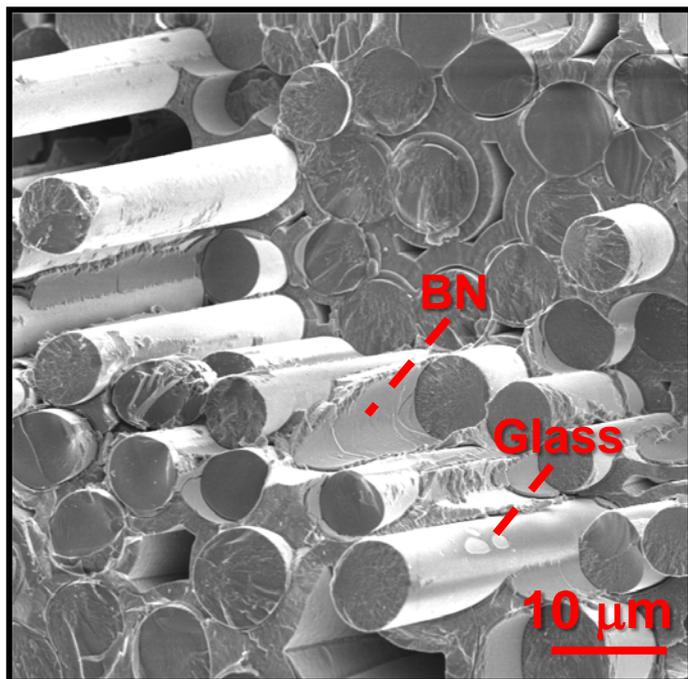


# Recession of BN and Formation of Glassy Phase in BN-Coated Hi-Nicalon MI SiC Composites

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(Courtesy M. Singh)

**T = 973 K;  $\sigma$  = 250 MPa; 1000 h in air**





# Important Question

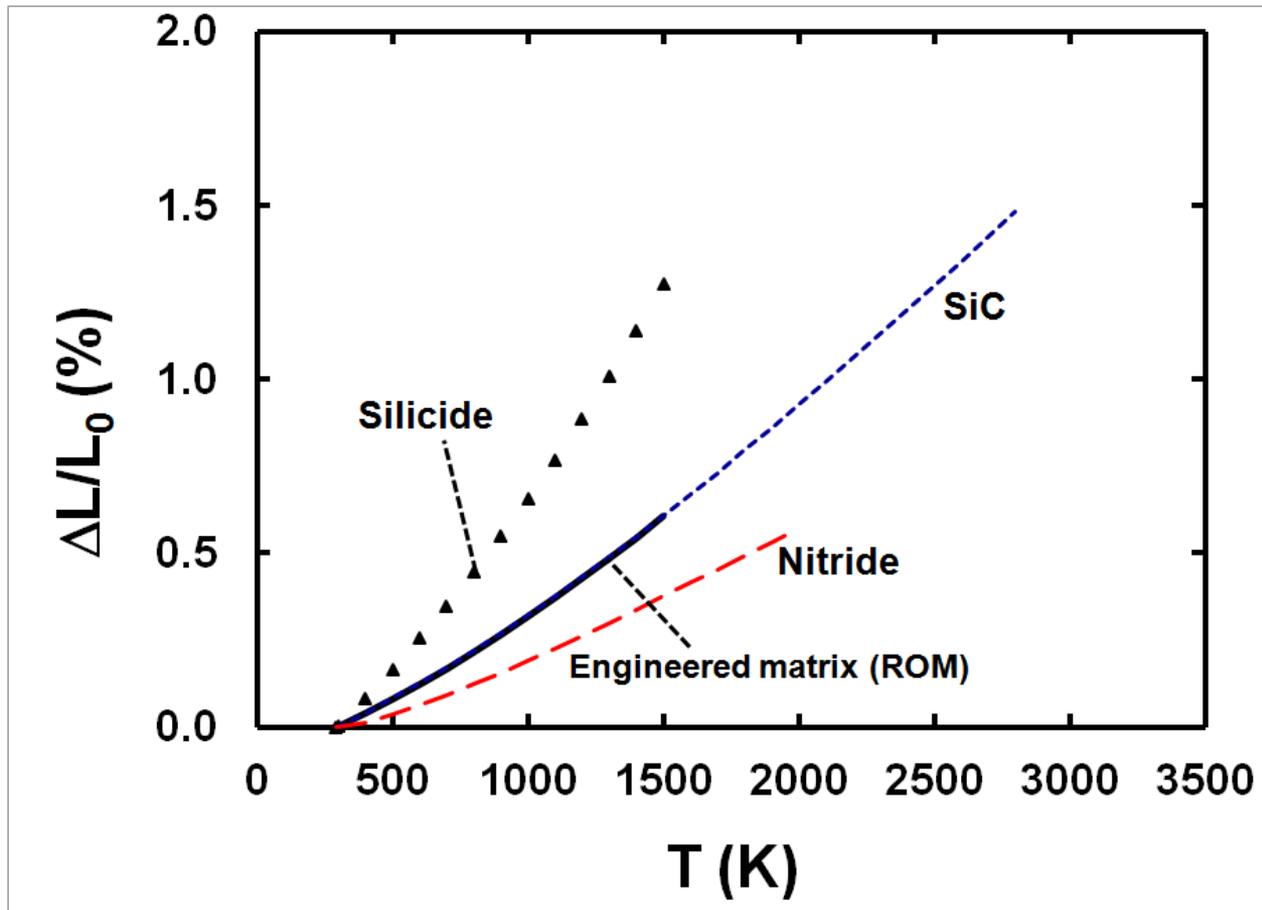
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**Can the matrix properties be suitably engineered to ensure certain desirable characteristics?**



# Present Concept

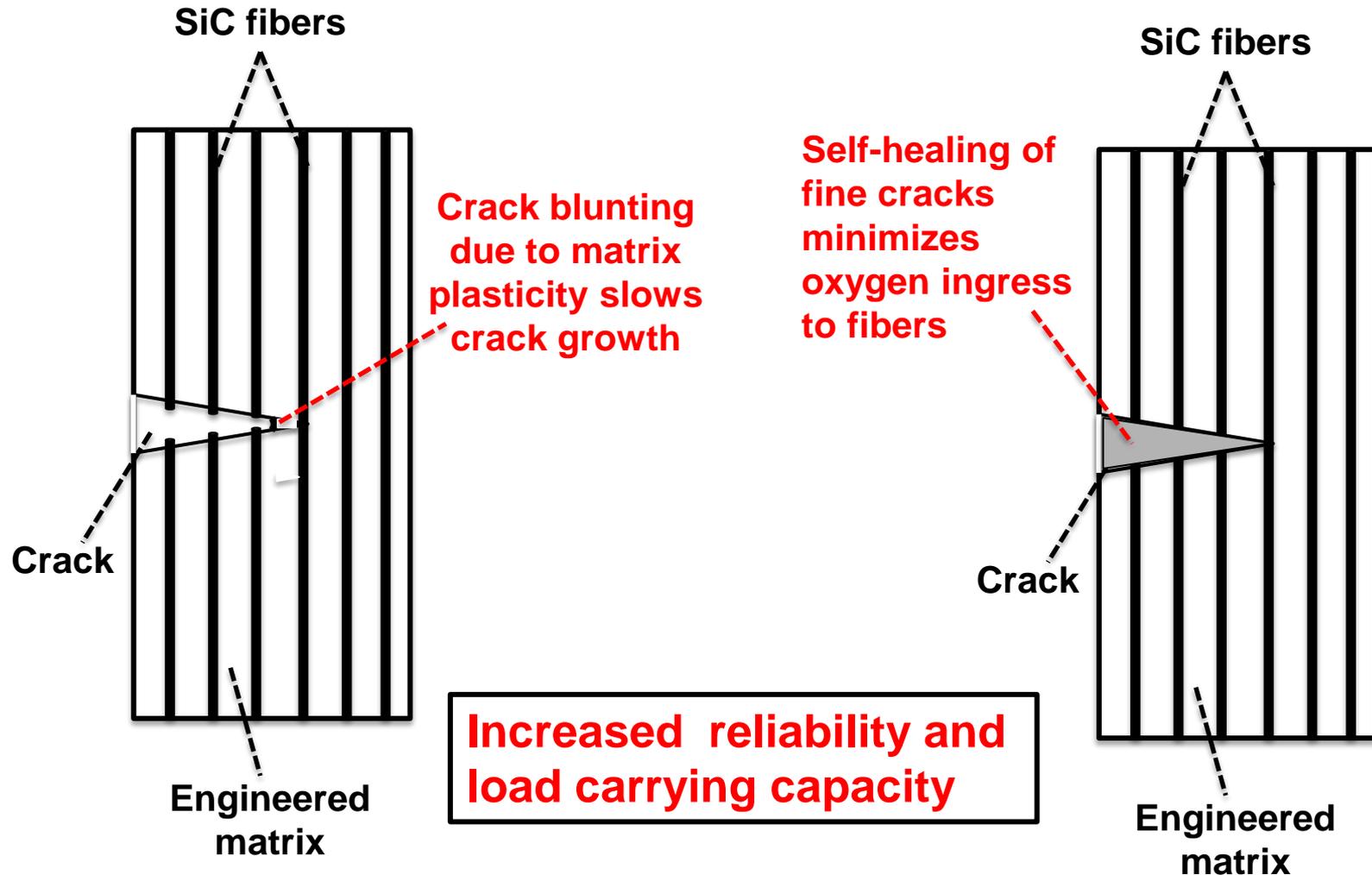
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# Crack Tip Blunting and Self-Healing

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# **Innovation: Desired Characteristics of the Engineered Matrix (EM)**

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- ❖ **Thermal strain compatibility of the matrix with the SiC fibers.**
- ❖ **Plastically compliant matrix to blunt cracks.**
- ❖ **Self-healing crack capabilities to minimize ingress of oxygen.**
- ❖ **Minimize the volume fraction of unreacted silicon to prevent corrosive attack of fibers and incipient melting.**
- ❖ **Dense matrix to increase thermal conductivity.**



# Expected Impact of Innovation

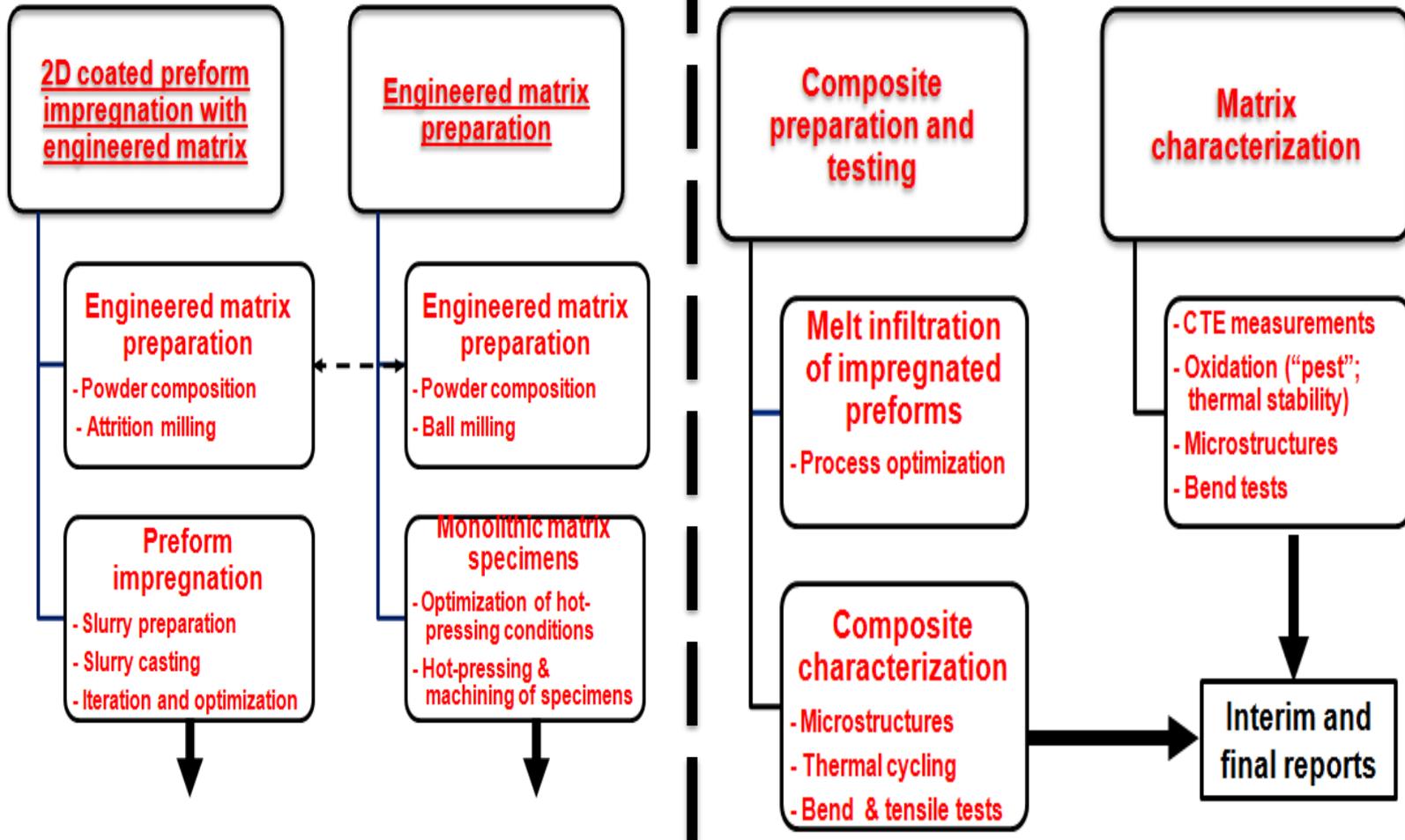
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- **Matrix plasticity** - increased reliability, compliant matrix.
- **Self-healing matrix** - prevents or minimizes oxygen ingress.
- **Low free Si** - reduces fiber attack, reduces incipient melting, increased high temperature capability.
- **Dense matrix** - High thermal conductivity.



# Fabrication and Testing of Engineered Matrix Composites (EMC)

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# Hot-Pressed Plate and Optical Micrograph

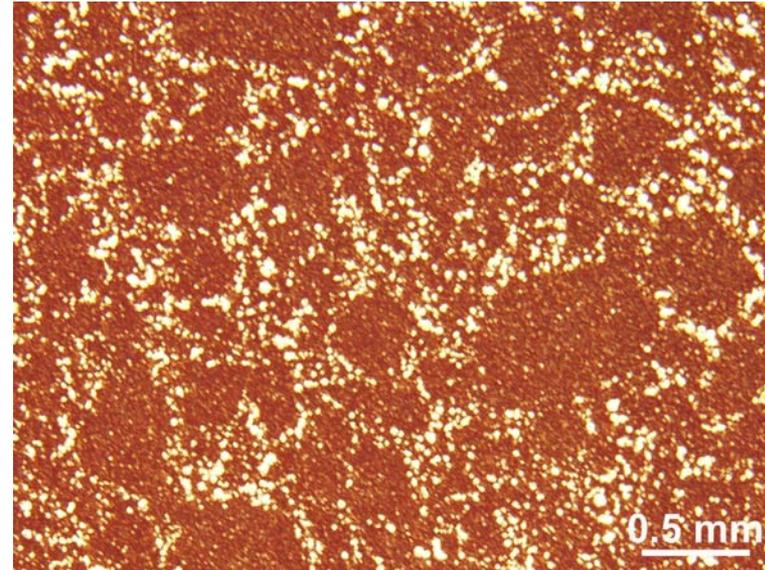
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## CrMoSi-EM

50 x 50 x 4 mm



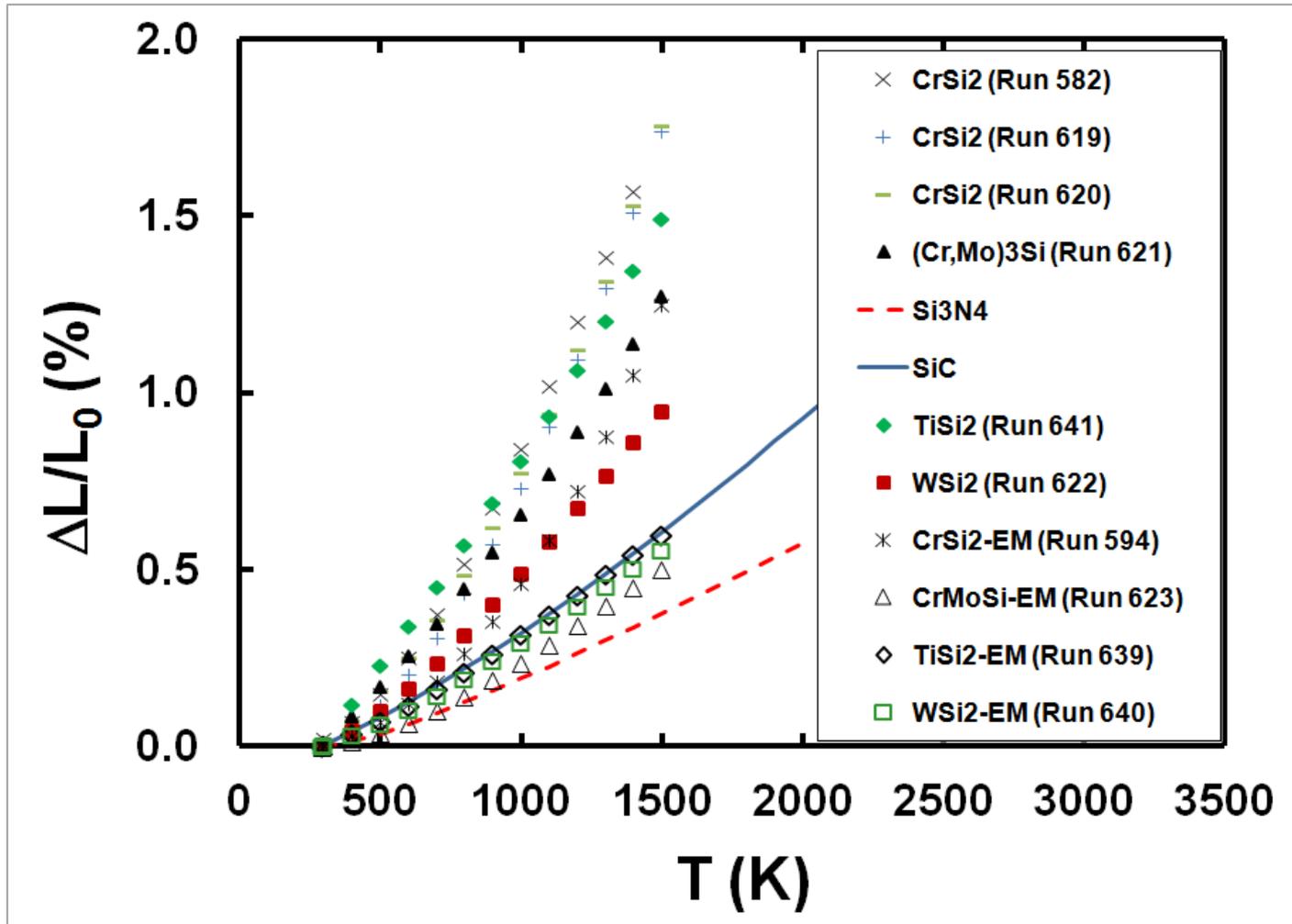
Optical micrograph





# Proof-of-Concept: Thermal Strains

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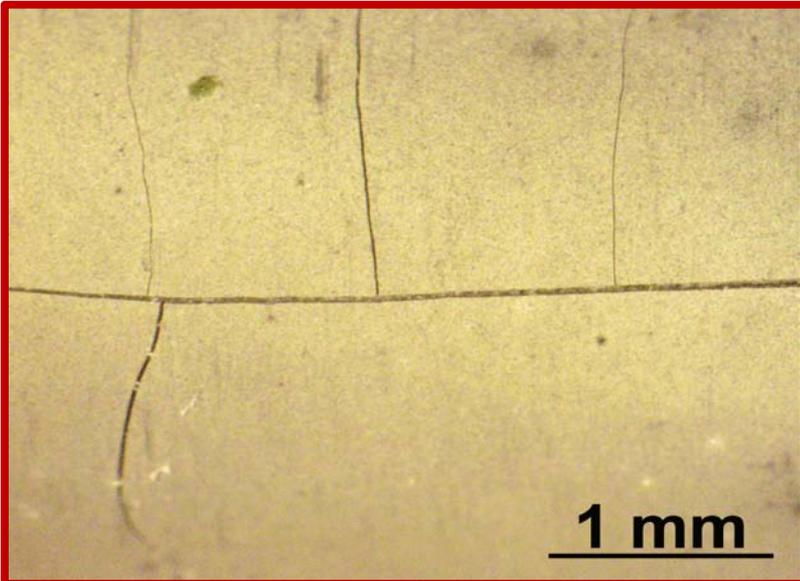


# Optical Macrographs of MoSi<sub>2</sub> Engineered Matrix

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Thermally cycled between room temperature and 1500 K (2240 °F) three times.

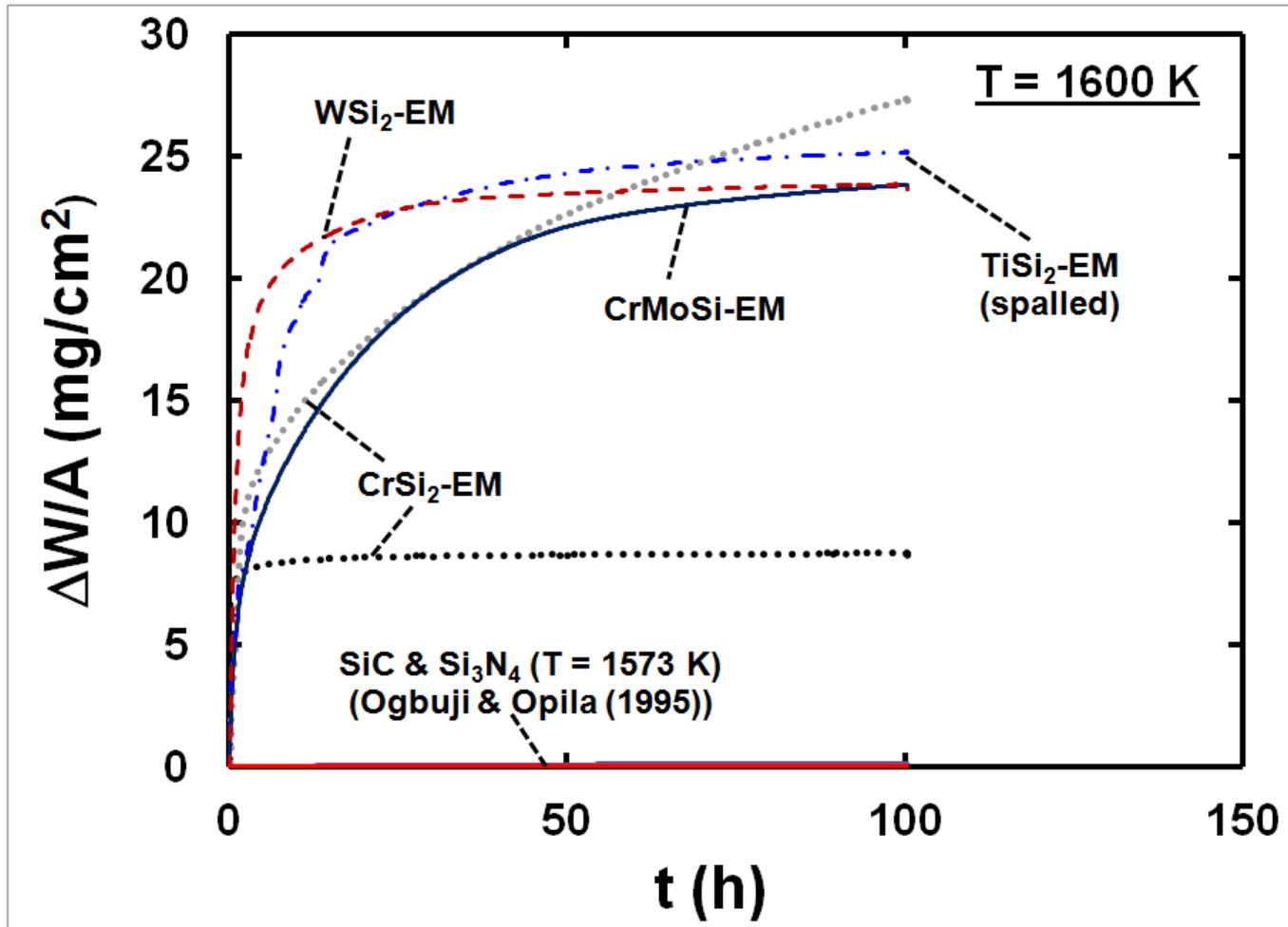


- Thermal cycling resulted in cracking.
- No longer considered in the program.



# Isothermal Oxidation Behavior

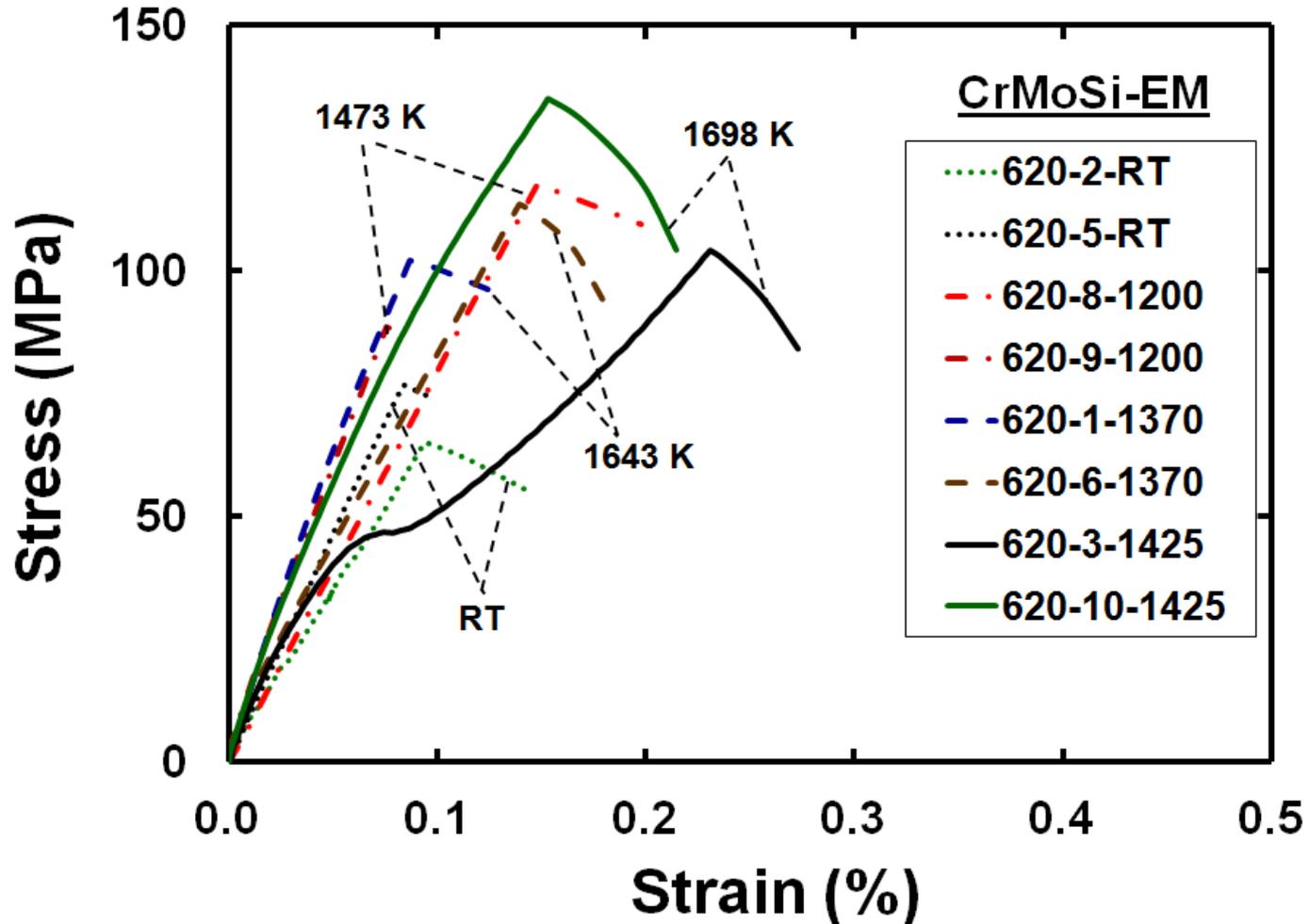
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# Bend Stress-Strain Curves for CrMoSi-EM

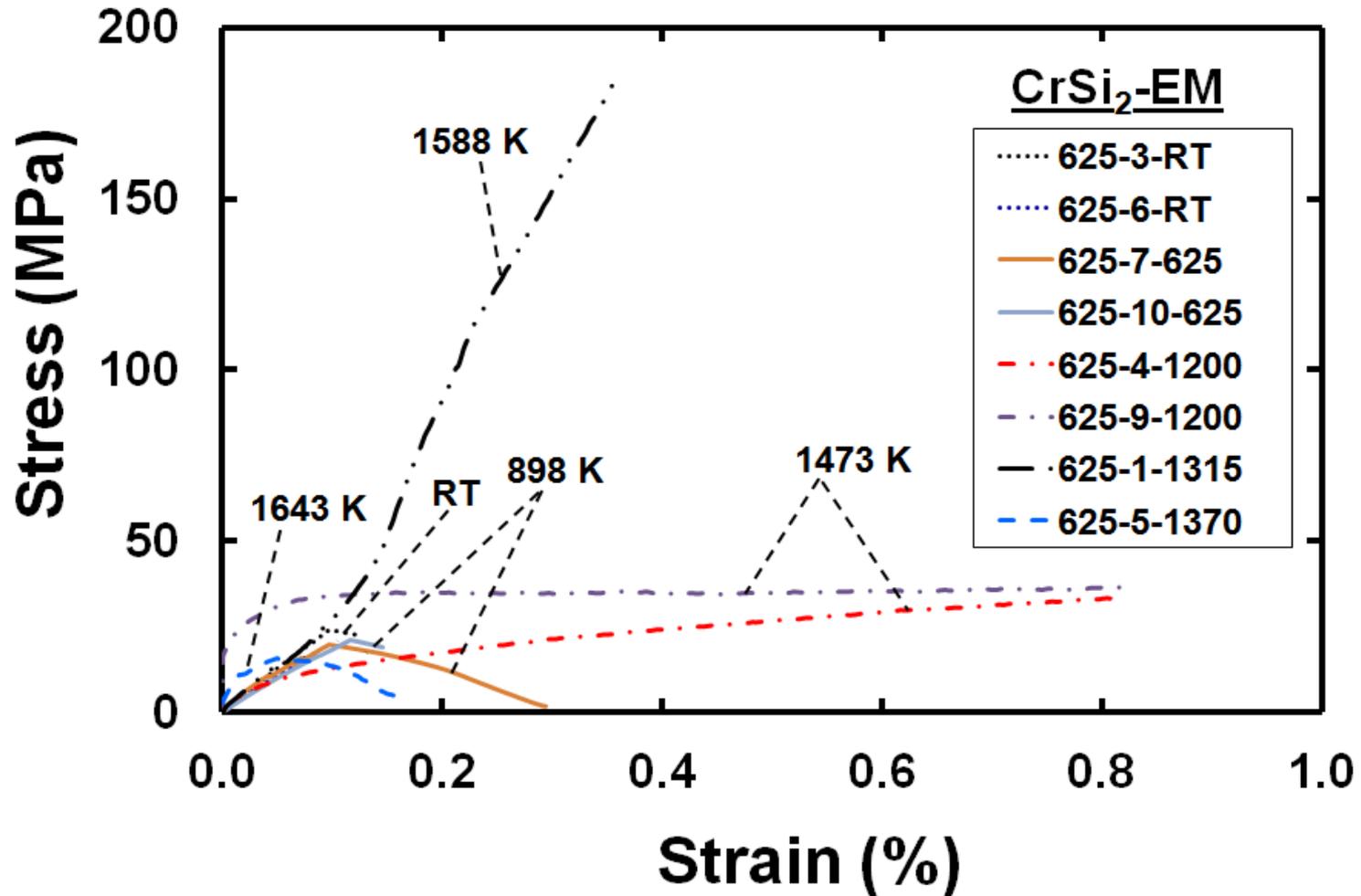
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# Bend Stress-Strain Curves for CrSi<sub>2</sub>-EM

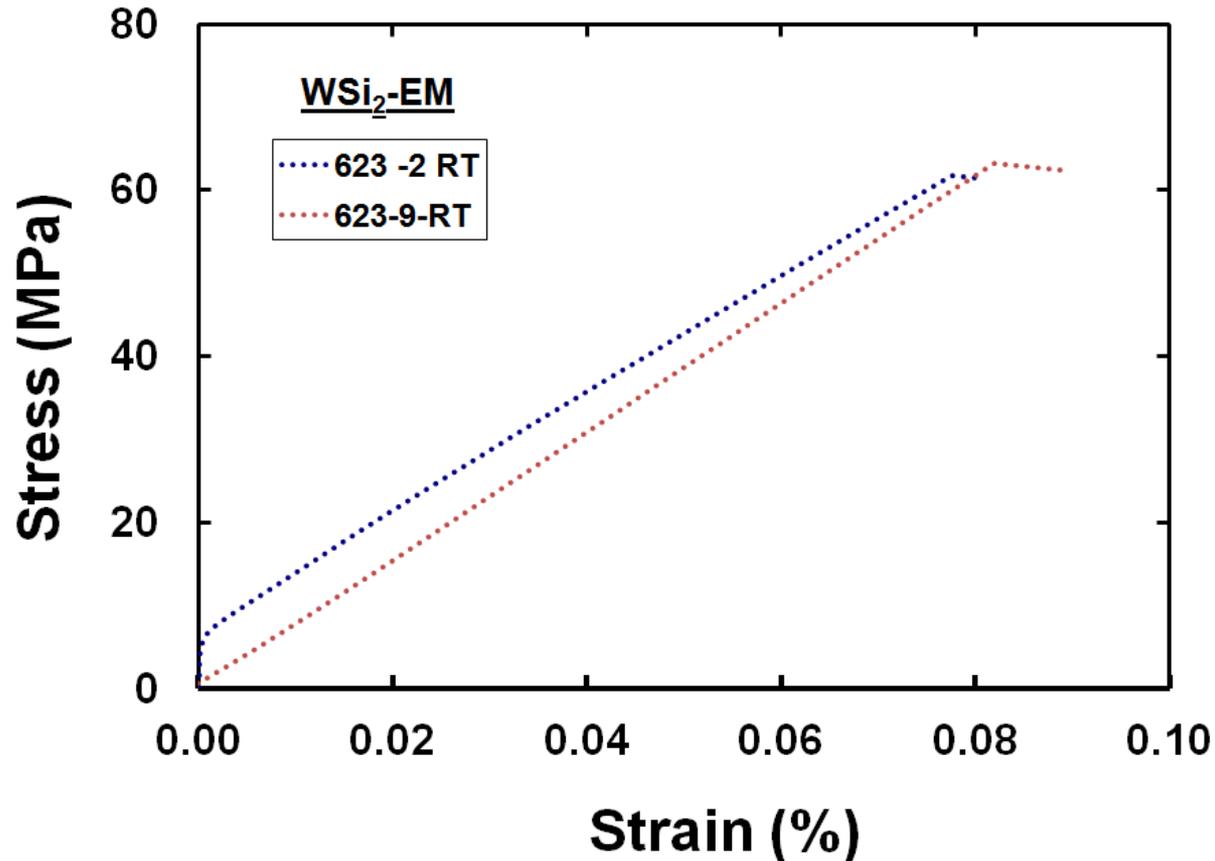
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# Bend Stress-Strain Curves for WSi<sub>2</sub>-EM

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**Catastrophic oxidation occurred during heat-up to 1473 K.**



# Engineered Matrix Composites Fabrication

(courtesy R. Bhatt)

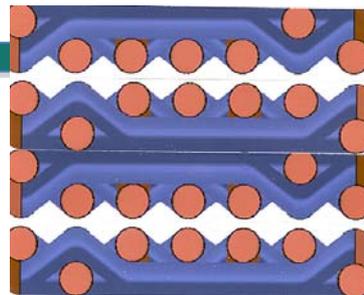
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SiC Fiber



0/90 Fabric Weaving

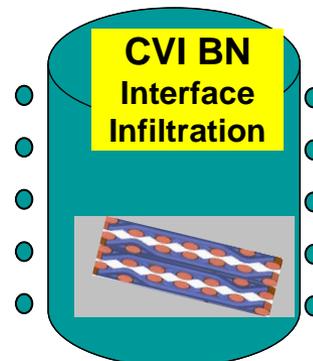
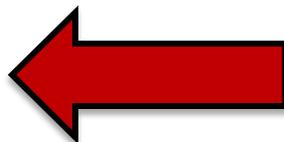


SiC fiber Preform Fabrication



CVI SiC Matrix Infiltration

Reactor

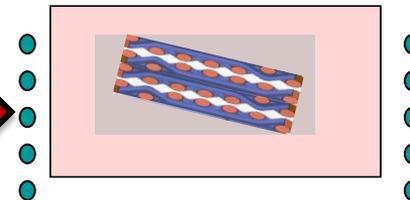
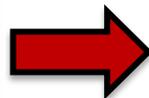
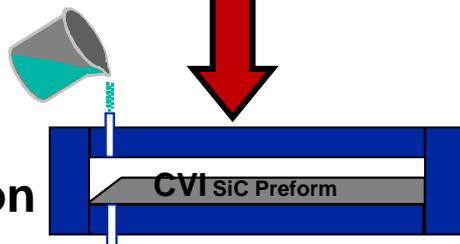


CVI BN Interface Infiltration

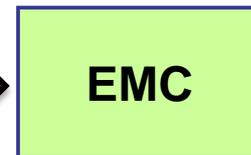
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Fiber coating

Engineered Matrix Infiltration



Melt Infiltration

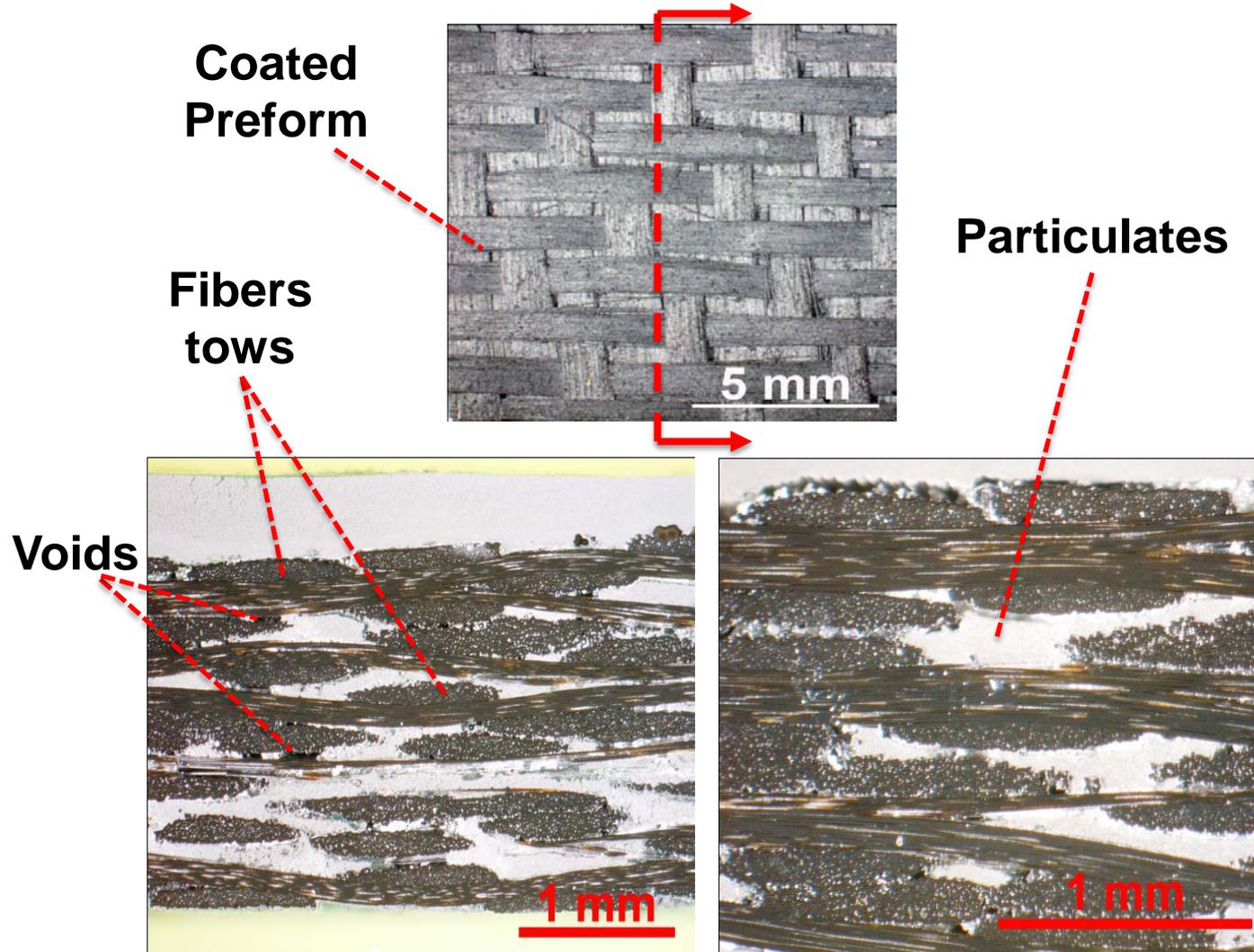


EMC



# Microstructures of Particulate-Infiltrated SiC Fiber Preform

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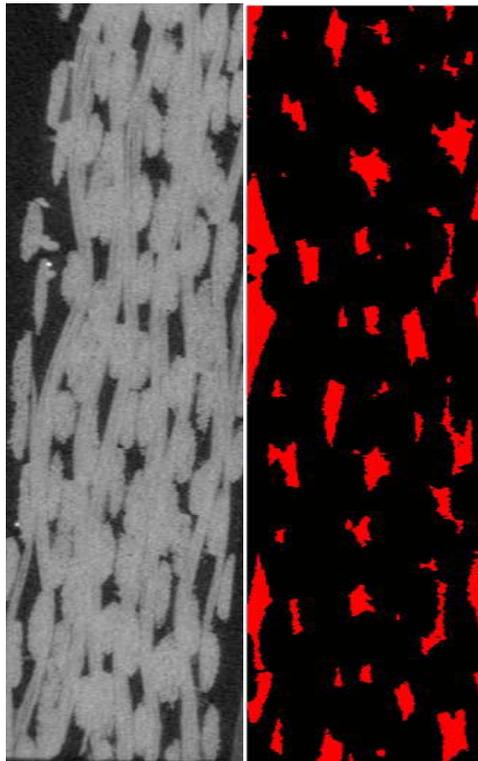


# CT Scans of Particulate Infiltrated Preform

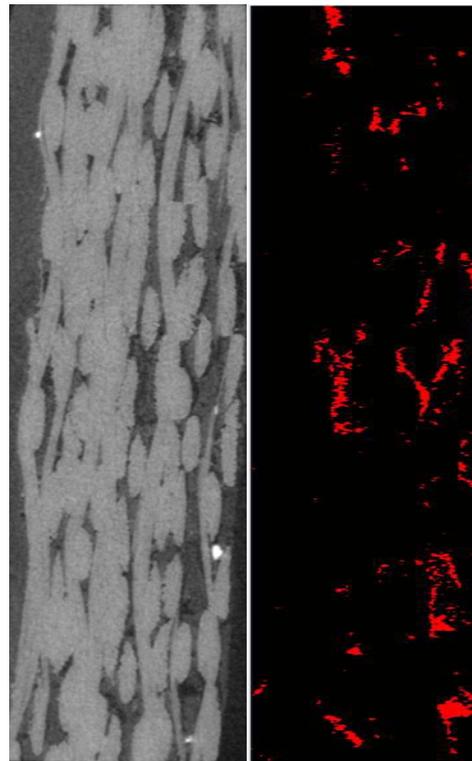
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As-received Preform

Particulate Infiltrated



Area fraction of porosity ~ 21-23%



Area fraction of porosity ~ 0.9%

The red regions are voids



# Summary and Conclusions

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- **Bend, CTE, isothermal oxidation and thermal cycling tests were conducted on several engineered silicide/SiC/Si<sub>3</sub>N<sub>4</sub> matrices.**
- **Two promising engineered matrix compositions were down-selected for further development.**
- **Trials to infiltrate one of these engineered matrices into SiC-coated fiber preforms have been completed. Microstructural analysis and CT scans demonstrated almost complete infiltration of the preform.**
- **Efforts are underway to produce Engineered Matrix Composites (EMCs) specimens to determine self-healing capabilities.**



# Summary of Phase I Accomplishments: TRL 2

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Milestone	Status
<b>Demonstrate thermal strains for engineered matrices match those of SiC.</b>	<b>Completed.</b>
<b>Generation of matrix properties and down-selection of promising compositions.</b>	<b>Completed.</b>
<b>Demonstrate high temperature matrix plasticity.</b>	<b>Completed.</b>
<b>Develop processing techniques for fabricating EMCs.</b>	<b>Particulate infiltration trials completed; melt infiltration trials to be completed.</b>
<b>Evaluate mechanical properties of EMCs and demonstrate self-healing capabilities.</b>	<b>To be completed.</b>



# Next Steps

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- **Complete tensile tests of monolithic specimens.**
- **Particulate and melt infiltrate bend specimens.**
- **Mechanical testing of EMC bend specimens.**
- **Evaluate self-healing properties in EMCs.**
- **Write and submit final report.**
- **Submit Phase II proposal.**



# Distribution and Dissemination

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- **Submitted NF 1679 to GRC patent attorney (LEW-18964-1).**
- **Submit papers to journals/NASA TMs after receiving approvals.**
- **Present papers at conferences after receiving approvals depending on the availability of travel funds.**