COE CST Fourth Annual Technical Meeting:

Ultrahigh Temperature Composites for Thermal Protection Systems (TPS)

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COE CST
Center of Excellence for Commercial Space Transportation

Agenda

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

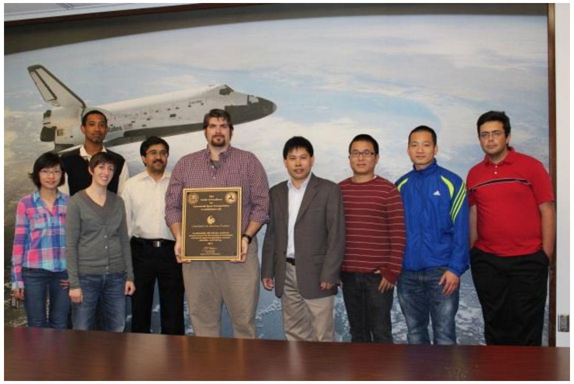
Team Members

Principle Investigators

- **Jan Gou -** Composite materials and structures, nanocomposite materials, advanced composites manufacturing: PMCs, CMCs and C/C composites
- **Jay Kapat -** Heat transfer, film cooling, aerodynamics testing

Graduate Students

- **Hongjiang Yang**: nanocomposite materials, design, manufacturing, characterization and testing of composite materials
- **Donovan Lui**: Ablation testing and CMC fabrication



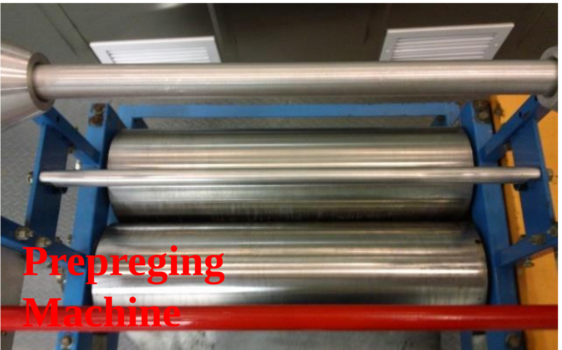
Task Description

Develop **anisotropic** thermal conductivity, **ultrahigh temperature**, **light weight** and **cost effective** carbon nanotube preform reinforced polymer derived ceramics (PDC) matrix composites for thermal protection systems.

STATEMENT OF WORK

- Develop carbon nanotube preforms (vertically aligned carbon nanotube (VACNT) arrays and buckypapers) for ceramic composites
- Design and fabrication of polymer derived ceramics (PDC) based ceramic matrix composites.
- Ground testing of CMC thermal protection systems with Oxyacetylene Exposure Test, Shock Tube Test and Hot Jet Facilities.
- Multi-scale modeling of CMC thermal protection systems.

Manufacturing of CMCs



Manufacturing of Fiber Reinforced Polymer Derived Ceramics (PDC) Composites

Prepregging with Resin Impregnator

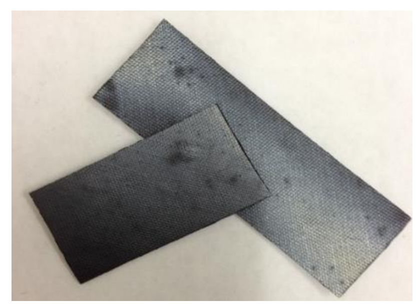
Prepreg woven fiber fabrics (ceramic fiber, carbon fiber, glass fiber, etc.) with polymer derived ceramics (PDC) resin

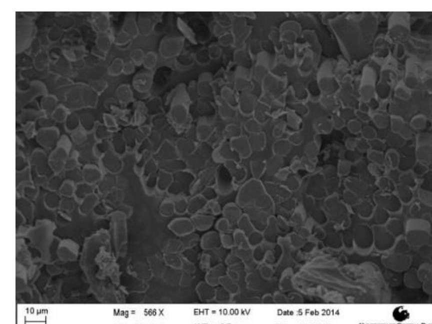
Autoclave Curing

PDC resin curing in an autoclave with computer control over all process variables (max pressure 200 psi, max temperature 800ºF) to become a greenware

Pyrolysis in Kiln

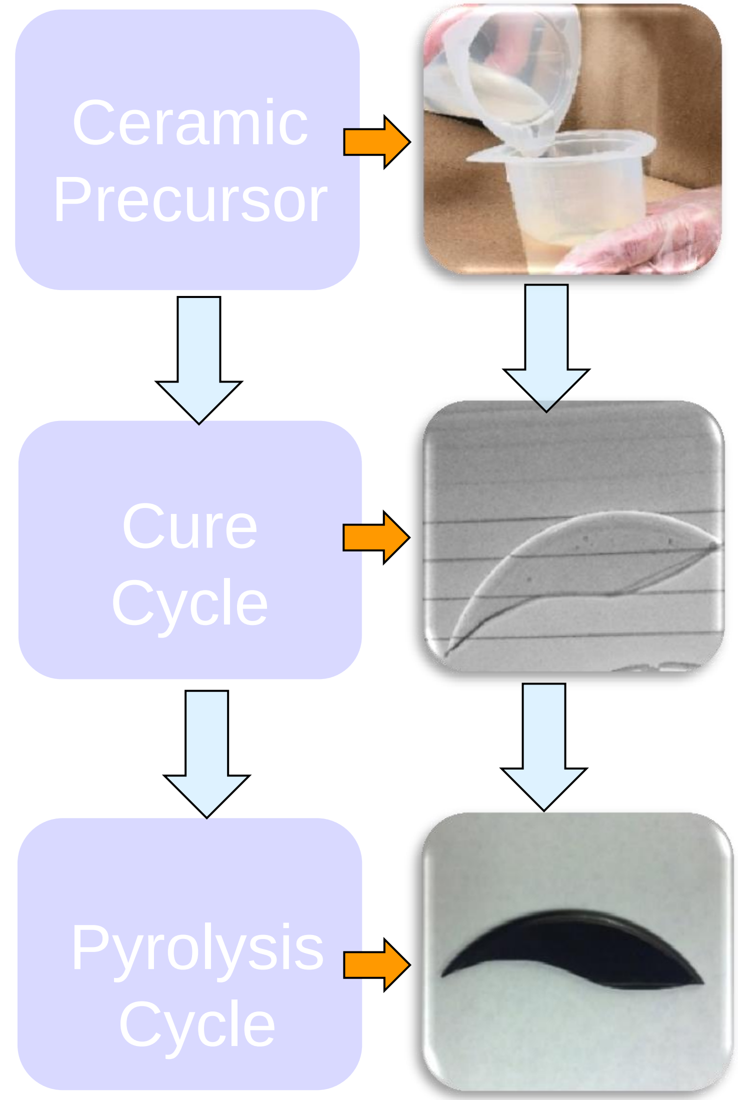
Pyrolyze the greenware into a ceramic part (max temperature 1,200 ºC)





Polymer Derived Ceramics Matrix

Property	Starfire System Polysiloxane(PSO)	Starfire System Polycarbosilane (PCS)
Denotation	SPR-688/SPR-212	SMP-10
Operating temperature	1,100 °C	1,800 °C
Density	1.11 g/cm^3	0.998 g/cm^3
Catalyst	Platinum CAT-776	Dicumyl Peroxide



High performances:

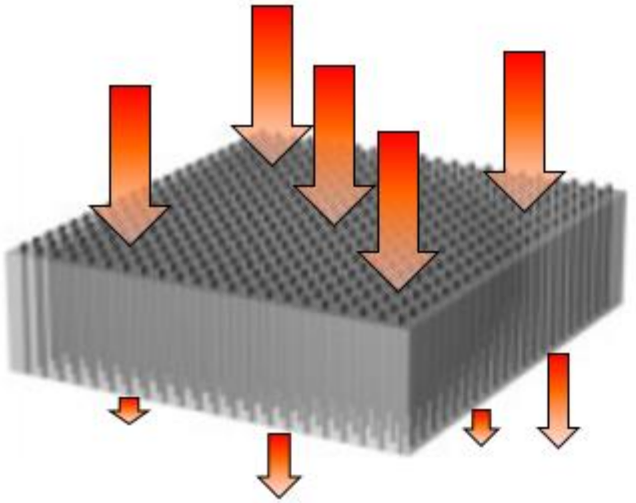
- Low cost
- Near net shape manufacturing
- Outstanding thermo-chemical stability

High Temperature Ceramic Fibers

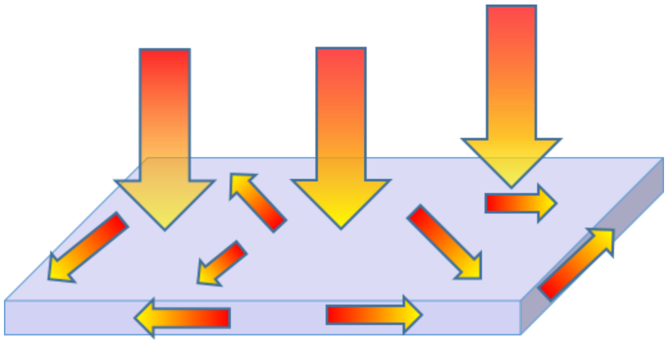
Trade-Name	Manufacturer	Use Temperature	Cost ($/Kg)	Filament Diameter (µm)	Density(g/cc)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Composition	Thermal Expansion (Ppm/°C)
T300	Toray	300~350 °C	68	7	1.74	3100	230	C	-0.7
Nextel 720	3M	1204 °C	660	10~12	3.4	2930	260	Al_2O_3/SiO_2	6
SCS-Utra	Specialty Material	1371 °C	~9000	142	3.08	3900	380	SiC	4.1
SiC-1900X	MATECH	~ 1482 °C	-	10~12	3.14	2500	367	β-SiC	-
Nicalon NL-200	Nippon Carbon	1100 °C	~2000	14	2.55	3000	220	SiC	3.1-3.2
Hi-Nicalon	Nippon Carbon	1230 °C	8000	14	2.74	2800	270	SiC	3.3-3.5
Hi-Nicalon Type S	Nippon Carbon	1450°C	13000	12	3.1	2600	420	SiC	3.5
Sylramic	COI Ceramics	1420 °C	10000	10	3.55	3200	380	SiC	5.4
Tyranno SA 1-3	Ube Industries	1700 °C	5000	10	3.02	2800	375	SiC	-

Research Methodology

Control the direction of heat transfer “pathway” by arranging the orientation of carbon nanotubes: <u>through-thickness direction</u> and <u>in-plane direction</u>



VACNT Array



Buckypaper

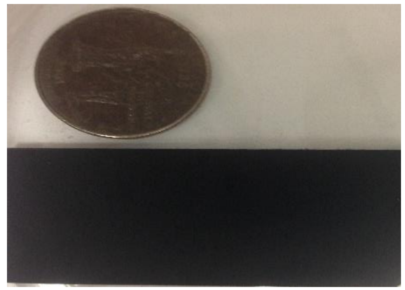
<u>RELEVANCE TO COMMERCIAL SPACE INDUSTRY</u>

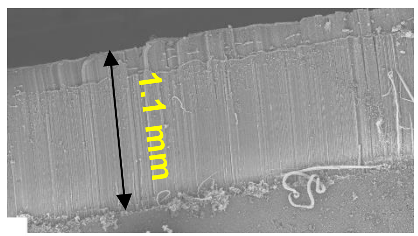
Ultra-high temperature, light weight, highly anisotropic thermal properties and cost effective thermal protection systems (TPS) are enabling technologies for viable commercial space transportation vehicles and their high-temperature systems.

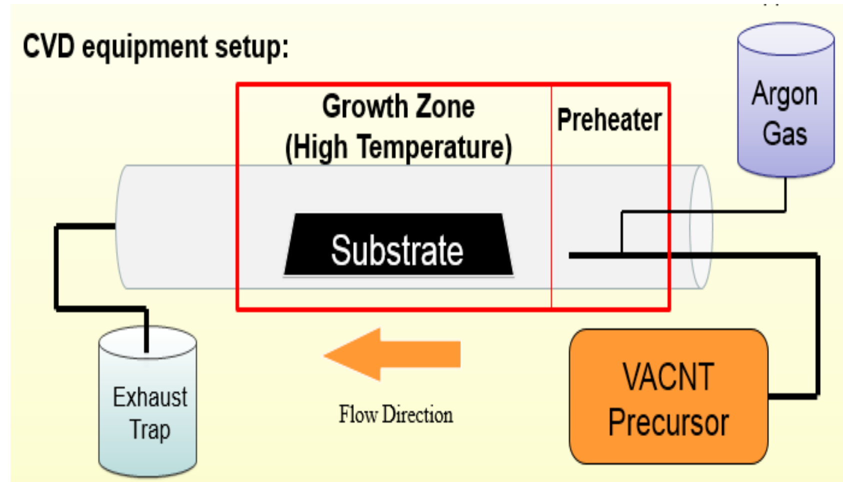
Research Methodology

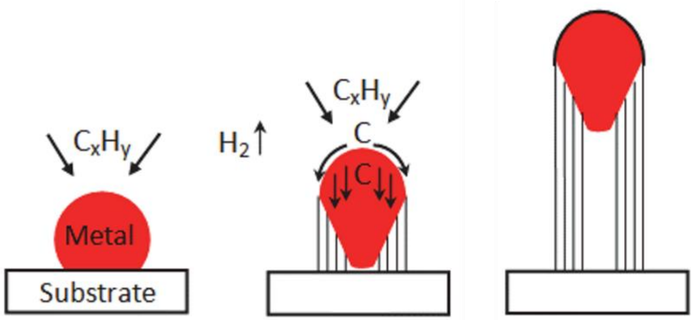
Vertically Aligned Carbon Nanotubes (VACNT)

- Grown by Chemical Vapor Deposition (CVD)
- Highly anisotropic properties









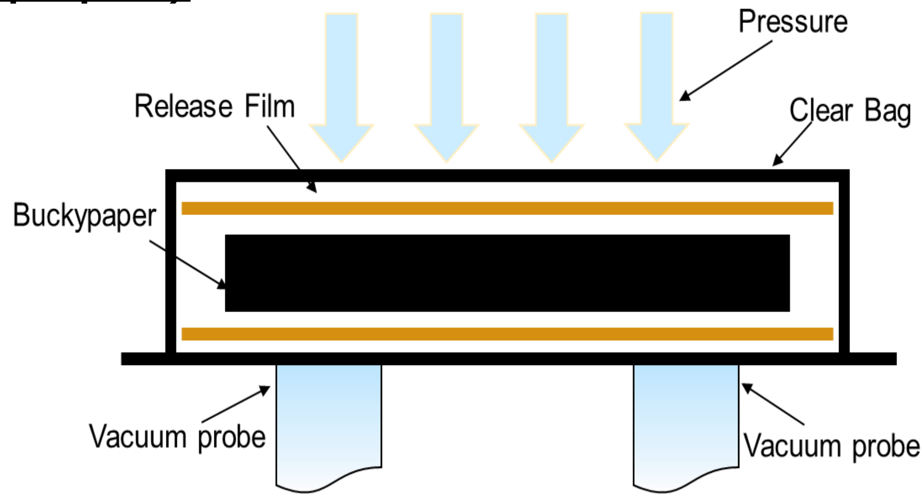
Chemical Vapor Deposition (CVD) Mechanism

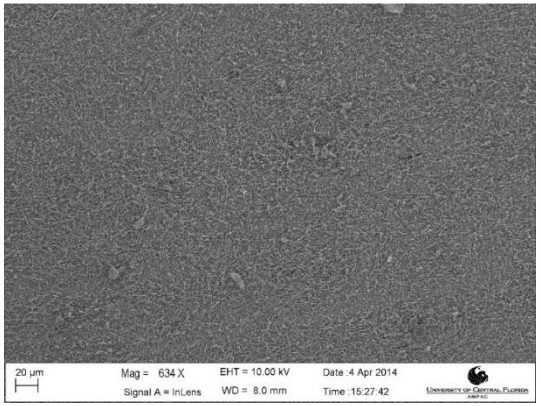
Tube Furnace

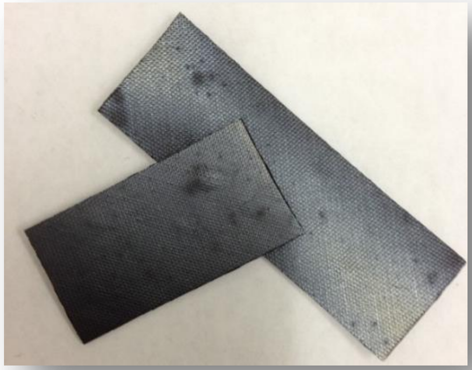
Research Methodology

Carbon Nanotube Paper (Buckypaper)

- Manufactured by using the pressure-assisted infiltration process
- Carbon nanotubes are aligned in the plane





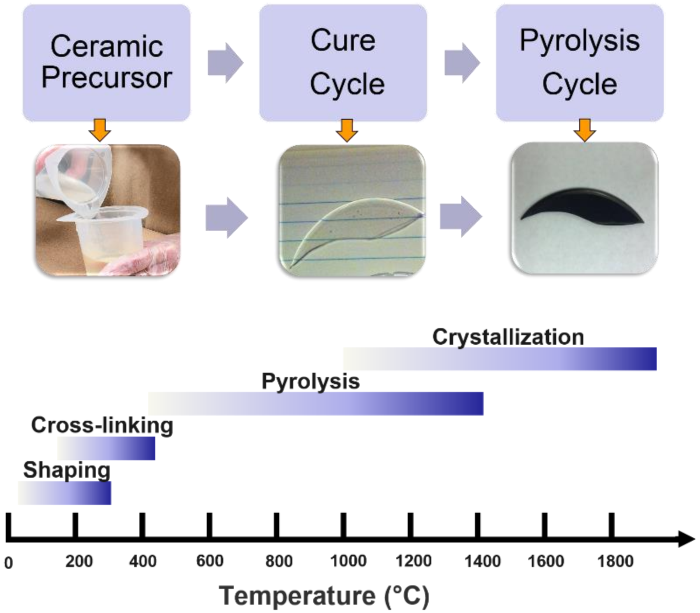


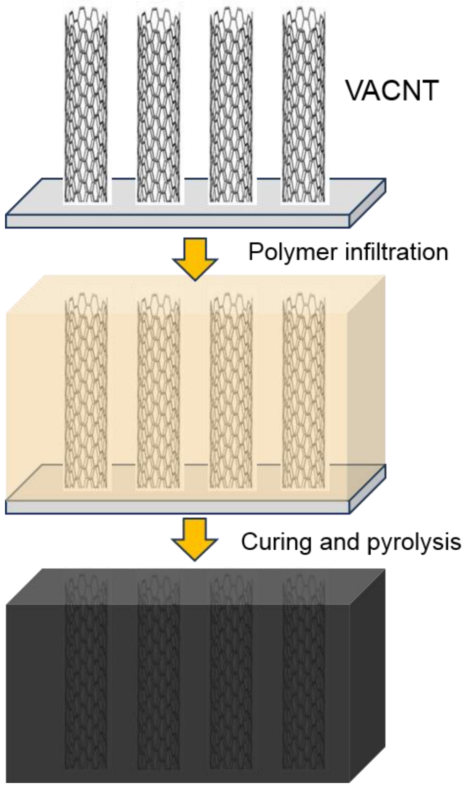
Buckypaper + PDC matrix

Autoclave

Research Methodology

Polymer Infiltration Process(PIP)





Kiln

VACNTs + PDC matrix

- Near net shape manufacturing
- Outstanding thermochemical stability
- Lower pyrolysis temperature, lower energy consuming

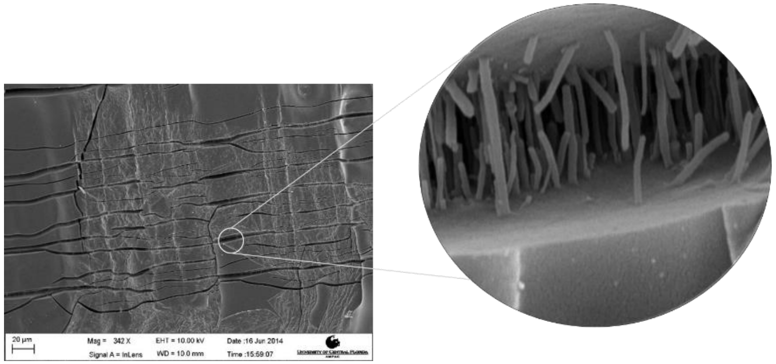
Results

Electrical conductivity in two directions

Sample	Conductivity Through-thickness σ (S/m)	Conductivity In-plane σ (S/m)
Buckypaper/PDC	~ 0	0.161
VACNT/PDC	80.1	~ 0

Hardness

Samples	Hardness (GPa)
Buckypaper/PDC	2.5
VACNT/PDC	1.85
Pure PDC	8.5



- Alignment in the nanocomposite
- VACNTs have a good bonding with PDCs

Conclusions

- Vertically aligned carbon nanotube (VACNT) forest was successfully grown with good quality.

- The VACNT/PDC ceramic composite was fabricated through curing and pyrolysis procedures.

- The highly anisotropic conductivity was observed.

- Scanning electronic microscopic images indicate that small cracks and pores have been developed after the PDC pyrolysis. The fracture toughness of the ceramic composite could be improved.

Future Work

- Resin transfer molding (RTM) process with carbon nanotube preforms will be designed to solve the shrinkage problem in PDC curing and pyrolysis cycles.

- Thermal conductivity in both in-plane and through-thickness directions will be characterized.

- Ground testing of ceramic composites will be conducted with Oxyacetylene Exposure Test, Shock Tube Test and Hot Jet Facilities