Multiscale Characterization

Characterization of Composite Layers
- Graded Ceramic/Metal Matrix Composites
- Polymer Matrix Composites
- Local Strain Fields/Damage Initiation

Interfaces and Bonded Joints
- Thermal Impedance
- Interfacial Delamination

Structural Performance
- Impact Response
- Vibration Analysis

Nancy R. Sottos
University of Illinois
Characterization Scales

- Reinforcement/matrix interface
- Microvascular channels
- Bonded interfaces
- Composite layer properties
- Structural response

**Materials**
- MAX Phase Metallayer (Ti or SMA)
- Oxide ceramic
- Functionally graded metal/ceramic
- Polymer matrix composite

**Function**
- Thermal/Environmental Barrier Coating
- Self-healing of Protective Coating
- Gradual Change in Thermal Expansion
- Thermal Management
- Mechanical Damping
- Load Bearing
- Host Sensors
- Damage Propagation Barrier

**SEM micrograph of 15 µm thick protective Al₂O₃ surface layer formed after 10,000 heating cycles of Ti₃AlC** [Ref]

**SEM of Ti₃AlC (light) + γTiAl (dark) as an example of MAX phase composite** [Ref]
Characterization of Composite Layers

- **Graded Ceramic/Metal Matrix Composites**
  - Ibrahim Karaman (TAMU), Miladin Radovic (TAMU), Ozden Ochoa (TAMU)

- **Polymer Matrix Composites**
  - Scott White (UIUC), Nancy Sottos (UIUC), Zoubeida Ounaies (TAMU)

- **Local Strain Fields/Damage Initiation**
  - Nancy Sottos (UIUC), Scott White (UIUC), Ibrahim Karaman (TAMU)
Structural Characterization of GCMeCs

High Temperature X-Ray Diffractometer (up to 1500K)

Micro CT for non-destructive characterization of metallic and ceramic phases and porosities

Electron Microprobe Analyzer and Wave Dispersive Spectroscopy (WDS) to study compositional variations across interfaces

Hermetic, beryllium dome high temp heating stage under 6x10^-7 mBar vacuum

SEM and Orientation Imaging Microscopy (OIM) for phase morphology, distribution, and texture
Thermomechanical Characterization of GCMeCs

Mechanical Testing (up to 1700 °C)

Elastic and Shear Moduli
Resonant Ultrasound Spectroscopy
(up to 1300 °C)

Fracture Toughness
High Temp Double Torsion

Ti$_3$SiC$_2$ cycling loading at 25 °C

Stress (MPa)
0 200 400 600 800 1000 1200 1400 1600
Strain
0 0.001 0.002 0.003 0.004 0.005 0.006

Young's modulus, GPa

Temperature, K
200 400 600 800 1000 1200 1400 1600

da/dt, mm/sec

K$_{IC}$, MPam$^{1/2}$
PMC Characterization

- A full suite of experimental techniques will be used to aid manufacturing development studies and mechanical/thermal performance assessment

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Thermal Performance Studies

- Camera FOV
- Resistive Heater
- Fin Specimen
- Heated Copper Stage
- Translation Stage

DeltaTherm 1560 infrared camera
(256 x 320 pixels Indium Antimonide infrared detectors)

Active Cooling
Deformation/Strain Measurements

Digital Image Correlation (DIC)

Can control resolution through speckle pattern:
- Macroscale: ±25 μm black spray paint
- Microscale: ±1 μm air-brushed pattern
- Nanoscale: ±10 nm fluorescent nanoparticles
Localized Strain Measurements

Strain concentrations due to vascular networks for active cooling

SEM of nanoparticles on sample surface

$\mu$VAC cross-section exposing channels
Micro and Nano X-Ray Tomography

MicroCT reveals material architecture

Lens based x-ray microscopy

FEA models will be based on the microscopy and micro-CT observations of functionally gradient surfaces.

AFOSR-MURI
Functionally Graded Hybrid Composites
Interfaces and Bonded Joints

- **Thermal Impedance**
  - Khalid Lafdi (UDRI)

- **Interfacial Fracture**
  - Zoubeida Ounaies (TAMU), Ozden Ochoa (TAMU)

- **Dynamic Shear Strength**
  - Nakhiah Goulbourne (UM/VT)
**Thermal Interface Materials**

- **TIM** are high thermal conductivity materials to fill or eliminate air gaps.

- Requirements of TIM: conform to mating surface, good wettability, provide high conductivity path.

- Examples of TIM: greases, reactive compounds, elastomers and pressure sensitive adhesive films.

Nanotube fuzzy fibers as TIM
Thermal Impedance to Characterize Interfaces

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ASTM D5470 Thermal Interface Material Testing System
Delamination Resistant Interfaces

I. Benchmark by testing sample coupons of TiGr/Ti and TiGr/PMC

II. Evaluate GCMeC/PMC interface:

- vertical nanocolumns followed by resin infusion
- bonding of metal to an intermediate fabric preform using vertical columns grown on both surfaces, with subsequent infusion of resin
- using Z-pinning technology

Carbon nanotube grown on a Nickel wire using CVD. The inset shows a magnified image of the forest.
Quasi-static Interfacial Fracture Testing

• Assess interfacial integrity of GCMeC/PMC through the double cantilever beam (DCB) test for pure Mode-I loading and end notch flexure (ENF) test for pure Mode-II loading from room temperature to 1000 °C.

• Utilize SEM at different stages of the bending experiments to qualify the crack opening profile.

• Combined mesoscale characterization (SEM) and macro-scale characterization will provide insight into the fracture process and qualify interfacial delamination for the three joint concept.
Dynamic Shear Strength of Bonded Interface

- Determine the dynamic shear strength of interlayer materials and joining mechanisms used in the assembly of hybrid composites.
- Determine the static and dynamic adhesive shear strength between functional layers will be determined as a function of component properties and process conditions.
Structural Performance

- Impact Response
  - Nakhiah Goulbourne (UM/VT)

- Vibration Analysis
  - Dan Inman (VT)
Impact Response Characterization

Effect of microstructure and functionalized assembly

- Quasi-static, drop tower, and instrumented gas gun experiments
- Damage mechanisms and dominant failure modes will be identified and the potential for crack-arrest functionality of the component layers will be verified.
- Interactions at the interfaces will be of primary importance.

High velocity
Dependence of Mechanical Properties on Strain Rate

- Mechanical properties of the hybrid composites will be determined as a function of strain rate and a suitable constitutive formulation for the stress will be developed.
- For monolithic polymer components, correlations between microstructure and strain rate will be determined.
- For hybrid composites, correlations between joining technologies and strain rate dependence are sought.
Vibration Analysis

- State of the art vibration measurement systems will be used to measure time responses of FGHC macro samples.
- Frequency response functions and time domain methods will then be used to determine the global modulus and damping parameters of the samples.
- These experiments will be designed to verify and/or modify the results of the multiscale modeling effort across various temperatures and pressures of flight.

VT altitude chamber capable of vibration measurements from -70°C to 170°C at pressures across the range of flight from sea level to 100,000 ft.
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