Additive Manufacturing of SiC-Based Ceramics and Ceramic Matrix Composites

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Outline

- **Objective and Benefits**
- **NASA GRC Efforts:**
  - Laminated Object Manufacturing (OAI)
  - NASA NARI Seedling Project: Non-Metallic Turbine Engine
  - 3-D Printing
    - Wood Containing Filament for Preforms
    - Powder Loaded Filament
    - Extrusion Printing of Pastes

Objective and Benefits of Additive Manufacturing Technologies

Objective: Utilize additive manufacturing technologies as alternative processing approaches for fabricating advanced ceramics and CMC components

Benefits:

- Ease of Fabrication and Manufacturing

- Simplified formation of Silicon Carbide-based matrix materials.
- Custom-made and complex geometries are possible which were previously limited by traditional CMC processing methods.
- Complex shapes involving the formation of curvatures and sharp part transitions can be fabricated.

- Tailorable Composition and Properties

- Hybrid composites can be fabricated by the manipulation of ceramic fiber preforms. Manual layer by layer assembly is time consuming and expensive.
- Fabrication of composites with multifunctional properties.

- Lower Cost

- Reduced cost through fewer processing steps and short production time from utilization of additive manufacturing.

Additive Manufacturing of CMCs

Conventional Manufacturing

- Customized parts in small volumes are time consuming and expensive to produce.
- Complex shape fabrication issues: mold design, dimensional tolerances, etc..
- Manufacturing of multifunctional parts are challenging.

Efforts in the last >30 years have now resulted in commercialized turbine engine applications.

Additive Manufacturing

- Small series of ceramic parts can be manufactured rapidly and cost-effectively.
- Specific molds are not required.
- Different designs can be optimized (no major cost of changes)
- Parts with significant geometric complexity.

Efforts in this very promising field are just now underway.

Material and Process Challenges

- Property and behavior of starting materials
- Sintering and densification challenges
- Process modeling
- Mechanical behavior
- NDE and in-situ damage characterization
- Material and property databases

Materials and processing challenges are quite similar

Largest barrier to CMC insertion has been high acquisition cost

For AM, the starting materials are very low cost (powders and fibers).

Overview of Additive Manufacturing Technologies

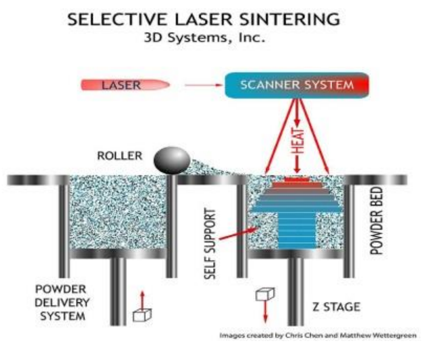
(many variants and combinations)

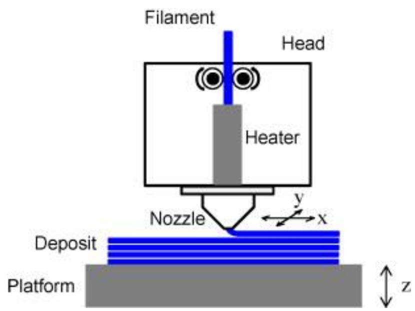
Selective Laser Sintering

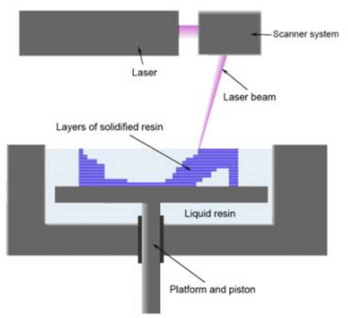
High powered laser fuses plastic, metal, or ceramic powders by moving along cross-sections repeating the process upon the addition of powder.

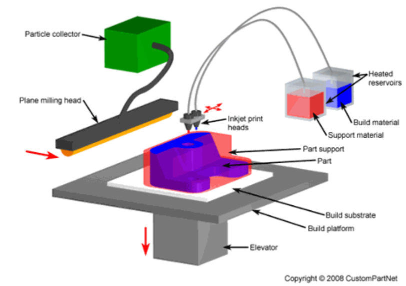
Stereolithography

A beam of ultraviolet light is directed onto a vat filled with a liquid ultraviolet curable photopolymer and moves along cross-sections of the object.









Fused Deposition Modeling

Plastic or metal is heated and supplied through an extrusion nozzle and deposited in a path determined by a CAD model.

3D printing

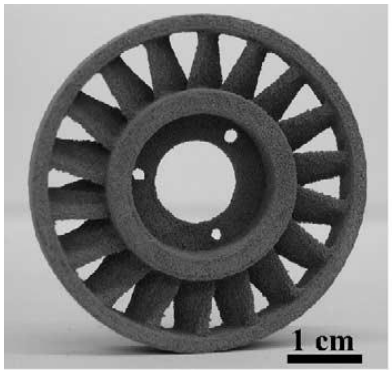
An inkjet-like printing head moves across a bed of powder and deposits a liquid binding material in the shape of the object's cross section

Material choices are limited by the machine's manufacturers
Fabrication of continuous fiber composites is not possible

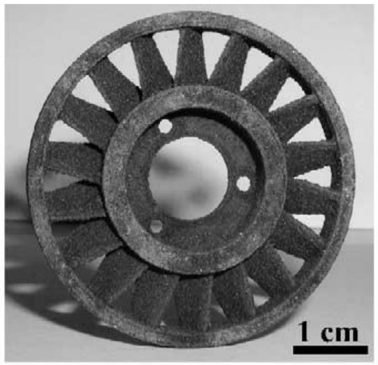
Selective Laser Curing (SLC) of Preceramic Polymers and 3D-Printing of SiSiC Ceramics

Starting Material: 50 vol.% Polysiloxane / 50 vol.% SiC

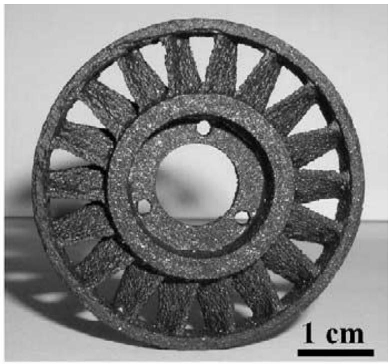
Polysiloxane + SiC **SiSiC**



SLC



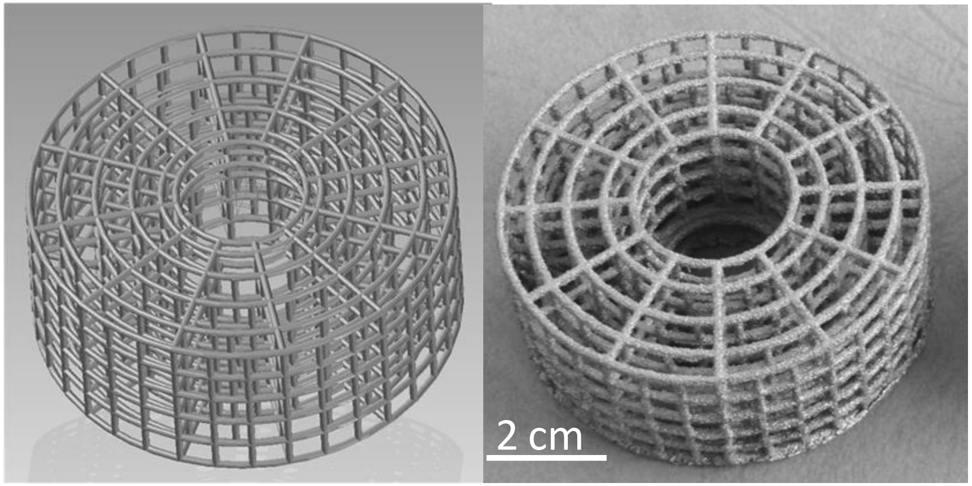
Pyrolyzed at 1200 °C



Infiltrated with Si

No Fiber Reinforcements in SLC and 3D Printing Process

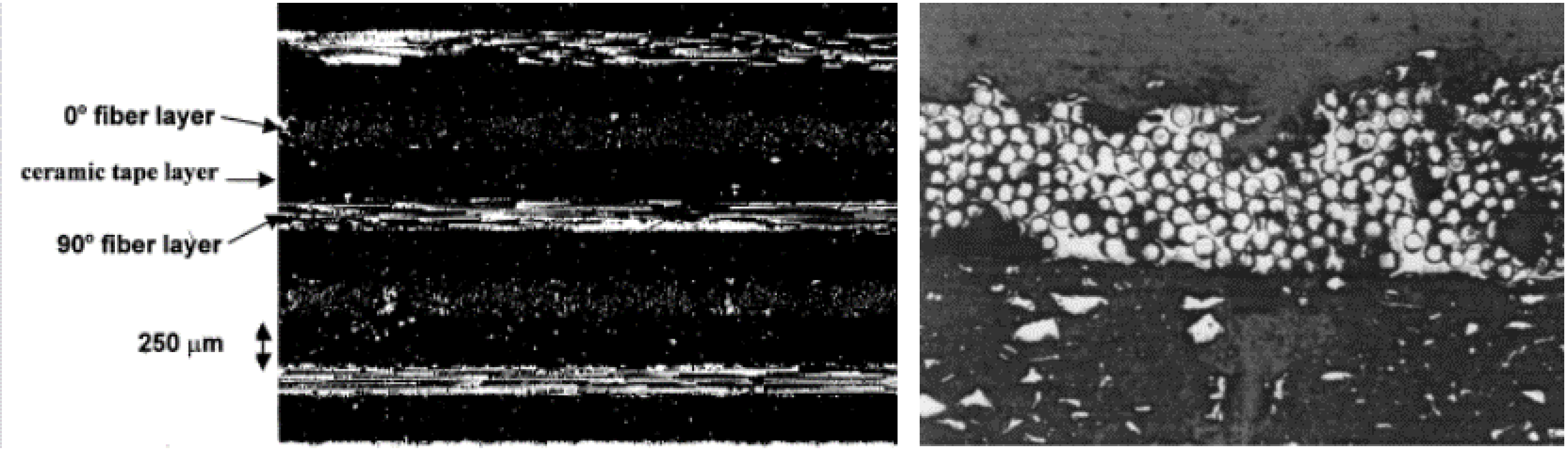
T. Friedel, et al, J. Eur. Ceramic Soc., 25, 2005, 193-197



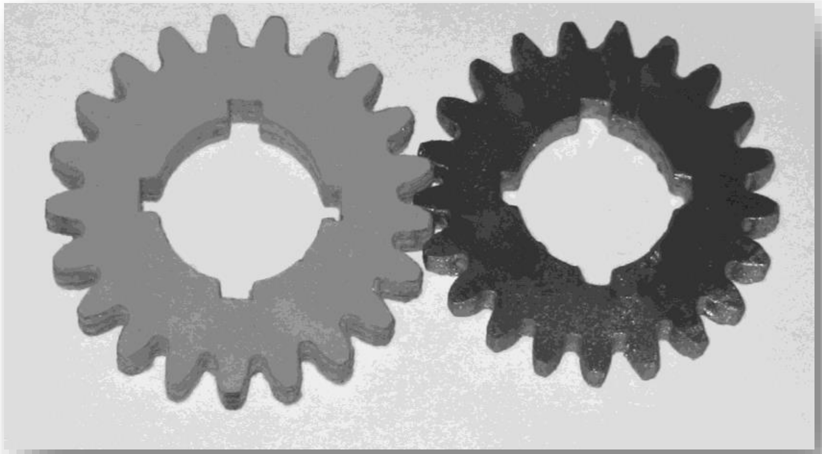
CAD design of macro-cellular lattice reactor structure (left) and SiSiC component fabricated by 3D printing (right)

L. Schlier, et al, Int. J. Appl. Ceram. Techn., 8 [5], 2011, 1237-1245

Laminated Object Manufacturing of SiC-Based Composites



Cross section of reaction bonded SiC/SiC composite showing alternating prepreg and ceramic tape layers. Fibers are carbon-coated CG-Nicalon SiC.



Donald Klosterman, et al, Composites Part A, 29A (1998) 1165–1174

Gear wheel (diameter 50 mm) manufactured from SiC-filled preceramic paper

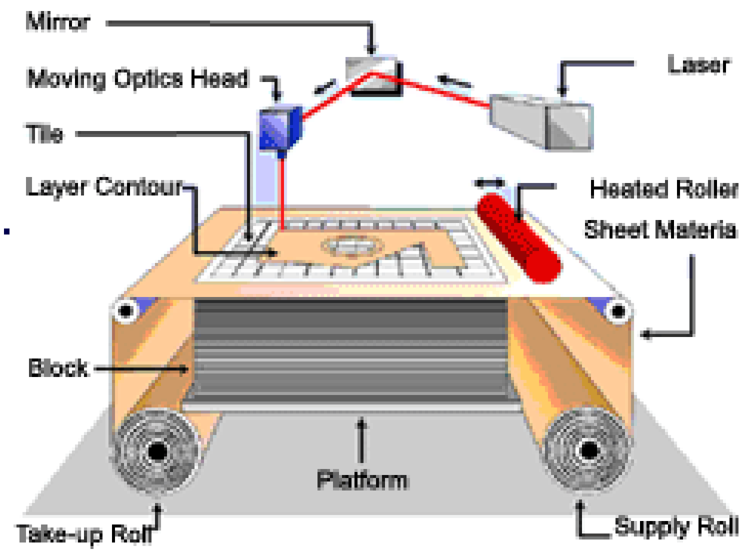
N. Travitzky, et al, J. Am. Ceram. Soc., 91 [11], 2008, 3477–3492.

Laminated Object Manufacturing of Ceramic Matrix Composites

- **LOM is a viable option for manufacturing fiber reinforced CMCs with modification to the machine.**
- **Issues with LOM machines manufacturing base.**

Typical Process:

1. CAD design is turned into computer generated cross sections.
2. Layers of adhesive coated materials adhered to substrate with heated roller.
3. Laser cuts cross-section of part.
4. Laser cross hatches non-part area.
5. Platform with completed layer moves down.
6. Fresh sheet moves over and platform moves up. Layers are stacked to form the shape with the desired thickness.



http://www.rpc.msoe.edu

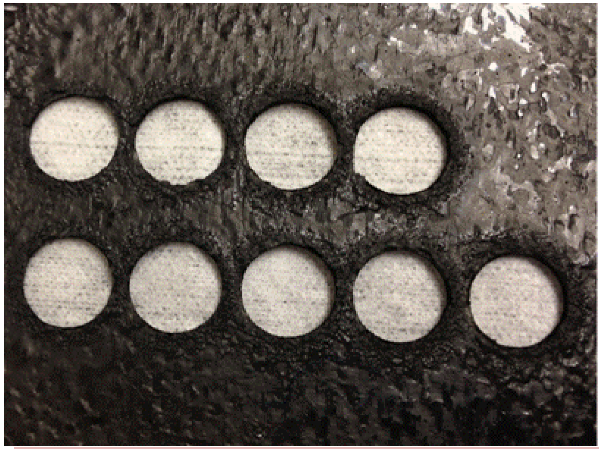
New CMC prepreg material development and characterization is a critical step

Evaluation of Laser Cutting Parameters for Silicon Carbide Fabrics and Prepregs

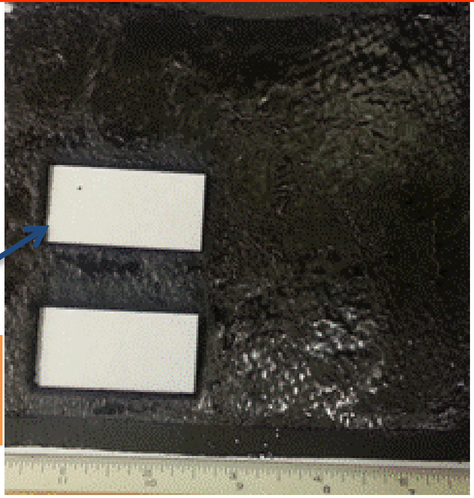
Prepregs for Composite Processing

- A number of SiC (Hi-Nicalon S, uncoated) fabrics (~6”x6”) were prepregged.
- These prepregs were used for optimization of laser cutting process.
- Baseline laser cutting data was also generated for different types of SiC fabrics (CG Nicalon, Hi-Nicalon, and Hi-Nicalon S)

Universal Laser System (Two 60 watt laser heads and a work area of 32”x18”)



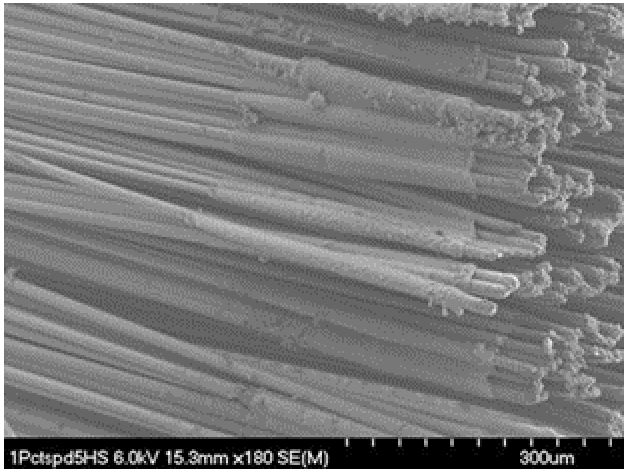
SEM specimens cut with different laser power/speeds



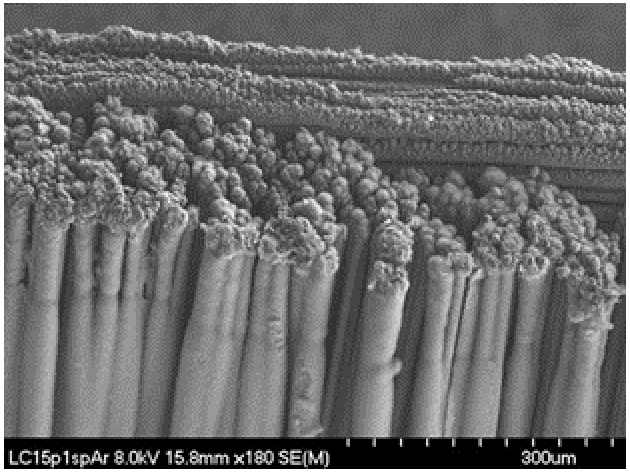
Laser cut prepregs used for composite processing

Investigation of Laser Cutting Parameters (Hi-Nicalon S, 5HS Fabric and Prepreg)

Fabrics

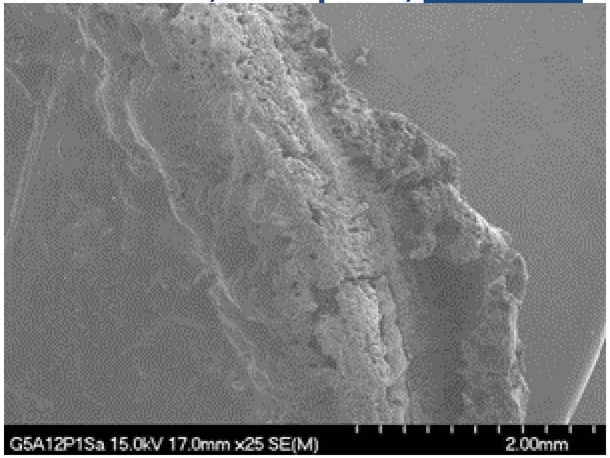


15% Power, 1% Speed, no purge

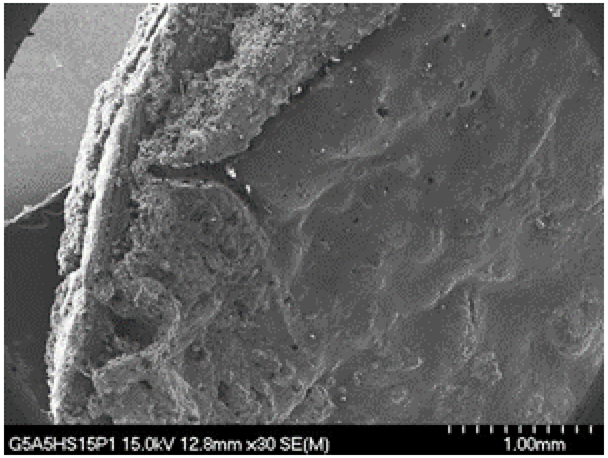


15% Power, 1% Speed, w/Ar Purge

Prepregs



12% Power, 1% Speed, no purge

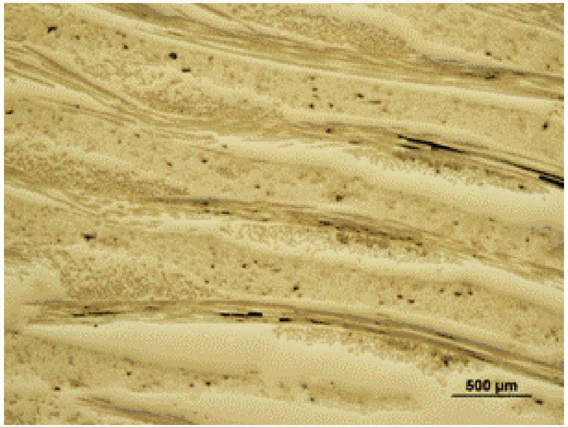


15% Power, 1% Speed, no purge

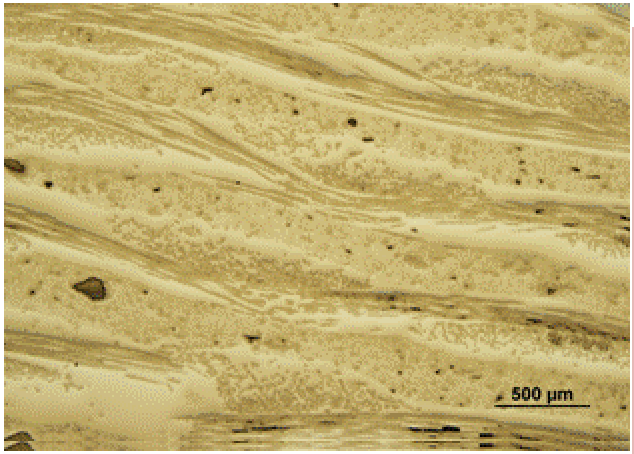
Microstructure of SiC/SiC Composites Fabricated Using Silicon Infiltration

Fibers Used for Prepregs: SiC (Hi-Nicalon S Fibers, 5 HS weave)
Fiber Interface Coating: None
Prepreg Composition: Prepreg 5A Nano 2 + Si

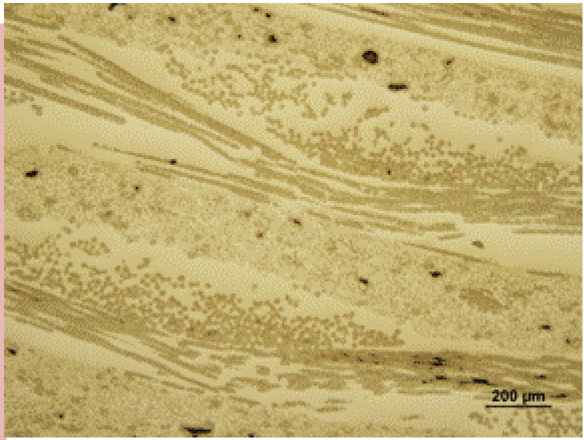
Green Preforms: 8 layers of prepregs;warm pressed @75-85°C



Silicon Infiltration: 1475 C, 30 minutes in vacuum



- Dense matrix after silicon infiltration. However, uncoated fibers are damaged due to exothermic Si+C reaction.
- Fiber coatings needed to prevent silicon reaction and provide weak interface for debonding and composite toughness.



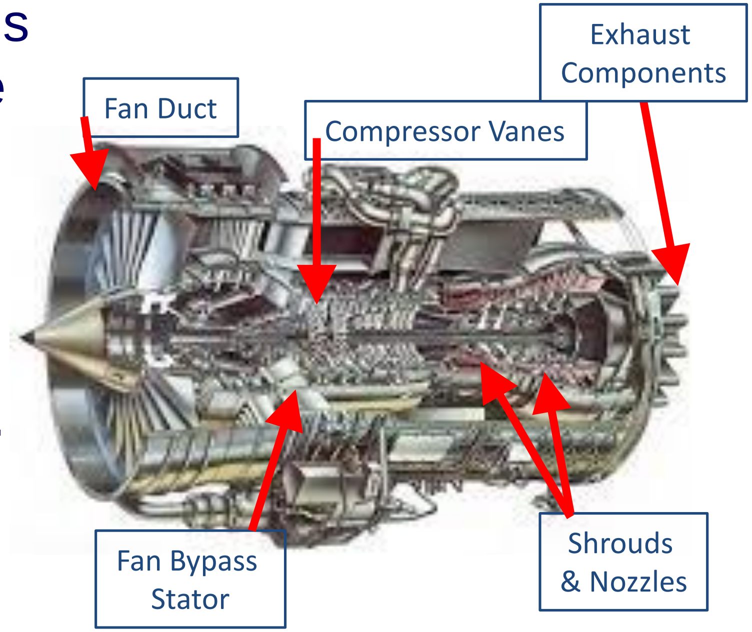
Non-Metallic Turbine Engine Project

TEAM: NASA GRC, OAI, Honeywell Aerospace, RP+M, NASA LRC

Project Objective: Conduct the first comprehensive evaluation of emerging materials and manufacturing technologies that will enable fully non-metallic gas turbine engines.

- Assess the feasibility of using additive manufacturing technologies to fabricate gas turbine engine components from polymer and ceramic matrix composites.
  - Fabricate and test prototype components in engine operating conditions
- Conduct engine system studies to estimate the benefits of a fully non-metallic gas turbine engine design in terms of reduced emissions, fuel burn and cost

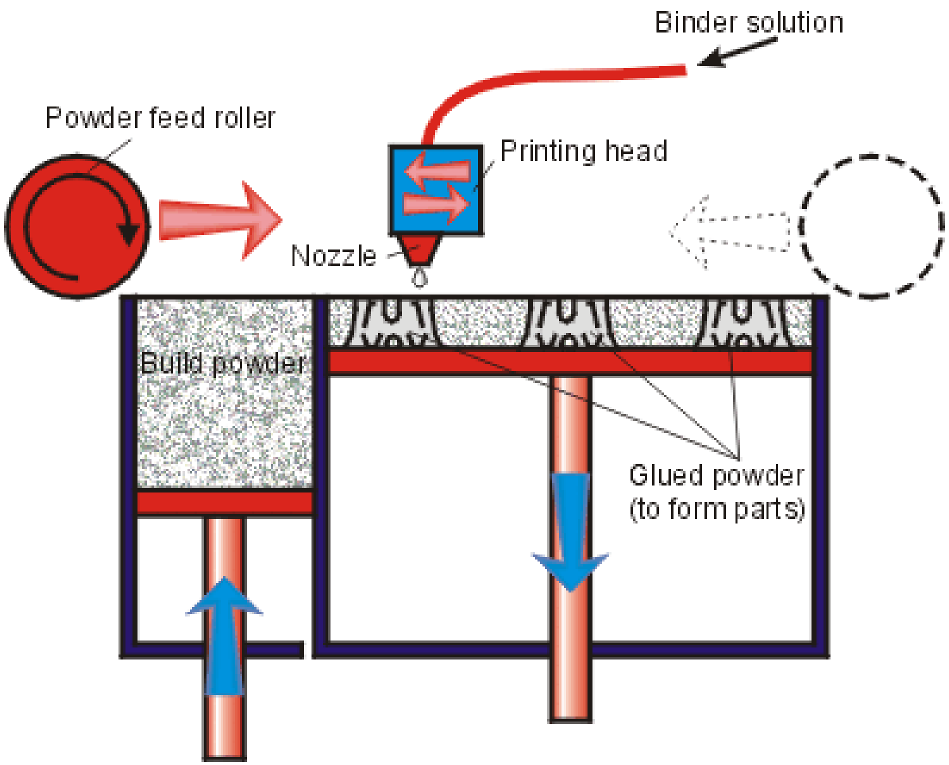
Targeted Components

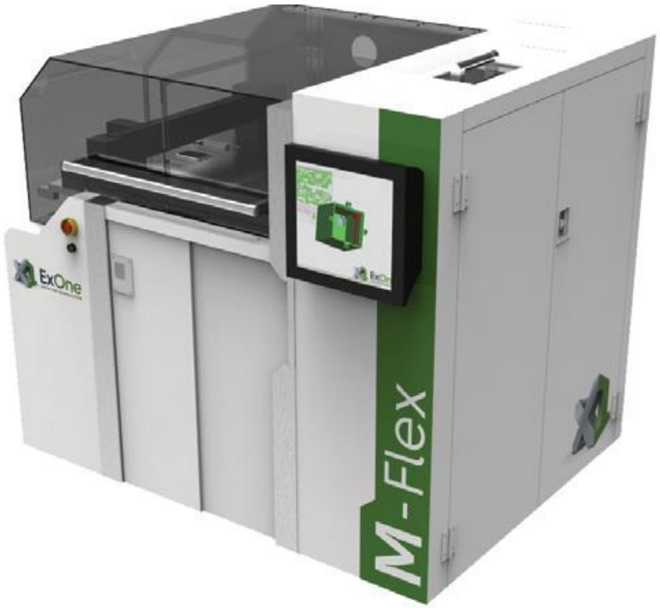


Business Jet size turbofan engine

Additive Manufacturing of Ceramics using Binder Jet Printing Technologies

In Collaboration with rp+m





ExOne's M-Flex print machine

Binder Jet printing

An inkjet-like printing head moves across a bed of powder and deposits a liquid binding material in the shape of the object's cross section

Binder jet printing capability will allow for powder bed processing with tailored binders and chopped fiber reinforcements for advanced ceramics.

Approach for Additive Manufacturing of CMCs

Processing

- Constituents
  - <u>SiC powders</u>: Carborex 220, 240, 360, and 600 powders (median grain sizes of 53, 45, 23, and 9 microns respectively). Used solely and in powder blends
  - <u>Infiltrants</u>: SMP-10 (polycarbosilane), SiC powder loaded SMP-10, phenolic (C, Si, SiC powder loaded), pure silicon
  - <u>Fiber reinforcement</u>: Si-TUFF SiC fiber; 7 micron mean diameter x 65-70 micron mean length, 350 GPa Modulus
  - Optimization of powder spreading and bimodal distributions of powders is critical

Microstructure

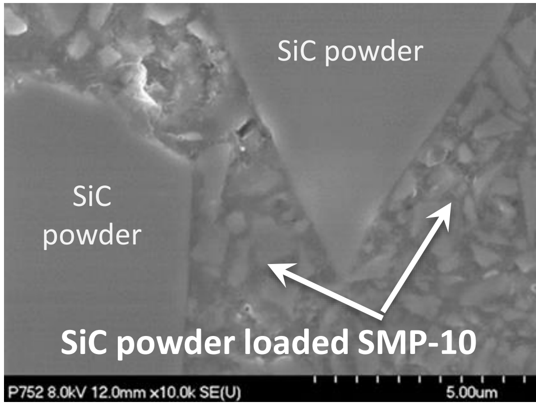
- Optical microscopy
- Scanning electron microscopy

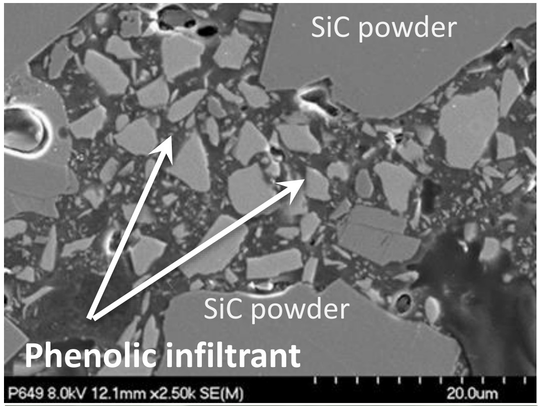
Properties

- Material density (as-manufactured and after infiltration steps)
- Mechanical properties: 4-point bend tests

Processing, microstructure, and property correlations provide an iterative process for improving the CMC materials.

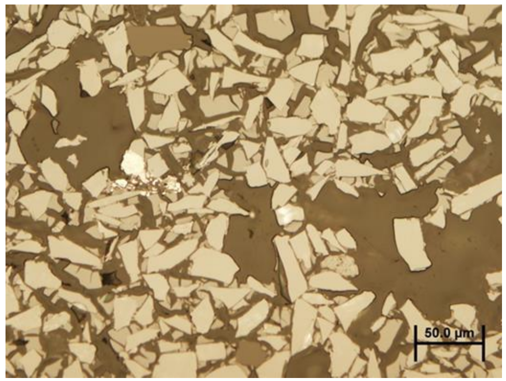
Constituents

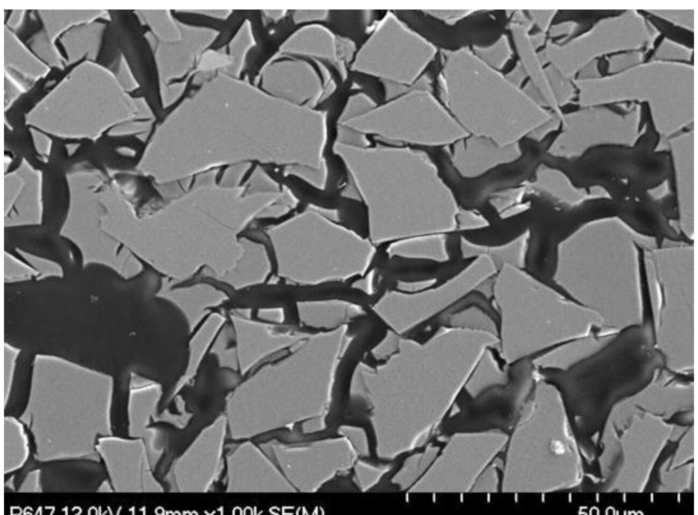




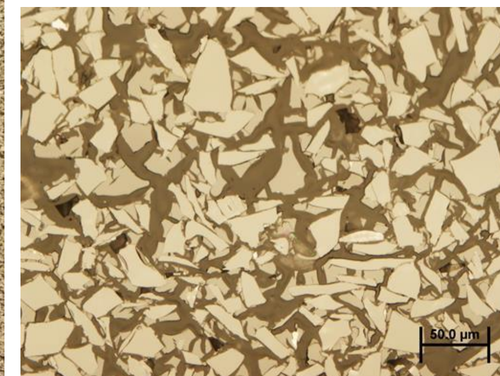
Fabrication and Microstructure of Monolithic SiC

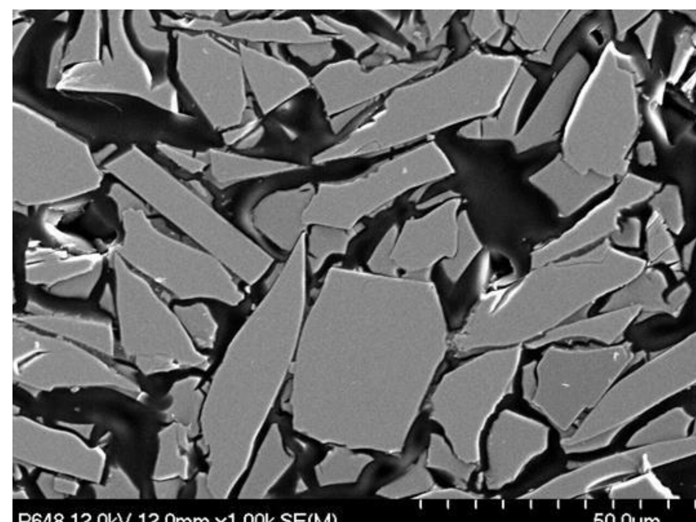
Carborex 240 SiC Powders with SMP-10 Infiltration



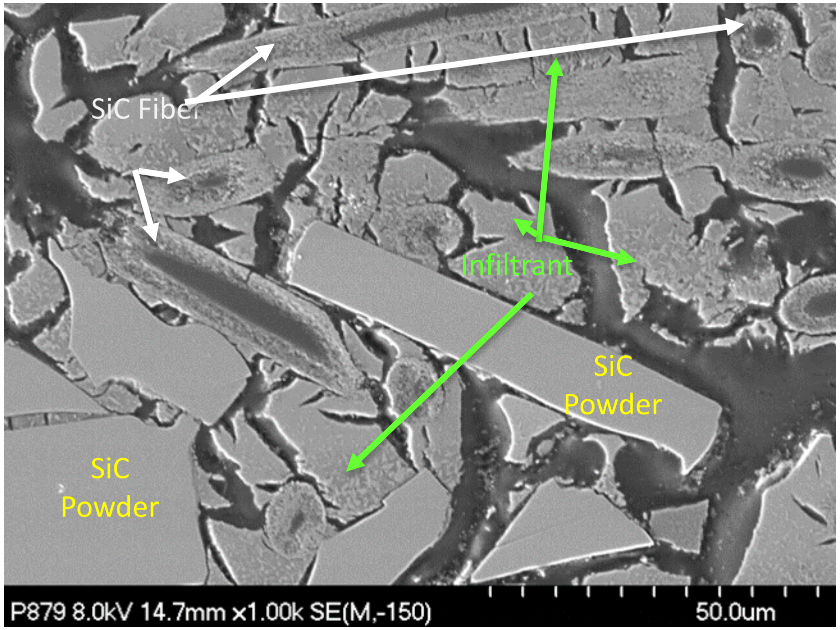


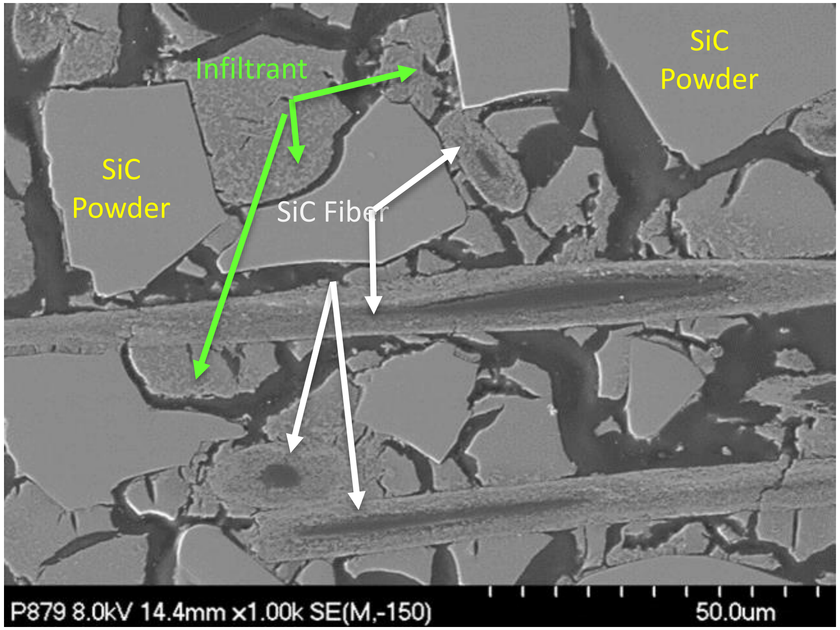
Carborex 360 SiC Powders with SMP-10 Infiltration

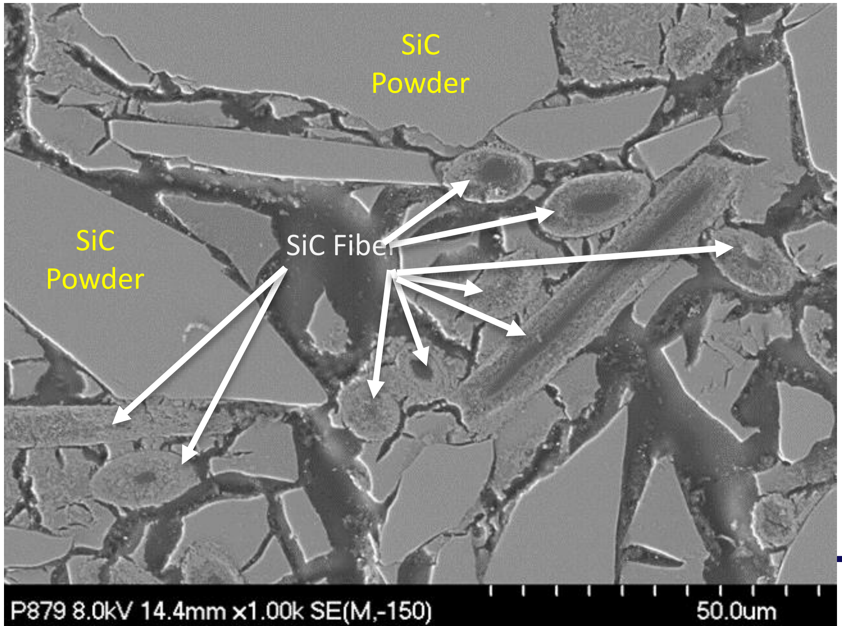




Fabrication and Microstructure of SiC Fiber Reinforced CMCs



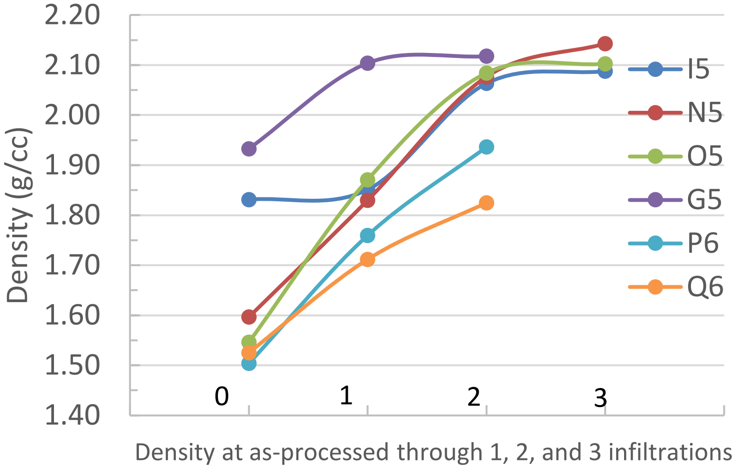




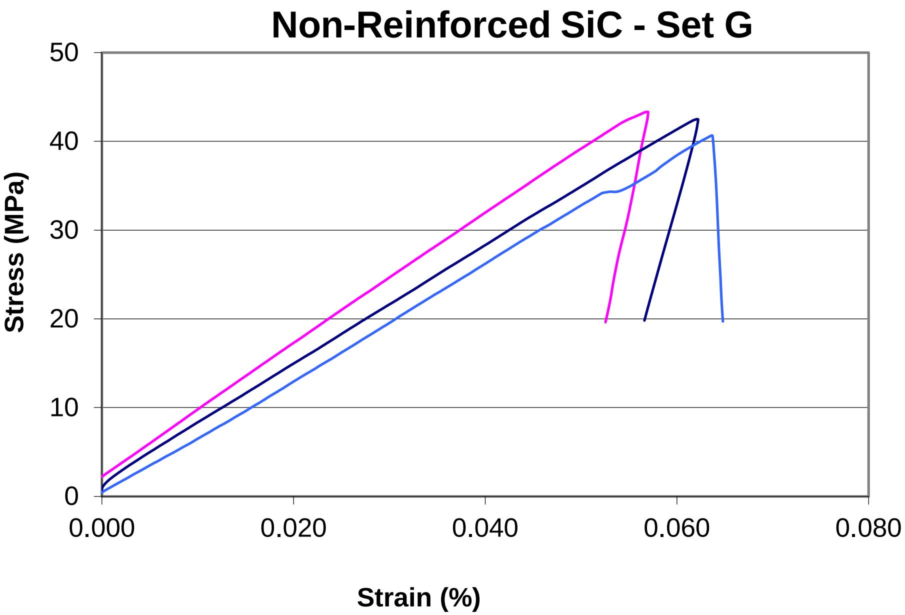
Different views of are shown of a CMC coupon with 35 vol% SiC fiber loading and infiltrant with smaller SiC powders.

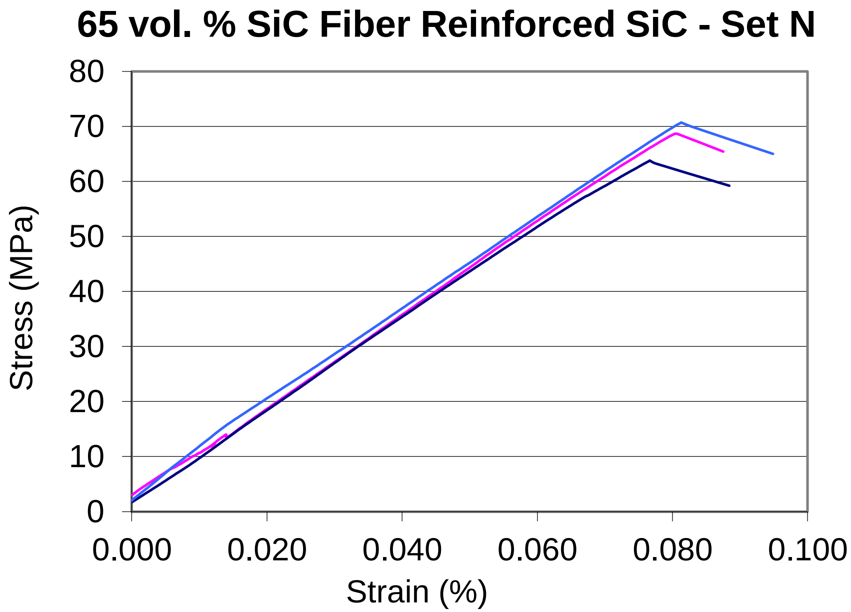
- ***Higher density observed due to powder loaded infiltrant***
- ***Good distribution and non-preferred orientation of SiC fibers is observed.***

4 Point Flexure Tests of the Monolithic SiC and CMC materials - at room temperature and 1200°C

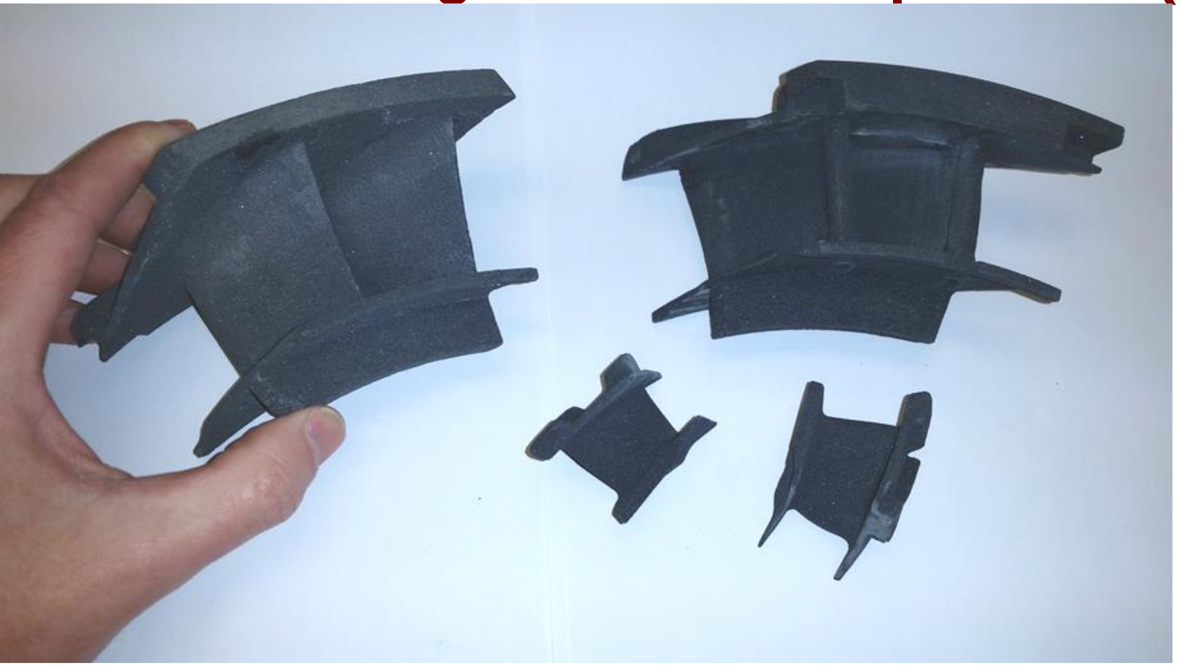


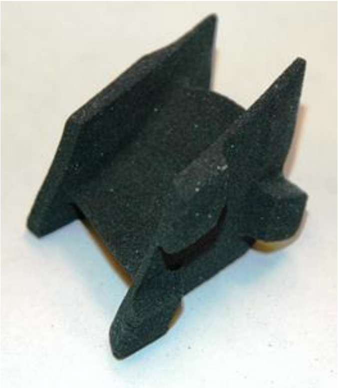
The fiber loaded SiC materials had significantly higher stresses and higher strains to failure.





Demonstration of the Additive Manufacturing of Turbine Engine CMC Components (20 vol.% SiC Fiber)





First stage nozzle segments.

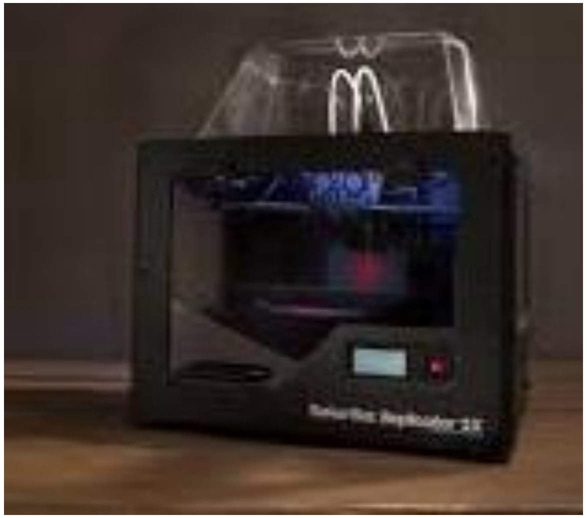
High pressure turbine nozzle segments: cooled doublet vane sections.

Additive Manufacturing of Ceramics using 3-D Printing Technologies

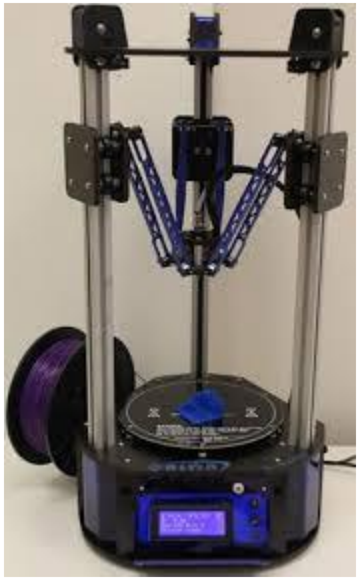
Objective: **To develop and characterize feed materials for 3-D printing of silicon carbide (SiC)-based ceramics.**

3-D Printing Efforts

- Powder Loaded Filament - direct printing of ceramic parts
- Wood Containing Filament - provide preforms for densification
- Slurry Dispensing of Pastes - evaluate pastes for full conversion to dense SiC



MakerBot Replicator 2X



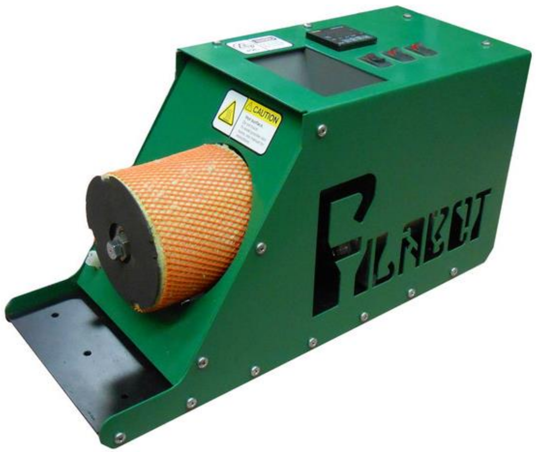
Orion Delta 3D Printer

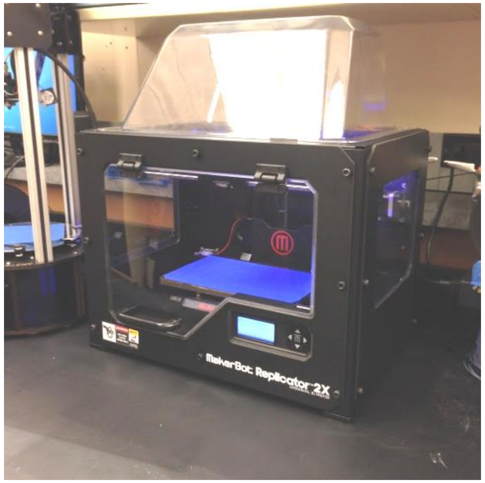
Rostock 3D Printer

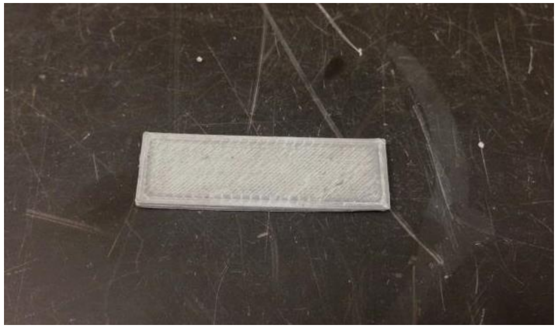
These printers can print polymers with specific filaments
Ability to fabricate ceramics is being investigated

3-D Printing: Powder Loaded Filament

- **Green SiC ceramic filament was extruded for the 3-D printing.**







3-D Printed Sample

3-D Printing: Wood Containing Filament Parts for Ceramic Preforms and Conversion

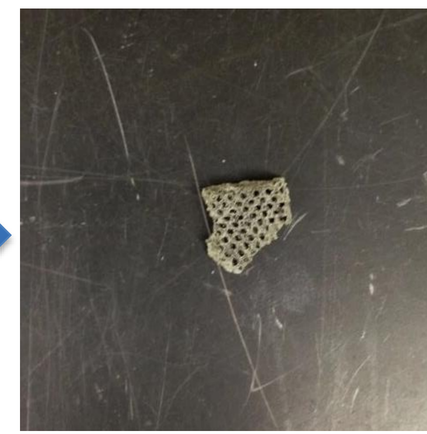
A 3-d printed disc is made using a commercially available wood filament. Printed part is pyrolyzed to serve as a preform.

Procedure:

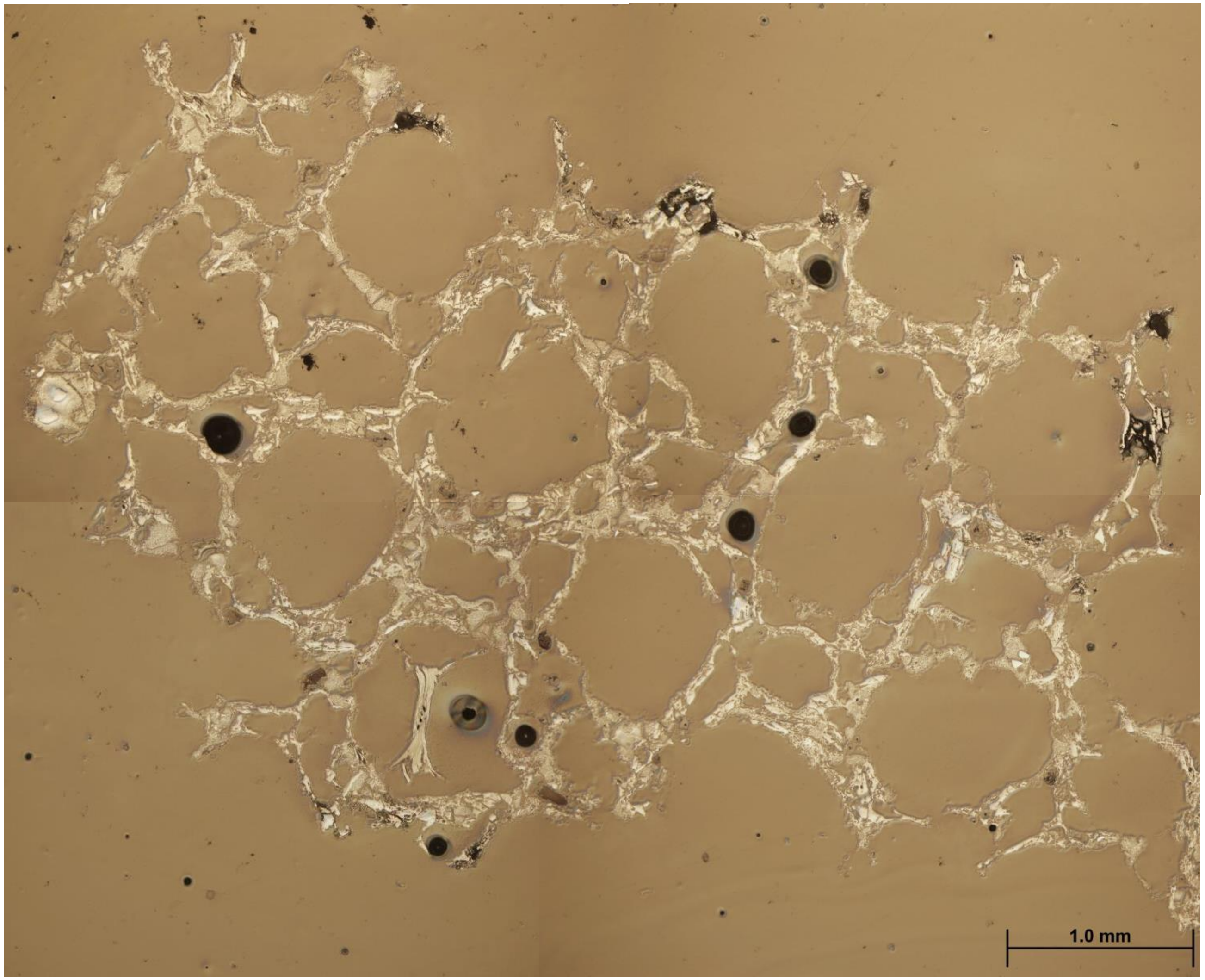
3-D printed porous disc → Dip-coated in Polycarbosilane (PCS) solution → Heat treated at 400°C in argon → Dip-coated in PCS solution → Exposed to 1000°C in argon → Pyrolyzed at 1450°C in vacuum

35% wt. Retention

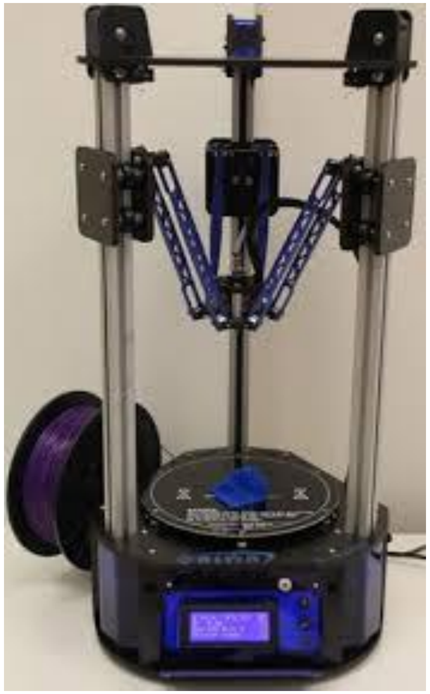
50% wt. Retention



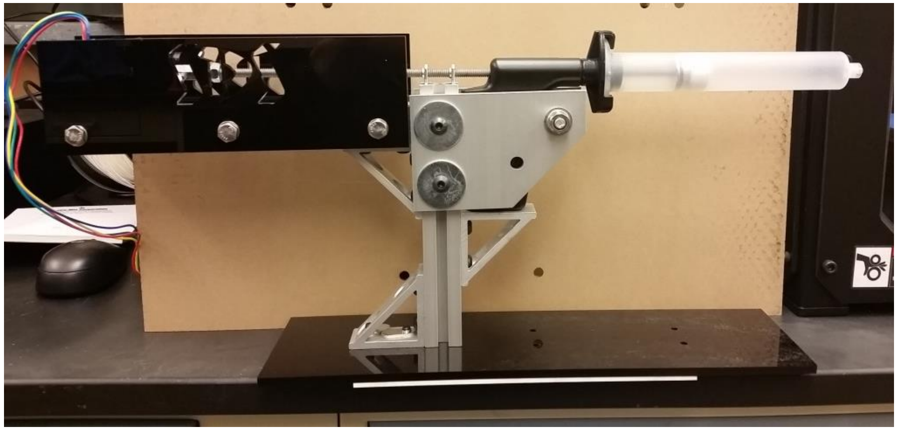
Wood Containing Filament – PCS/SiC then PCS –1450°C



3-D Printing: Slurry Dispensing of Pastes

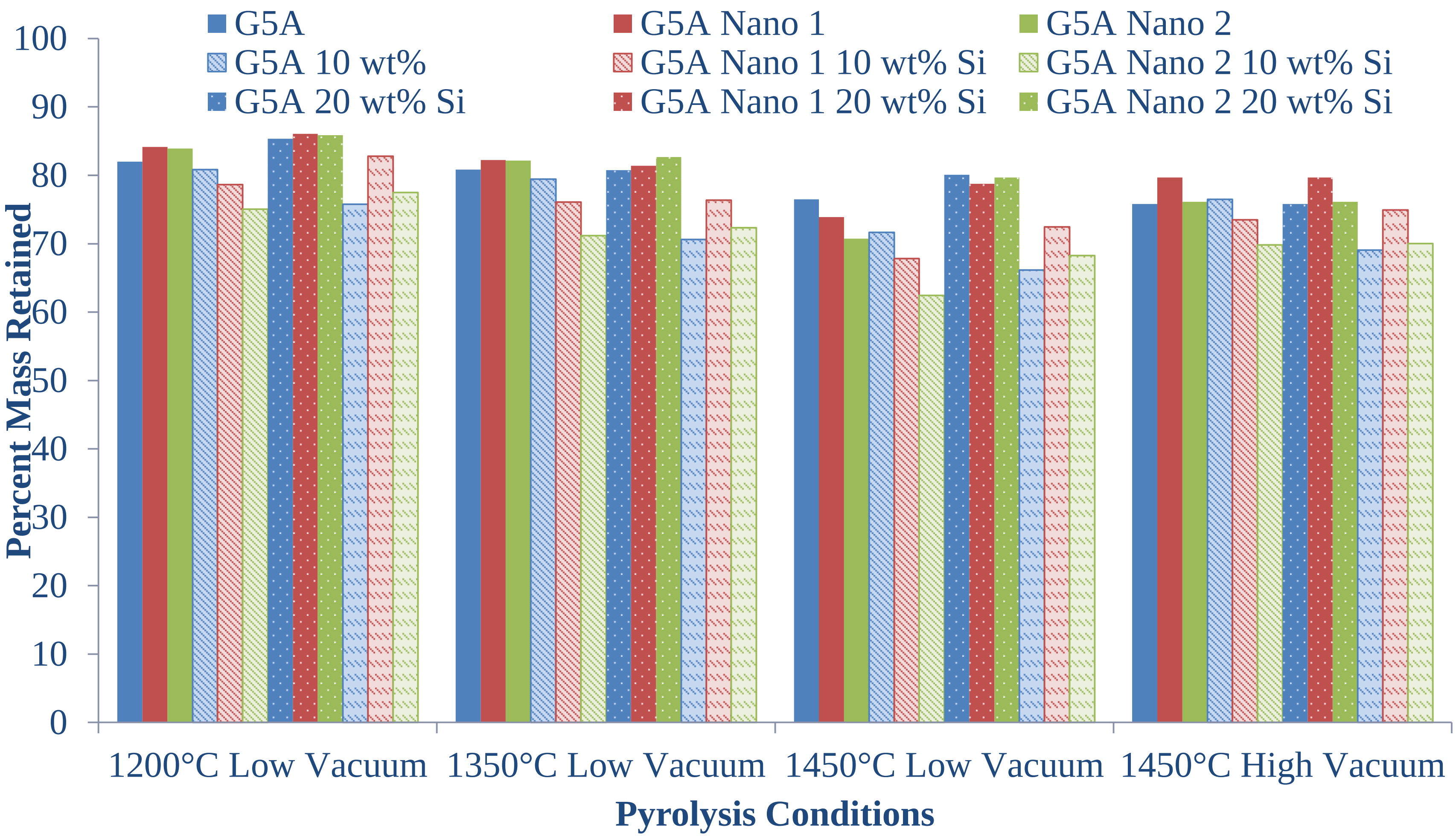


Orion Delta 3D Printer



Weight retention values are promising for all samples → high structure retention

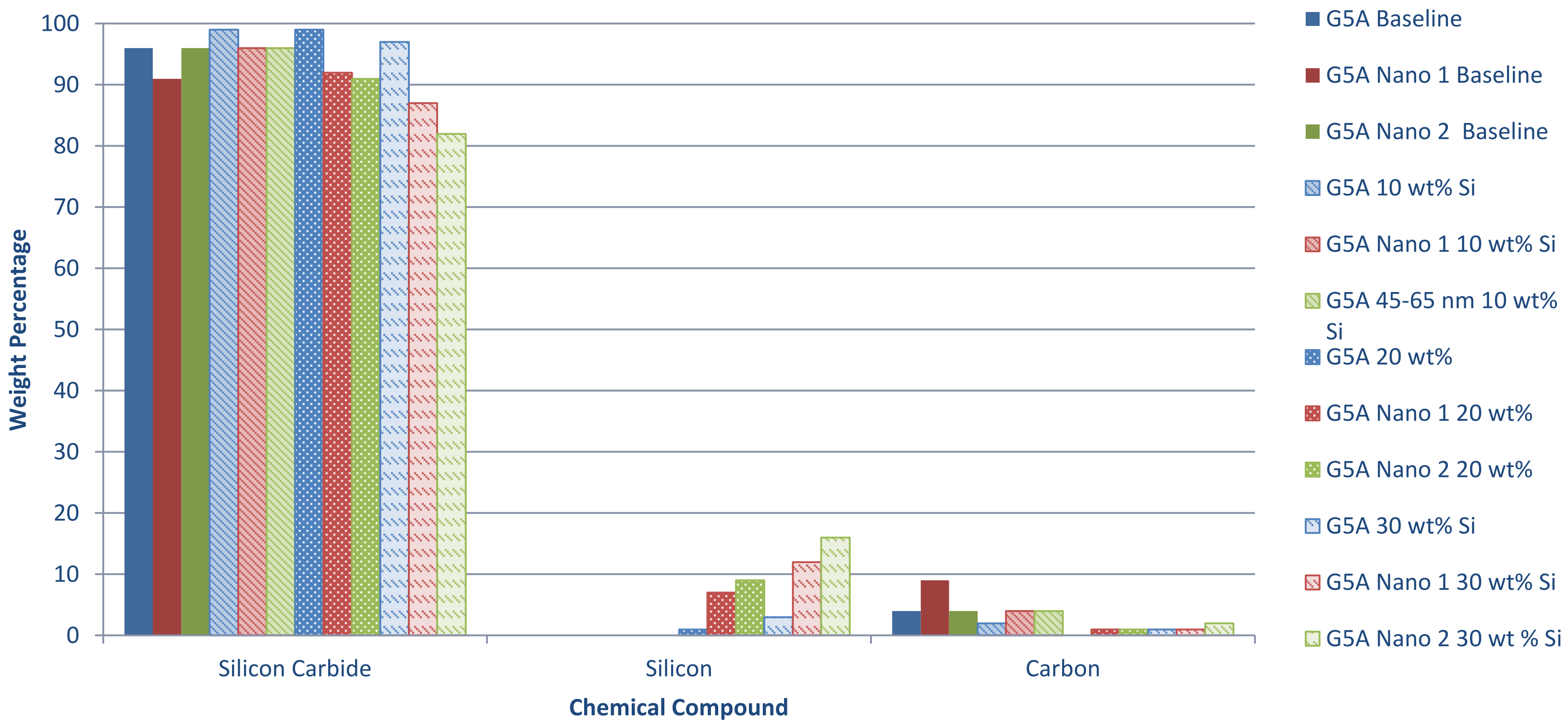
Weight Retention of Pre-Ceramic Pastes



Weight retention values are promising for all samples → secondary infiltration steps may not be necessary

Weight loss trends found in furnace weight loss studies similar to TGA data

Paste Evaluation: Composition of Samples after Heat Treatment at 1450°C in Low Vacuum



G5A has most SiC consistently
10wt% is the most promising

Summary/Conclusions

- Additive manufacturing can offer significant advantages in fabricating preforms, ceramics and CMCs.
- They will have to be selectively applied to "traditional" components but can also enable new applications.
- Good progress is occurring in binder jet processing.
- 3-D printing of ceramics has the potential to be game changing.

Acknowledgements

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- **The authors would like to thank personnel at rp+m for their collaborative efforts in binder jet printing.**

- **The authors would like to thank summer student, Shirley Zhu (The Ohio State University).**