TESTING OF CERAMIC MATRIX COMPOSITES
Challenges, Tools, Pitfalls, and Opportunities

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Outline

- Introduction
  -- CMC Mechanical Properties
  -- CMCs in Turbines and Nuclear Power

- CMC Material Challenges
  -- Compared to Ceramic Monoliths
  -- Compared to Polymer Matrix Composites

- Current ASTM C28 CMC Test Standards
  -- Testing Objectives and Requirements
  -- ASTM C28 CMC Tests

- Testing Issues in CMCs
- Need/Opportunities for New CMC Test Standards
What is a Ceramic Matrix Composite?

Fiber Reinforced Ceramic Matrix Composite – a ceramic matrix composite in which the reinforcing phase consists of a continuous fiber, continuous yarn, or a woven fabric.

These components are combined on a macroscale to form a useful engineering material possessing certain properties or behavior not possessed by the individual constituents. (ASTM C1275)

Melt Infiltrated SiC-SiC Composite (GE)  
N610-Alumina-Silica Composite (Zawada, USAF)

High Strain Failure

CMCs exhibit improved strain (~0.5%) damage tolerant failure from a cumulative damage process, unlike monolithic advanced ceramics that fracture catastrophically with low strain from a single dominant flaw.

Tensile Stress-Strain Diagrams for Two CMCs

High Density, High Modulus MI SiC Matrix w/ Fiber Interface Coating on Nicalon (WIC)  
Porous, Low Modulus Al-Si-O Matrix No Fiber Coating on Nextel 720 (WMC)
Ceramic Matrix Composites for Turbines

**CMC Benefits --**

High Temperatures and Lower Density and No Cooling Compared to Metals → Fuel Efficiency

Damage Tolerance Compared to Ceramic Monoliths

**CMC Applications in Turbines --**

Exhaust Components → Shrouds/Seals → Combustors → Stators/Vanes → Rotors/Blades

Recent CMC Progress for Turbines

**Oxide-Oxide CMC Nozzle and Center Body**

90" Long, 40" Ø,

FAA CLEEN Program -- Boeing, ATK-COIC, AEC, Rolls Royce

Courtesy of FAA Cleen Program and Boeing
Recent CMC Progress for Turbines

Oxide-Oxide CMC
Mixer, Center Body, and Engine Core Cowl
on the GE Passport 20 engine
16,500 # thrust

MI SiC-SiC CMC
High Pressure Shrouds
in GE-CFM LEAP Engine
30,000 # thrust

CMC Challenges -- Design Methodology, Cost, Reliability, HT Life, QA / NDE

CMCs for Nuclear LW Reactors

SiC-SiC CMC
Channels and Other Structures

SiC-SiC CMC
Based Fuel Cladding

BWR
Boiling Water Reactor

PWR
Pressurized Water Reactor

Channel, Zircaloy-2
Fuel rods
Zr alloy cladding containing UC₂ pellets

CMC Challenges -- Cost, Reliability, HT Life, Design Methodology, QA / NDE
We Need Material Design Data

An anisotropic composite consisting of ceramic fibers in a ceramic matrix with porosity and coatings (sometimes).

Designers Need Material Design Data

What does the designer need for his FEA and life models?

- Model the stress-strain relationships
  Elastic Stress-strain-- Modulus (Tension, Compression, Shear).
  Poisson's ratio
  Pseudo-ductile - \( f(\sigma, \varepsilon) \)

- Design limit stress and strains
  Prop. Limit stress and strain, Ultimate Stress and Strain

- Model the thermal load-temperature-thermal strain relationships
  Thermal Conductivity, Specific Heat, C-Thermal Exp.,

- Model stress concentration effects from features and flaws
  Feature/ flaw geometry and size effects

- Predict life and reliability
  Temperature limits
  Temp-time-stress effects --Creep, fatigue, material degradation, flaw growth
  Oxidation, corrosion, environmental effects
  Damage and Failure Mechanisms

Complication -- Consider fiber architecture and component anisotropy on directional properties
What does the production engineer need for product acceptance?

- Product and component tests to verify quality, condition, and properties
  - Mechanical data
  - Thermal data
  - Physical data density, tolerances -- form, fit, features, finish
  - NDE acceptance for flaws and defects

Complication -- Consider fiber architecture and component anisotropy on directional properties

What do we want from our mechanical testing?

Test data that is accurate and repeatable and statistically valid for
  - material development,
  - material acceptance,
  - QA and proof testing,
  - reliability and life assessment

Testing has to produce data that distinguishes between material variation and experimental variation.

And that means --

“Control and measure ALL the experimental variables and the material variables”

Designers Need High Confidence, Statistically Proven Data
QUESTION -- Why can’t we just use the SAME MECHANICAL TESTS that we use for ceramic monoliths and polymer matrix composite?

Because CMCs are different in their constituents and their properties. They differ in their stress-strain response and fail by different mechanisms than either ceramic monoliths or PMCs.

The temperature regimes for CMCs are much higher than for PMCs.

Tests have to be set up for the challenges of higher (~500-1500°C) temperatures.

<table>
<thead>
<tr>
<th>Property</th>
<th>Ceramic Composite</th>
<th>Ceramic Monolith</th>
</tr>
</thead>
<tbody>
<tr>
<td>Failure Mode</td>
<td>Pseudo-ductile Failure with improved-strain, moderate strength.</td>
<td>Elastic Low strain Brittle Failure, High strength</td>
</tr>
<tr>
<td>Critical Flaws</td>
<td>Flaw tolerant</td>
<td>Very flaw sensitive</td>
</tr>
<tr>
<td>Failure Mechanisms</td>
<td>Matrix failure → fiber damage → fiber failure</td>
<td>Brittle crack propagation in tension</td>
</tr>
<tr>
<td>Homogeneous??</td>
<td>Multiple components (fiber, matrix, interface coatings(?), porosity) combined at</td>
<td>One or two fine-grain components</td>
</tr>
<tr>
<td></td>
<td>the macro level</td>
<td></td>
</tr>
<tr>
<td>Directional Properties?</td>
<td>strong directional effects with fiber architecture and matrix vs fiber loading.</td>
<td>Generally Isotropic</td>
</tr>
<tr>
<td>Temperature HOT!!! 500C-1500C</td>
<td>Multiple Thermal/Oxidation Effects on Fibers, Matrix, Coatings</td>
<td>Thermal/Oxidation Effects on Material</td>
</tr>
</tbody>
</table>
CMCs versus Polymer Composites

<table>
<thead>
<tr>
<th>Property</th>
<th>Ceramic Matrix Composite</th>
<th>Polymer Matrix Composite</th>
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<tbody>
<tr>
<td>Component Properties</td>
<td>Brittle, Cracked/ing Matrix w/ High Strength Fibers</td>
<td>Low Modulus, High Strain Matrix w/ High Strength Fibers</td>
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<tr>
<td>In-Plane 0° Failure</td>
<td>WIF -- Matrix failure → fiber failure</td>
<td>Fiber Damage → Fiber Failure</td>
</tr>
<tr>
<td>Mechanisms</td>
<td>WMF - fiber damage → fiber failure</td>
<td></td>
</tr>
<tr>
<td>Homogeneous ??</td>
<td>NO -- Multiple components (fiber, ceramic matrix, porosity/</td>
<td>NO -- Two components – fiber and resin</td>
</tr>
<tr>
<td></td>
<td>cracks, interface coatings)</td>
<td></td>
</tr>
<tr>
<td>Directional Properties?</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>Temp. Regime</td>
<td>500C up to 1500C</td>
<td>Up to ~300C</td>
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</tbody>
</table>

NASA SiC-SiC Combustor Liner

Japanese SiC-SiC Rocket Combustion Chamber

QUESTION – What do we need in our “perfect” mechanical tests?

- Accurate force application and measurement
- Accurate strain measurement in the gage section.
- Consistent failure in the gage section
  - Uniform strain and/or stress in the gage
  - Maximum Stress in the gage
  - Uniform temperature in the gage
  - No uncontrolled stress concentrations or secondary stresses
- NDE and failure analysis to assess material variation
ASTM Standard Test Method -- a concise description of an orderly procedure for
determining a property or constituent of a material, an assembly of materials, or a
product.

- Includes all the essential details as to apparatus, test specimen, procedure, and
calculations, and reporting needed to achieve satisfactory precision and bias.
- Represents a consensus as to the best currently available test procedure for the use
intended
- Supported by experience and adequate data obtained from cooperative tests.

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<th>General Outline</th>
<th>Test Specimens, Sampling</th>
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<td>Preparation of Apparatus</td>
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<td>Conditioning</td>
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<tr>
<td>Summary of Test Method</td>
<td>Procedure</td>
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<tr>
<td>Significance and use</td>
<td>Calculation or Interpretation of Results</td>
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<tr>
<td>Interferences</td>
<td>Report Requirements</td>
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<tr>
<td>Apparatus</td>
<td>Precision and Bias</td>
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<tr>
<td>Reagents and Materials</td>
<td>Keywords</td>
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<tr>
<td>Hazards</td>
<td></td>
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</table>

What Does a Test Standard Tell you

What Happens without Standards?

AT BEST!!!
- Confusion over terminology, test procedures, protocols, and data requirements.
- Conflicting and/or incomplete data comparison
- Multiple test requirements from multiple users and developers.

AT WORST!!!
- BAD TESTS with uncontrolled variables giving invalid, unreliable, and incomplete test data.
CMC Tensile Tests

C1275 / C1359 – RT and HT Tensile Strength of CMC Flat Specimens
C1773 - RT Tensile Strength of CMC Tubes (New)

- Flat Specimen Geometries
  - Straight Side with tabs
  - Edge Loaded - Reduced Gage Section
  - Face Loaded - Reduced Gage Section
  - Pin Loaded - Reduced Gage Section
- Key Experimental Variables
  - Test Rate (Creep, Degradation, and Damage)
  - Size Effects and Gage Section Geometry
  - Surface Condition
- Interferences and Challenges
  - Out-of-Gage Failure
    - Alignment and Bending Stresses
    - Grip Stresses
  - HT Strain Measurement

CMC Durability Tests

ASTM C1337 – HT Tensile Creep and Creep Rupture of CMC Flat Specimens
ASTM C1360 - RT Tensile Fatigue CMC Flat Specimens

- Flat Specimen Geometries
  - Same as Tensile Tests
- Key Experimental Variables
  - Test Rate (Creep, Degradation, and Damage)
  - Size Effects and Gage Section Geometry
  - Surface Condition
  - Controlled and Uniform Temperature
  - Atmosphere Monitoring and Control
  - R ratio for fatigue
- Interferences and Challenges
  - High Temperature Gripping
  - Out-of-Gage Failure
  - Alignment, Bending, and Grip Stresses
  - HT Strain Measurement
**CMC Shear Tests**

**ASTM C1292 and C1425 -- RT and HT Shear – CMC Flat Specimens**

- Specimen Geometries
  - Double Notch Shear (Interlaminar)
  - Iosepescu (In Plane)
- Key Variables
  - Notch Geometry and Specimen Size
- Interferences and Challenges
  - Notch Stress Concentrations
  - Alignment and Mixed Mode Stresses
  - No Easy Strain Measurement
  - Fixture Limits on HT testing

**CMC Transthickness Tensile Test**

**ASTM C1468 -- RT Transthickness Tensile – CMC Flat Specimens**

- Specimen Geometries
  - Round and Square

- Key Variables
  - Specimen Size

- Interferences and Challenges
  - Epoxy Failure
  - Epoxy Limits the HT testing
  - Edge Effects
  - Bending Stresses
  - No Easy Strain Measurement
CMC Compression Tests

**ASTM C1358 -- RT Compression -- CMC Flat Specimens**

**Specimen Geometries**
- Straight with Tabs
- Tapered Dog-Bone

**Key Variables**
- Specimen Size and Flatness
- Alignment and Bending Stresses
- Specimen Anti-Buckling Fixture

**Interferences and Challenges**
- Out of Gage Failures
- Buckling Failure

CMC Flexure Tests

**ASTM C1341 -- RT and HT Flexure -- CMC Bars**

**Specimen and Loading Geometries**
- 3-Point and 4-Point Loading
- Semi and Fully Articulating Fixtures

**Key Variables**
- Flatness and Twist in Specimens
- Specimen Size and Geometry
- Span-to-Depth Ratio

**Interferences and Challenges**
- Bending Stress vs True Tensile Face Stress (Elastic Beam Equations)
- Size Effects
- Invalid Failures (Shear, O-Gage, Contact)
- Mixed Mode Stresses and Surface Damage
- Temperature Variation in the Gage
**ASTM C 1557 - RT Tensile Strength and Young’s Modulus of Fibers**

**Specimen Geometries**
- Single Filament in Paper Tabs

**Key Variables**
- Specimen Length
- Strain by Flags or Calculation

**Interferences and Challenges**
- Alignment and Bending Stresses
- Test Rate and SC Growth from Moisture
- Surface Damage
- Measurement of Filament Cross-Section Area

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**Other Possible C28 Test Standards**

**MODULUS AND ULTRASONIC TESTING**
- **C1198** Dynamic Young’s Modulus, Shear Modulus, and Poisson’s Ratio for Advanced Ceramics by Sonic Resonance
- **C1259** Dynamic Young’s Modulus, Shear Modulus, and Poisson’s Ratio for Advanced Ceramics by Impulse Excitation of Vibrations

- **C1331** Ultrasonic Velocity in Advanced Ceramics with the Broadband Pulse Echo Cross Correlation Method
- **C1332** Ultrasonic Attenuation Coefficients of Advanced Ceramics by the Pulse Echo Contact Technique

**CMC Issue – Anisotropic Properties and Heterogenous Structure**
Other Possible C28 Test Standards

MECHANICAL TESTS AND DATA ANALYSIS (Monolithic)

- C 1421 Fracture Toughness of Ceramics (SENB)

CMC Issue – $K_{IC}$ versus Crack Growth Resistance (Strain Energy Release Rate)

- C1239 Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics
- C1683 Size Scaling of Tensile Strengths Using Weibull Statistics for Advanced Ceramics

DURABILITY TESTS (Monolithic)

- C1368 Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Rate Flexural Testing at Ambient Temperature
- C1465 Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Rate Flexural Testing at Elevated Temperature
- C1576 Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Flexural Testing (Stress Rupture) at Ambient Temperature
- C1525 Thermal Shock Resistance for Advanced Ceramics by Water Quenching
THERMAL AND PHYSICAL TESTS (Monolithic)

- **C1470** Guide for Testing the **Thermal Properties** of Advanced Ceramics
  
<table>
<thead>
<tr>
<th>Coef. Thermal Expansion</th>
<th>Thermal Diffusivity/Conductivity</th>
<th>Specific Heat</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1" alt="Schematic of Thermal Diffusivity Method" /></td>
<td><img src="image2" alt="Schematic of Flash Diffusivity Apparatus" /></td>
<td><img src="image3" alt="Schematic of Differential Scanning Calorimeter" /></td>
</tr>
</tbody>
</table>

- **C1274** -- **Specific Surface Area by Physical Adsorption**
- **C1039** – **Density and Porosity by Archimedes Method**
- **D4284** - **Mercury Porosimetry**

CMC Testing Challenges

**CMC Material and Challenges**

- **Anisotropic Properties and Heterogeneous Structure**
- **Material Variability** (Structure and Defects)
  - In-specimen, between specimen, lot-to-lot
  - Internal Porosity, Flaws, and Defects
  - NDE of Test Specimens
- **Specimen Size Effects**
  (Architecture and Processing Effects)
- **Hole, Notch, and Edge Stresses**
- **Surface Flaws from Spec Prep**
- **Temperature, Strain Rate, and Atmosphere Effects** (Creep and SC Growth)
- **Determining High Temp Failure/Degradation Mechanisms**
  (Fiber, Matrix, Interface?)
  Intermediate Temp. Degradation

**Steam, Temp and Strain Rate Effects on Tensile Strength of Oxide-Oxide Composite (N720, Alumina Matrix)**
CMC Testing Challenges

High Temperature (>800°C) Experimental Challenges

- Temperature Uniformity and Accurate Measurement
- Strain Measurement at HT (Accurate and Representative)
- High Temperature Gripping and Loading Fixtures
  (Alignment/Ext.Stresses, Stability)
- Complex Environment (Combined Stress, Temperature, Exhaust, Atmosphere, Cycles)

→Burner Rig Testing (HiV, HiP, HiT)
  NASA Burner Rig
  6 atm, 200m/s, 2500F

- Thermal Shock Effects versus Thermal Degradation

Precision and Repeatability

Key Question --What are the design limit factors, per the designer’s criteria?
Ultimate stress and strain or proportional limit stress and strain

What is the 95% or 99% reliable stress level?

Do either of these strengths depend on weakest link mechanisms with probabilistic strength distributions, based on the inherent variability in the composite:
  fibers, matrix, porosity, fiber interface coatings, surface seal coats
  fiber architecture, alignment, and anisotropy, inherent surface and volume flaws.

When are Weibull statistics the best way to assess failure probability?
Precision and Repeatability

*Where does the data variability come from??*

**Material Variability occurs**
- spatially within a single test specimen,
- between test specimens,
- and between lots.

**Data variation also develops from experimental variability** –
- test specimen dimensions and volume/size effects,
- extraneous bending stresses, notch effects
- slow crack growth, temperature and humidity effects

The tester and the date user need to know what the source of variability is in a given set of data.

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**Needs for New CMC Test Standards**

**High Temp** CMC Tests
- Interlaminar Tension
- Compression
- Thermal Gradient Stresses
- Fatigue (Tensile and Bending)

**Complex** High Temp Test
- Thermocyclic Tests
- Oxidation and Corrosion (Stressed)
- Thermal Shock Damage
- Burner Rig Tests
Needs for New CMC Test Standards

Level 2 CMC Tests
- Crack Growth Resistance (Work of Fracture??)
- Open Hole and Notch Effects
- Mixed Stress State Tests

Level 3 CMC Tests
- Impact Damage Tolerance
- Wear, Abrasion, Erosion
- Subelement Tests (Tubes, T's, L's)
- Strength of Joints
- Sandwich Structure Tests

A Guide to NDE of CMCs

Summary of CMC Testing

1. Testing of CMCs is not simple, especially at high temperatures, because of the range of material variables, properties, and fracture/damage mechanisms.

2. ASTM C28 has many of the baseline mechanical property test standards.

3. There is a need for more high temperature and Level 2 and Level 3 test standards.

If you and your market need performance-oriented ceramic test standards to stay competitive and move your ceramic technology forward, use C28 standards and join us on the C28 committee!!

www.astm.org/COMMITTEE/C28.htm