COE CST Tenth Annual Technical Meeting

FAA COE CST Task #253: Ultra-High Temperature Composites Thermal Protection Systems

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Center of Excellence for Commercial Space Transportation

Agenda

- Team Members
- Task Description
- Schedule
- Goals
- Results
- Conclusions and Future Work

Team Members

- **People**
  - PIs: Drs. Jan Gou & Jay Kapat
  - Students: Derek Saltzman, Haonan Song and Shengheng Gu
- **Organizations**
  - Industry and Research Partners: Composites Development & Research and Spectral Energies
  - Organizations Providing Matching Funds: University of Central Florida

Task Description

- **PDC Precursor Development**: synthesis of polymer derived ceramic (PDC) precursor and modification with nanomaterials (i.e. carbon nanotubes, boron nitride, etc.) for high thermal stability, oxidation stability, and chemical stability
- **Manufacturing Process Development**: additive manufacturing (AM) process and polymer infiltration and pyrolysis (PIP) process of PDCC thermal protection systems
- **Testing & Performance Evaluation**: ground testing of PDCC thermal protection systems with oxyacetylene torch test, shock tube test, rocket plume test, and arc jet test
- **Thermal-Mechanical Modeling**: development of thermal-mechanical models to uncover the thermal damage mechanism

Schedule

2020 - 2021

- **Q1**: Synthesis of polymer derived ceramic (PDC) precursor
- **Q2**: AM process and PIP process of PDCC thermal protection systems
- **Q3**: Oxyacetylene Torch Test of PDCC thermal protection Systems
- **Q4**: Rocket Plume Test and Shock Tube Test of PDCC thermal protection systems

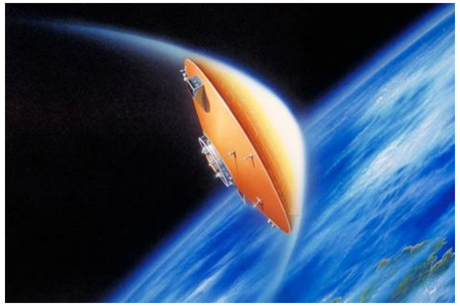
Goals

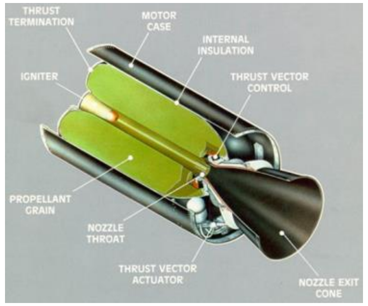
Research Objective:

Develop polymer derived ceramics composites (PDCC) for **ultra-high temperature**, **light weight**, **low erosion**, and **cost effective** composites thermal protection systems

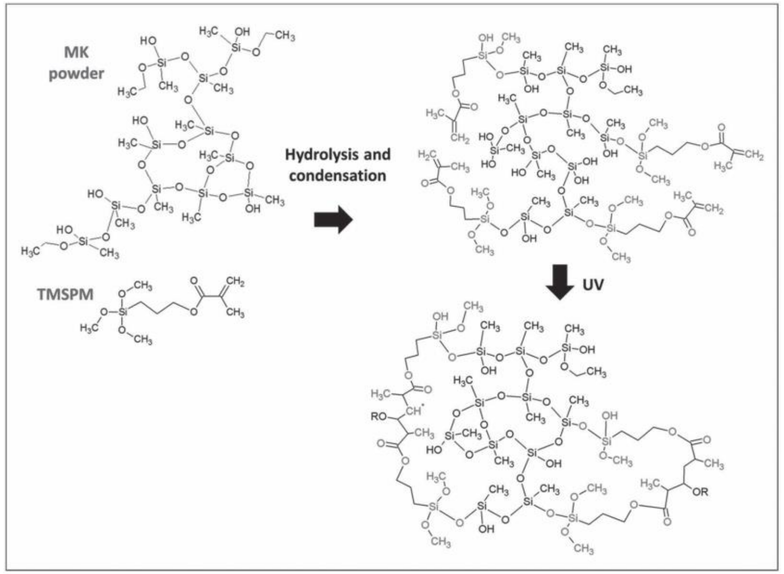
Relevance to Commercial Space:

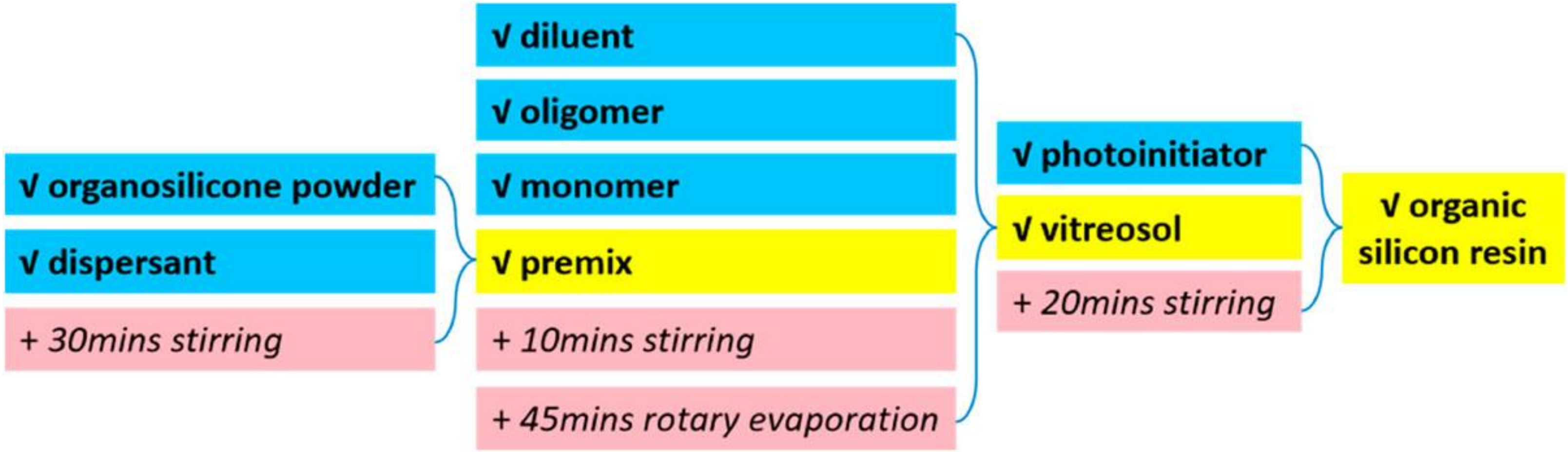
The PDCC thermal protection systems potentially can be used for a variety of applications such as re-entry vehicles, launch vehicles, hypersonic, combustors, gas turbine blades, and heat exchangers in high temperature and high pressure harsh environments.

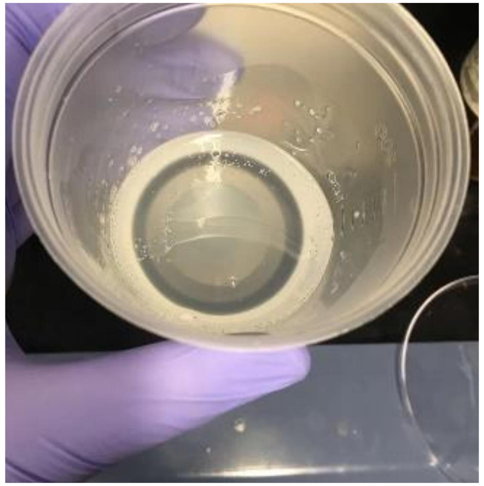




Results: Synthesis of PDC Precursor

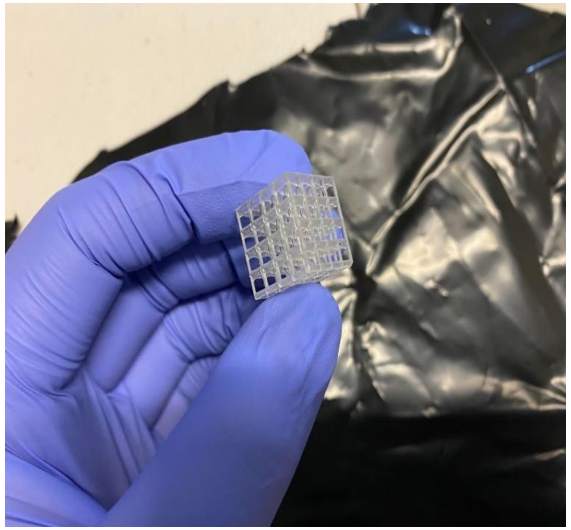






- The photosensitive methyl-silsesquoxane preceramic polymer was synthesized from silicone powder (CH_3-$SiO_{3/2}$)x and silicone alkoxide (3-(trimethoxysilyl)propyl methacrylate, TMSPM) through the sol-gel hydrolysis process.
- The radical based photo-initiator 819 (phenylbis (2,4,6-trimethlbenzoyl) phosphine oxide) was used to make the precursor photo-curable.

Results: Additive Manufacturing and FTIR Characterization

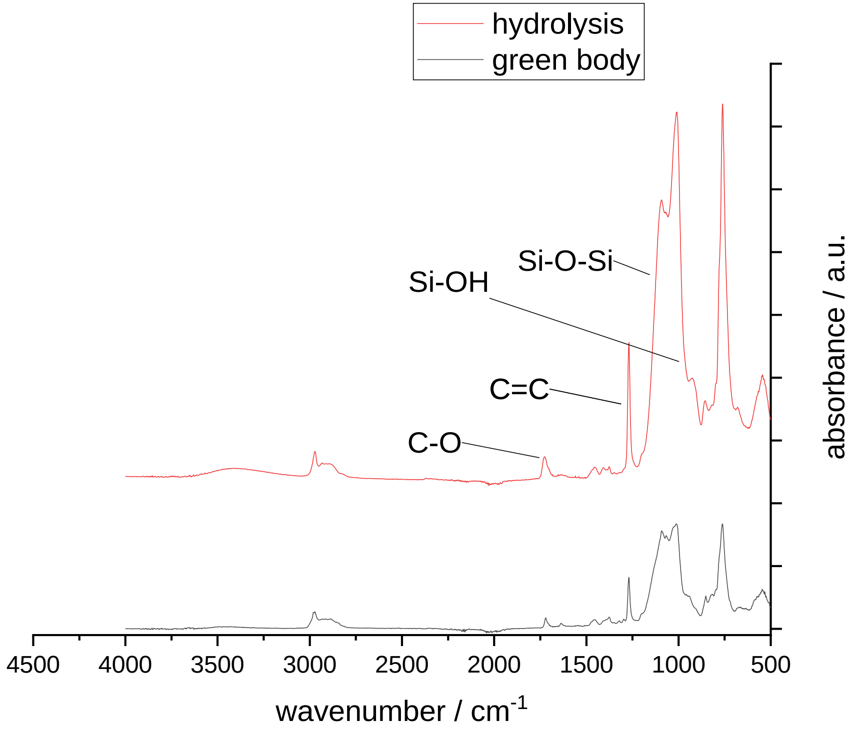


Quality

Layer Height	0.05	mm
Initial Layer Height	0.1	mm
Line Width	0.067	mm
Wall Line Width	0.067	mm
Outer Wall Line Width	0.067	mm
Inner Wall(s) Line Width	0.067	mm
Top/Bottom Line Width	0.067	mm
Infill Line Width	0.067	mm

Speed

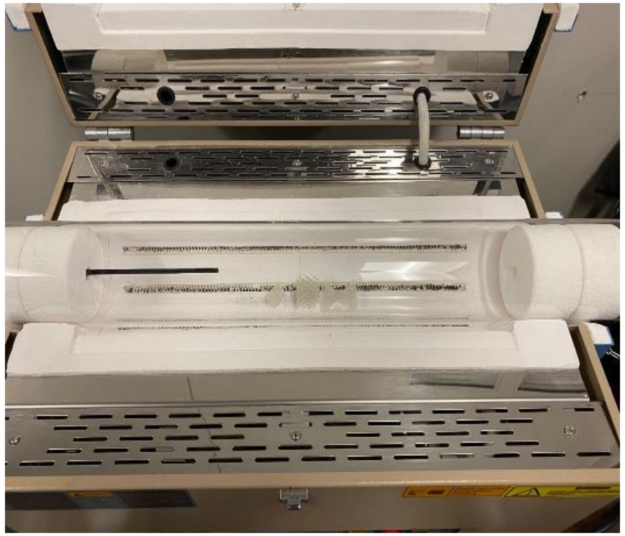
Print Speed	130	mm/s
Infill Speed	130	mm/s
Wall Speed	130	mm/s
Outer Wall Speed	130	mm/s
Inner Wall Speed	130	mm/s
Top/Bottom Speed	130	mm/s
Travel Speed	300	mm/s
Initial Layer Speed	5	mm/s
Initial Layer Print Speed	5	mm/s
Initial Layer Travel Speed	300	mm/s
Maximum Z Speed	0	mm/s
Number of Slower Layers	2	

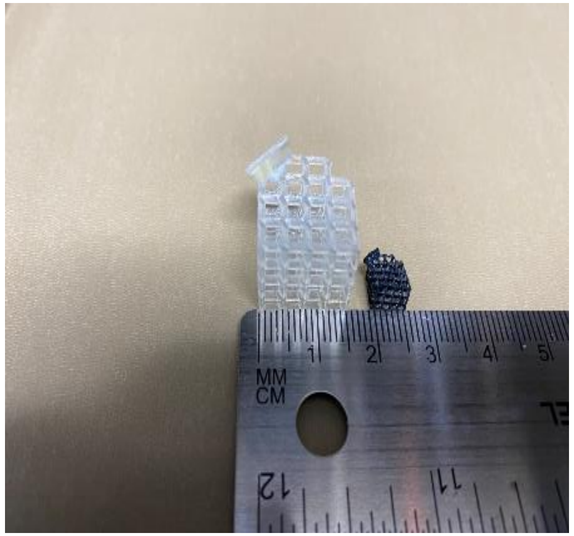


- Changes in function groups were detected by FTIR spectrum to confirm the polymerization after 3D printing.
- The declination in peaks of C-O, C=C, Si-O-Si implied that the unsaturated function groups have been converted during the radical photo-polymerization process.

Results: Pyrolysis Process and XRD Characterization

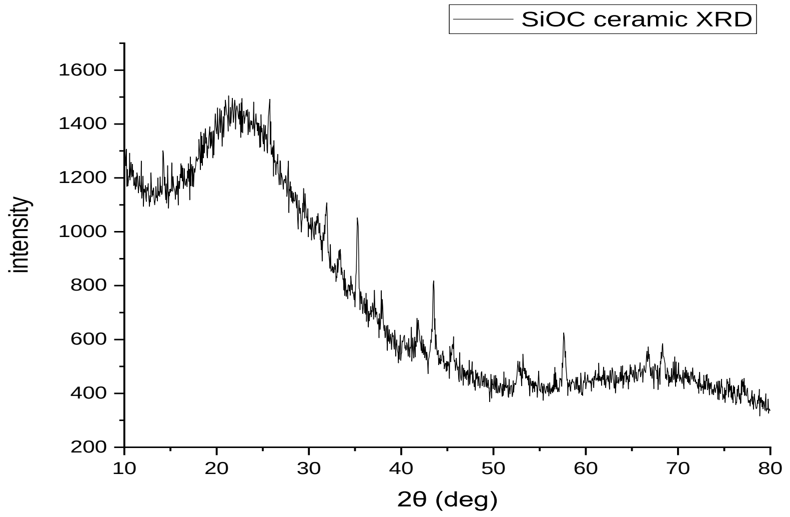
Pyrolysis Process





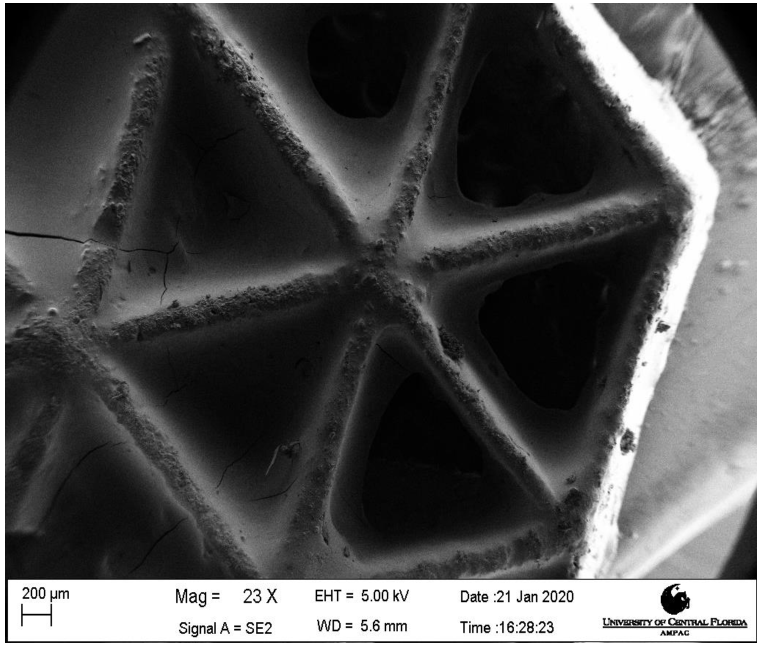
Pyrolysis Parameters:
The 3D-printed green body was pyrolyzed at 1,000°C for 1 h in nitrogen atmosphere with a heating rate of 1°C/min in a tube furnace.

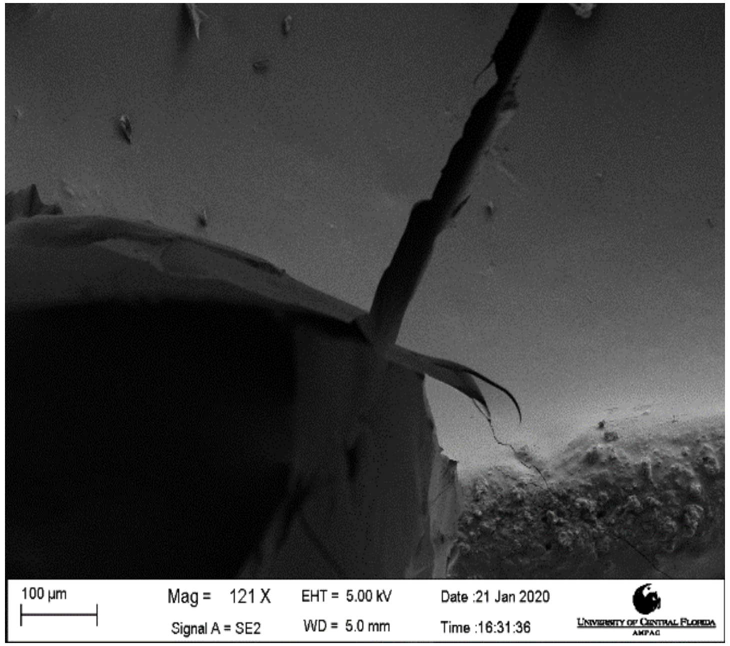
XRD Characterization

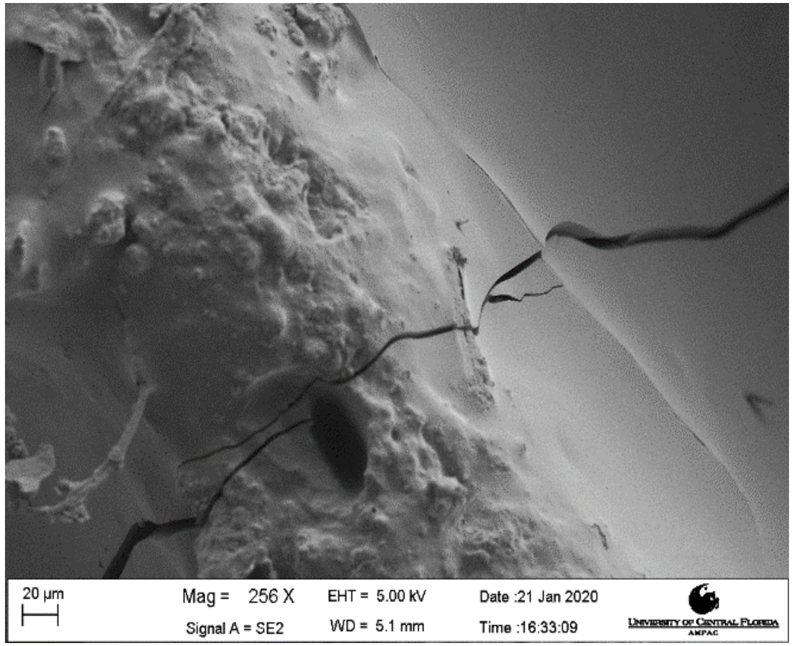


An absorption peak was observed at 43 degree corresponding to the characteristic diffraction of ß-SiOC, suggesting the possible crystallization of the SiOC ceramics.

Results: Microstructural Characterization

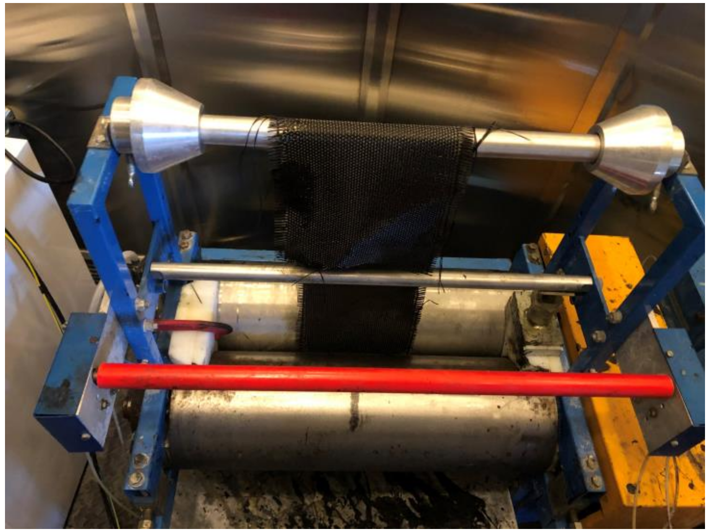






- Dense structures and smooth surfaces formed on the lattice ceramic structure after the pyrolysis process of the green body.
- Small cracks occurred due to residual thermal stress in the pyrolysis process of the green body.

Results: Polymer Infiltration & Pyrolysis (PIP) Process



Prepregging Stage

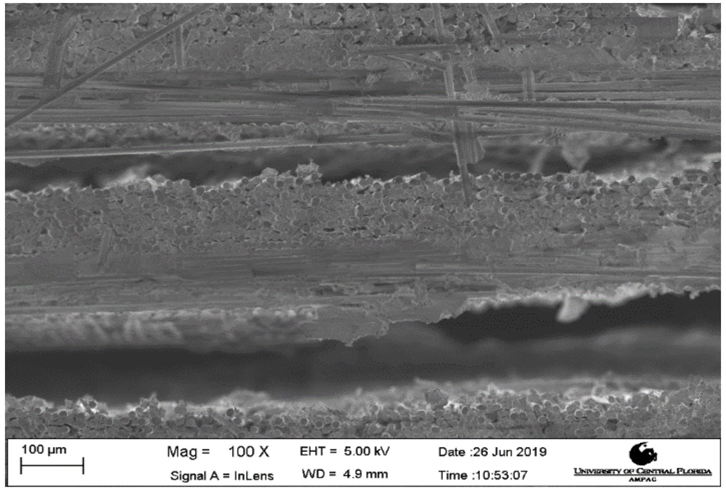
Autoclave Curing Stage

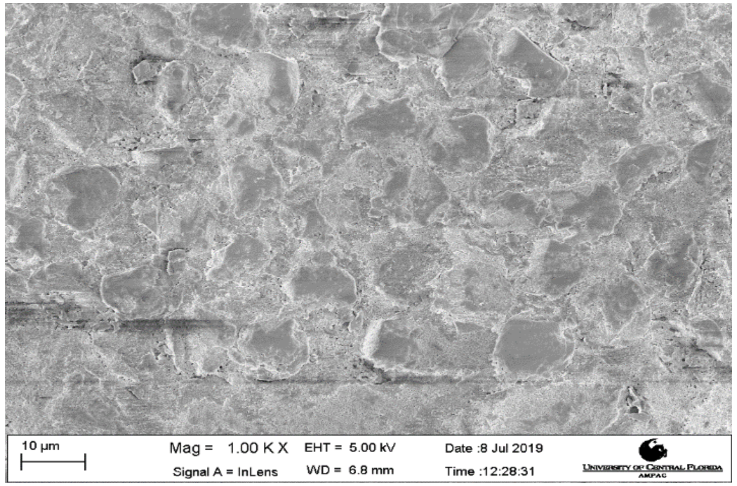
Pyrolysis Stage

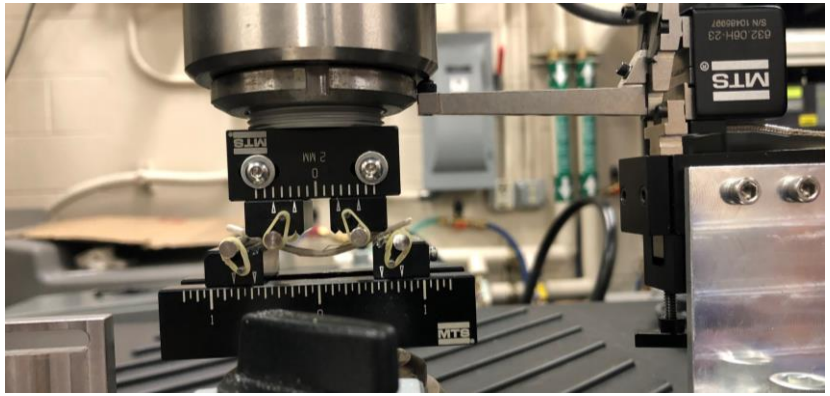
3M™ Nextel™ 610 Ceramic Fiber

- Chemical composition: >99% Aluminum Oxide (Al_2O_3), Crystal phase: α-Al_2O_3
- Melting point: 2,000°C, Continuous use temperature (single filament <1% strain): 1,000°C
- Filament diameter: 11-13 um, Filament tensile strength: 2,800 MPa, Filament tensile modulus: 370 GPa
- Density: 3.9 g/cm^3

Results: Microstructural Characterization and Mechanical Testing







Load-Deflection Relationship

Flexural strength:

$$\sigma = \frac{3PL}{4bd^2}$$

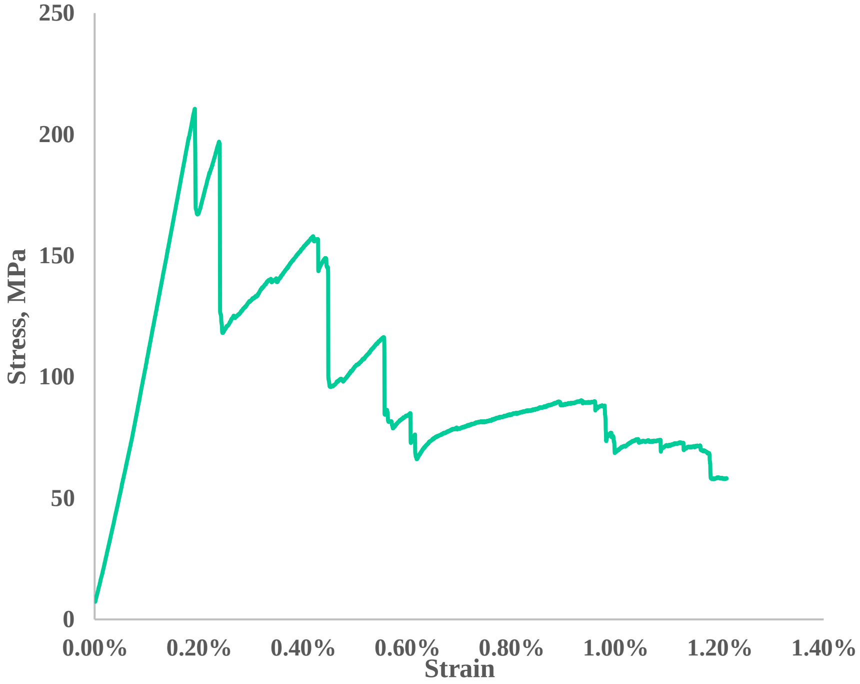
Flexural strain:

$$\varepsilon = \frac{4.36Dd}{L^2} \times \frac{8}{11}$$

Four-Point Bending

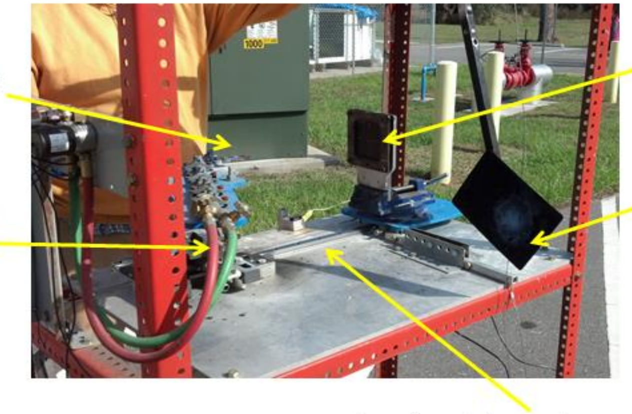
Flexural Strength: $\sigma = 210\ MPa$

Flexural Modulus $E = 77\ GPa$

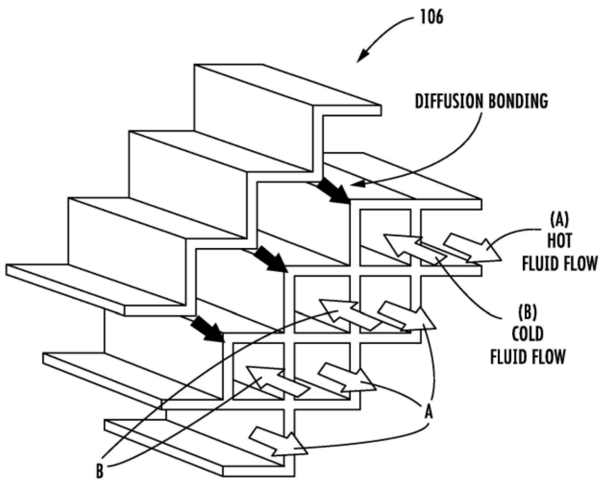


Results: Oxyacetylene Torch Testing

- **Oxyacetylene Torch Testing (On-Going)**
  - Referencing ASTM E285
  - Heat flux of 835 W/cm^2



Results: Polymer Derived Ceramic Composites (PDCC) for CMC Heat Exchanger in Harsh Environments



Low Cost PDCC Heat Exchanger

Technical Objective:

Develop high temperature, low-cost and lightweight polymer derived ceramics composites (PDCC) for high temperature, high pressure heat exchanger in powder generation, industrial components and aerospace systems, including supercritical CO_2 cycles.

Technical Approach:

- Materials development of PDCC composites with anisotropic thermal properties
- Manufacturing techniques of the PDCC heat exchanger
- Testing of the PDCC heat exchanger
- Thermo-mechanical and corrosion degradation study of PDCC in high temperature and high pressure environment

Power generation system using closed or semi-closed Brayton cycle recuperator, patented by

- *U.S. Patent,* Publication No. US10,598,093 B2, Issue date: 03/24/2020
- *European Patent,* Publication No. EP3 277 939 B1, Issue date: 05/06/2020
- *World Intellectual Property Organization (WIPO) Patent,* International Publication No. W O 2016/161052 A1, International Publication date: 10/06/2016
- **Inventors: Jay Kapat, Jihua Gou, Narasimha Nagaiah, Joshua Schmitt**

Publications, Presentations, Awards, & Recognitions

PUBLICATIONS

- J. Kapat, J. Gou, N. Nagaiah, J. Schmitt, Power generation system using closed or semi-closed Brayton cycle recuperator, *U.S. Patent,* Publication No. US10,598,093 B2, Issue date: 03/24/2020
- J. Kapat, J. Gou, N. Nagaiah, J. Schmitt, Power generation system using closed or semi-closed Brayton cycle recuperator, *European Patent,* Publication No. EP3 277 939 B1, Issue date: 05/06/2020
- J. Kapat, J. Gou, N. Nagaiah, J. Schmitt, Power generation system using closed or semi-closed Brayton cycle recuperator, *World Intellectual Property Organization (WIPO) Patent,* International Publication No. W O 2016/161052 A1, International Publication date: 10/06/2016
- S.S. Gu, J. Kapat, J. Gou, "Additive Manufacturing of Silicon Oxycarbide Ceramic Structures," *CAMX 2020*, Orlando, FL, September 21-24, 2020 (Virtual Conference)

PRESENTATIONS

- D. Poljak, D. Saltzman, J. Gou, "Mechanical Characterization and Evaluation of Oxide/Oxide Ceramic Matrix Composites," NSF REU Site: Advamced Technologies for Hypersonic, Propulsion, Energetic, and Reusable Platforms (HYPER) Program, University of Central Florida, August 2, 2019
- S.S. Gu, J. Kapat, J. Gou, "Additive Manufacturing of Silicon Oxycarbide Ceramic Structures," *CAMX 2020*, Orlando, FL, September 21-24, 2020 (Virtual Conference)

AWARDS

RECOGNITIONS

Conclusions and Future Work

- **Final Remarks**
  - The photosensitive PDC precursor has been developed towards additive manufacturing of ceramic structures.
  - The continuous fiber reinforced polymer derived ceramic composites has been developed using the PIP process.
- **Next Steps**
  - Formulation optimization of PDC precursor with nanomaterials for high temperature performance.
  - Additive manufacturing of continuous ceramic fiber/short fiber reinforced polymer derived ceramic composites.
  - PIP Manufacturing of polymer derived ceramic composites reinforced with 3D ceramic fabrics against the inter-laminar delamination caused by the thermal-mechanical loading.