TESTING OF CERAMIC MATRIX COMPOSITES

Challenges, Tools, Pitfalls, and Opportunities

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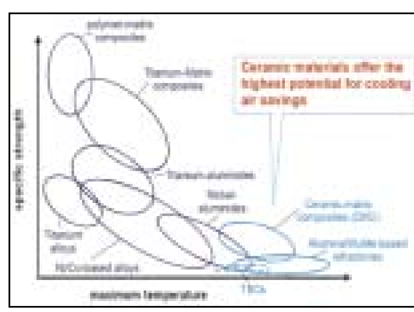
Outline

- **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**

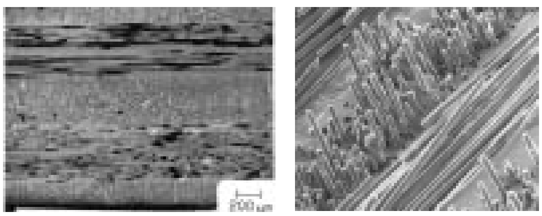


CMC Exhaust Seals in F414 Engine of F/A-18E/F (Super Hornet), The US Navy

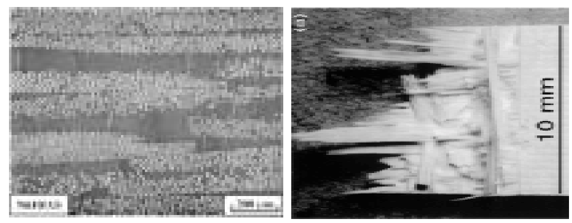
What is a Ceramic Matrix Composite?

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)

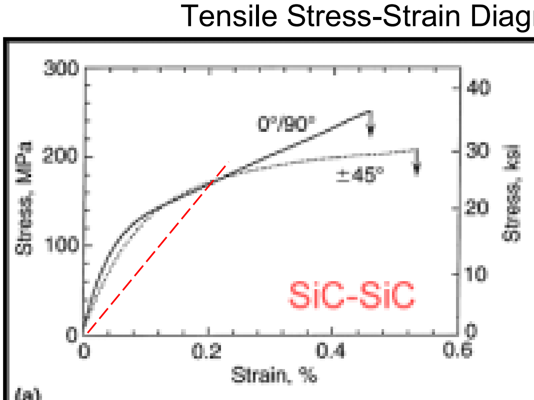


N610-Alumina-Silica Composite (Zawada, USAF)

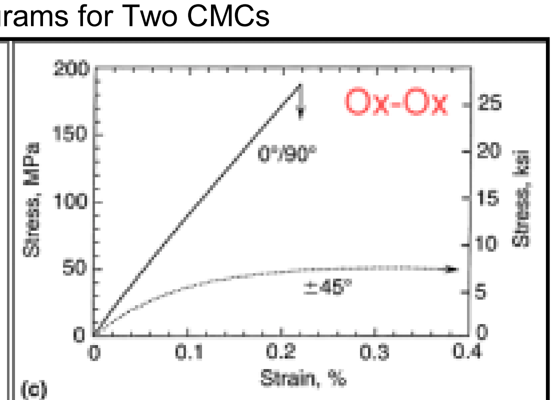
High Strain Failure

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)



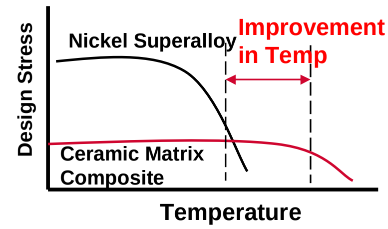
Porous, Low Modulus Al-Si-O Matrix <u>No</u> Fiber Coating on Nextel 720 (WMC)

Ceramic Matrix Composites for Turbines

CMC Benefits --

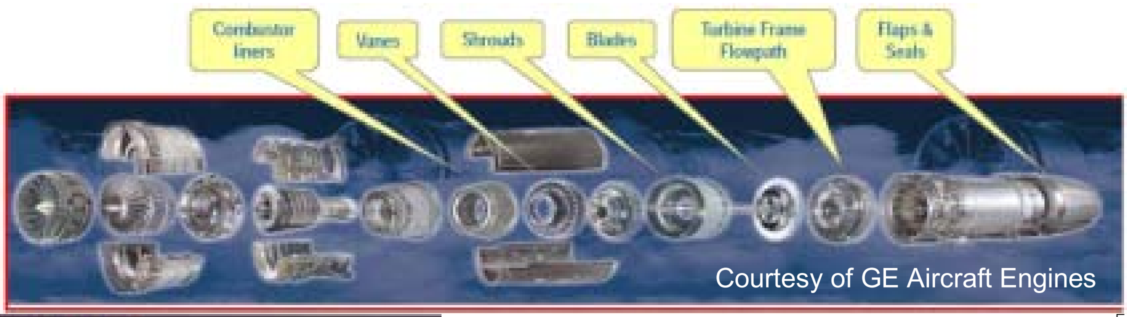
High Temperatures and Lower Density and No Cooling Compared to Metals → Fuel Efficiency

Damage Tolerance Compared to Ceramic Monoliths



CMC Applications in Turbines --

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



Courtesy of GE Aircraft Engines

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

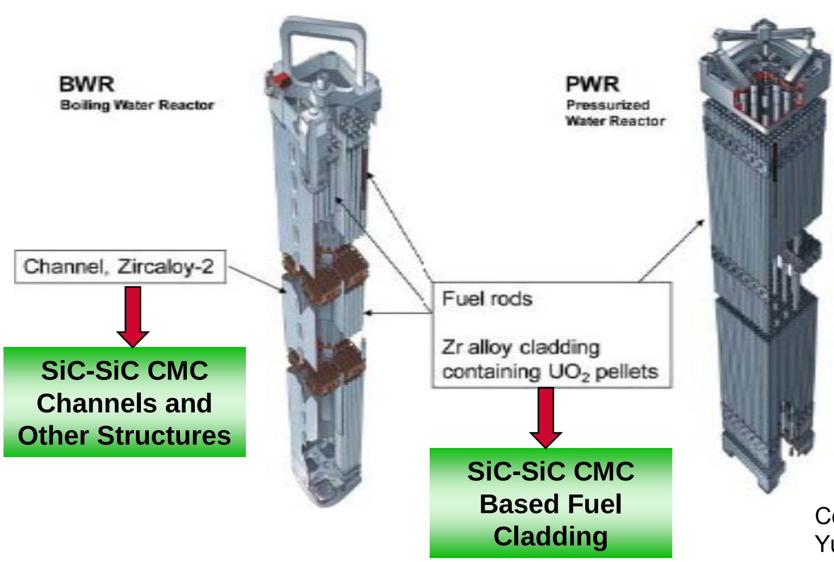
Courtesy of Aviation Week and GE

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



Courtesy of
Yutai Katoh, ORNL

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

We Need Material Design Data

Temperature

Durability over Time and Temp in Variable Stress Conditions–
fatigue, impact, thermomechanical, erosion/wear/abrasion, thermal shock, etc

Life Time

Environmental Durability over Time, Temperature, and Steady State Stress
oxidation, creep/rupture, corrosion, radiation

Corrosion, Radiation)

Abrasion)

Stress Concentration Effects | **Mixed Stress States**

Temperature Effects and Limits

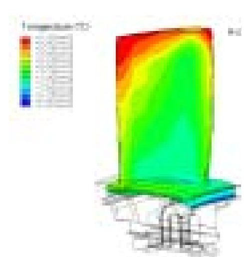
Baseline/RT Mechanical and Physical Properties

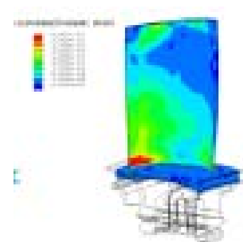
An anisotropic composite consisting of ceramic fibers in a ceramic matrix with porosity and coatings (sometimes).

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 (σ, ε)
- **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

Engineers Need Product Acceptance Data

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

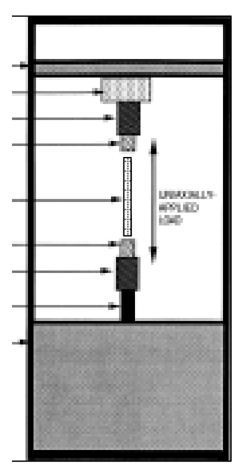
Mechanical Testing

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

design data (FEA, allowables),
product specifications,
failure analysis.



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

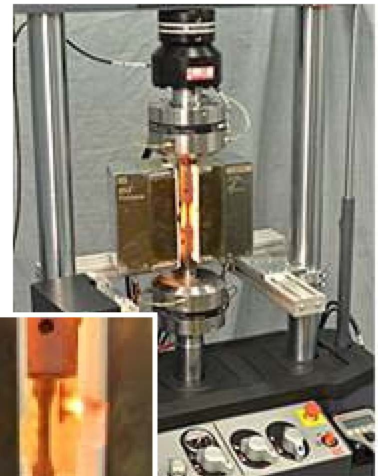
And that means --

"Control and measure ALL the experimental variables and the material variables"

Designers Need High Confidence, Statistically Proven Data

Mechanical Testing

QUESTION -- Why can't we just use the SAME MECHANICAL TESTS that we use for ceramic monoliths and polymer matrix composite?



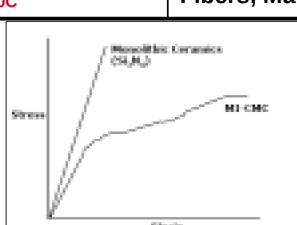
Because CMCs are different in their constituents and their properties. They differ in their stress-strain response and fail by different mechanisms than either ceramic monoliths or PMCs.

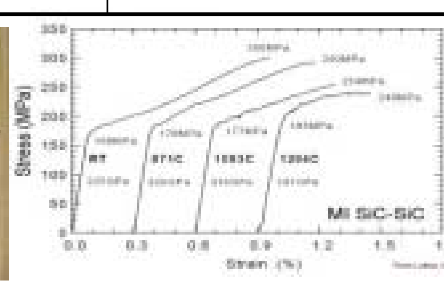
The temperature regimes for CMCs are much higher than for PMCs.

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

CMCs versus Ceramic Monoliths

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





CMCs versus Polymer Composites

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

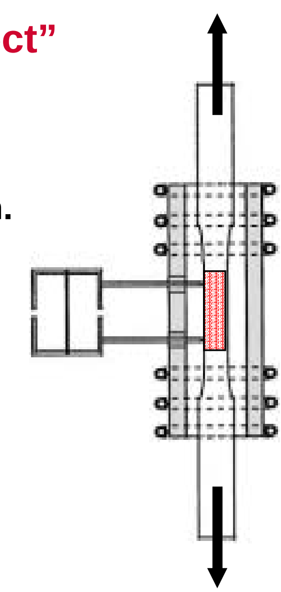
NASA SiC-SiC Combustor Liner

Japanese SiC-SiC Rocket Combustion Chamber

Mechanical Testing

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



What Does a Test Standard Tell you

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

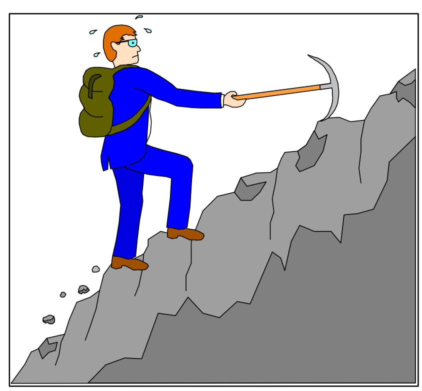
General Outline

Scope
References
Terminology
Summary of Test Method
Significance and use
Inteferences
Apparatus
Reagents and Materials
Hazards
Test Specimens, Sampling
Preparation of Apparatus
Calibration and Standardization
Conditioning
Procedure
Calculation or Interpretation of Results
Report Requirements
Precision and Bias
Keywords

What Happens without Standards?

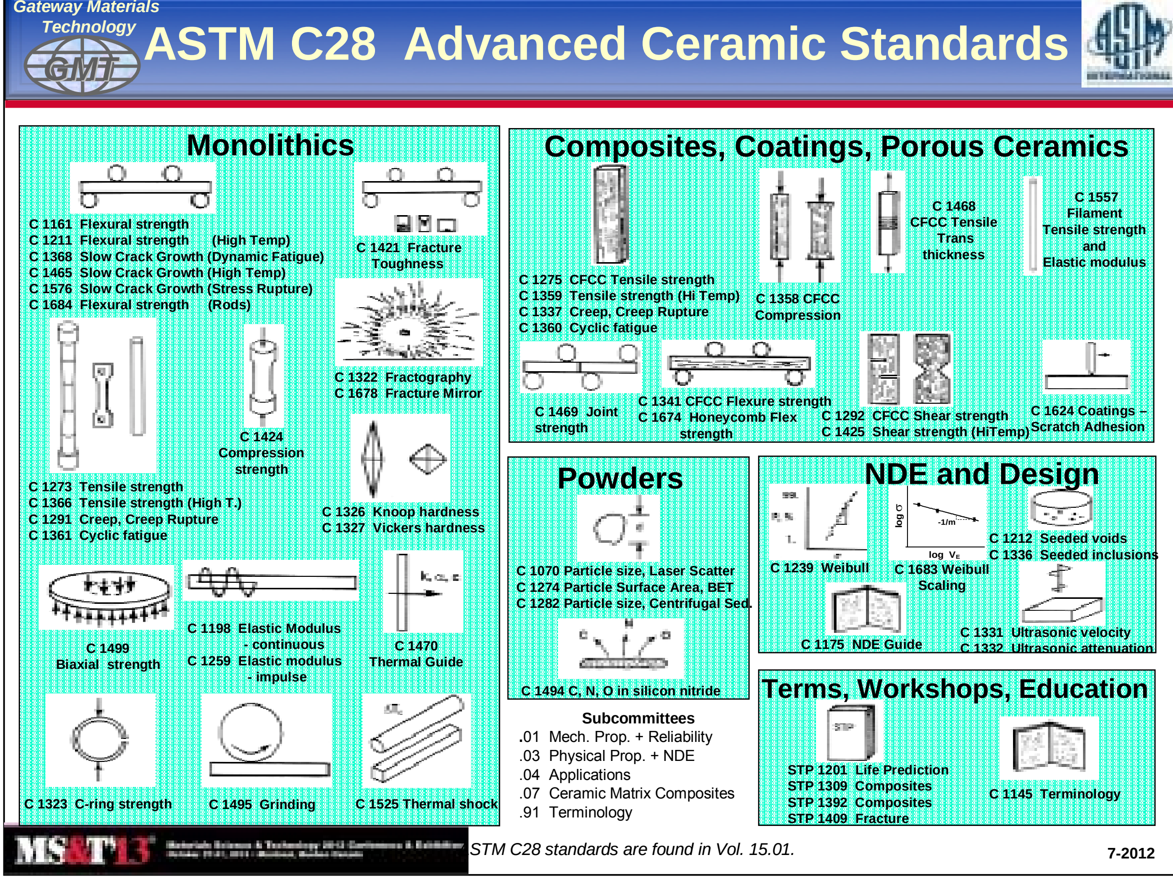
AT BEST!!!

- **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!!!

- **<u>BAD TESTS</u> with uncontrolled variables giving invalid, unreliable, and incomplete test data.**



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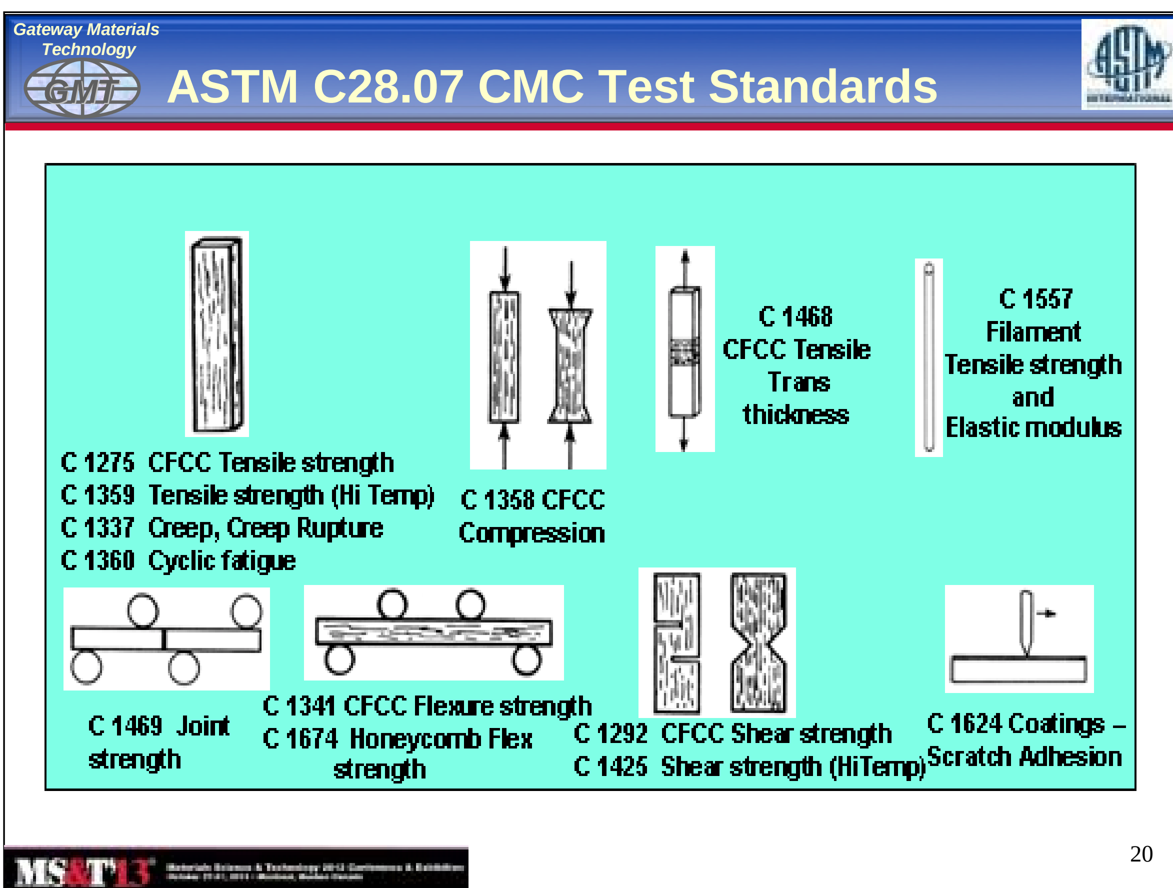
GMT
ASTM C28 Advanced Ceramic Standards
Monolithics
C 1161 Flexural strength
C 1211 Flexural strength (High Temp)
C 1368 Slow Crack Growth (Dynamic Fatigue)
C 1465 Slow Crack Growth (High Temp)
C 1576 Slow Crack Growth (Stress Rupture)
C 1684 Flexural strength (Rods)
C 1421 Fracture Toughness
C 1322 Fractography
C 1678 Fracture Mirror
C 1424 Compression strength
C 1273 Tensile strength
C 1366 Tensile strength (High T.)
C 1291 Creep, Creep Rupture
C 1361 Cyclic fatigue
C 1326 Knoop hardness
C 1327 Vickers hardness
C 1499 Biaxial strength
C 1198 Elastic Modulus - continuous
C 1259 Elastic modulus - impulse
C 1470 Thermal Guide
C 1323 C-ring strength
C 1495 Grinding
C 1525 Thermal shock
Composites, Coatings, Porous Ceramics
C 1275 CFCC Tensile strength
C 1359 Tensile strength (Hi Temp)
C 1337 Creep, Creep Rupture
C 1360 Cyclic fatigue
C 1358 CFCC Compression
C 1468 CFCC Tensile Trans thickness
C 1557 Filament Tensile strength and Elastic modulus
C 1469 Joint strength
C 1341 CFCC Flexure strength
C 1674 Honeycomb Flex strength
C 1292 CFCC Shear strength
C 1425 Shear strength (HiTemp)
C 1624 Coatings – Scratch Adhesion
Powders
C 1070 Particle size, Laser Scatter
C 1274 Particle Surface Area, BET
C 1282 Particle size, Centrifugal Sed.
C 1494 C, N, O in silicon nitride
NDE and Design
C 1239 Weibull
C 1683 Weibull Scaling
log σ
log V
-1/m
C 1212 Seeded voids
C 1336 Seeded inclusions
C 1175 NDE Guide
C 1331 Ultrasonic velocity
C 1332 Ultrasonic attenuation
Subcommittees
.01 Mech. Prop. + Reliability
.03 Physical Prop. + NDE
.04 Applications
.07 Ceramic Matrix Composites
.91 Terminology
Terms, Workshops, Education
STP 1201 Life Prediction
STP 1309 Composites
STP 1392 Composites
STP 1409 Fracture
C 1145 Terminology
MS&T'13
STM C28 standards are found in Vol. 15.01.
7-2012



Gateway Materials Technology
GMT
ASTM C28.07 CMC Test Standards
C 1275 CFCC Tensile strength
C 1359 Tensile strength (Hi Temp)
C 1337 Creep, Creep Rupture
C 1360 Cyclic fatigue
C 1358 CFCC Compression
C 1468 CFCC Tensile Trans thickness
C 1557 Filament Tensile strength and Elastic modulus
C 1469 Joint strength
C 1341 CFCC Flexure strength
C 1674 Honeycomb Flex strength
C 1292 CFCC Shear strength
C 1425 Shear strength (HiTemp)
C 1624 Coatings – Scratch Adhesion
MS&T'13

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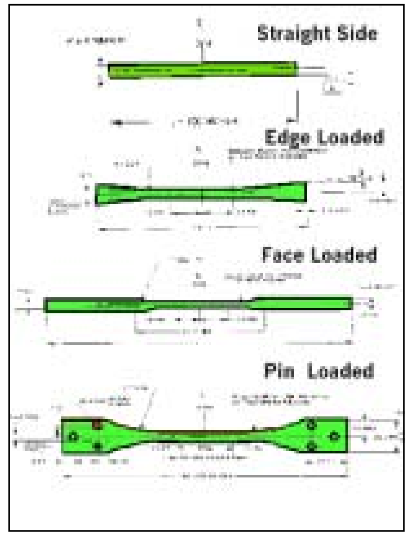
CMC Tensile Tests

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**



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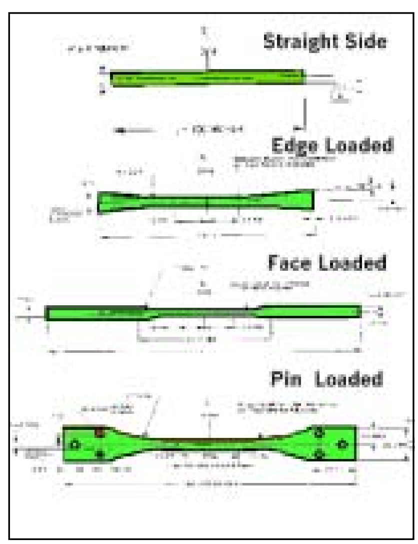
CMC Durability Tests

ASTM C1337 – HT Tensile Creep and Creep Rupture of CMC Flat Specimens

ASTM C1360 - RT Tensile Fatigue CMC Flat Specimens

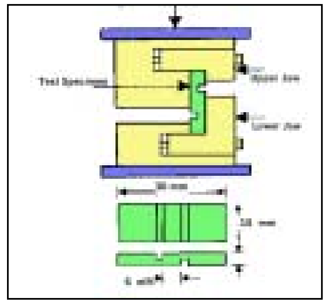
- **Flat Specimen Geometries**
  - **Same as Tensile Tests**
- **Key Experimental Variables**
  - **Test Rate (Creep, Degradation, and Damage)**
  - **Size Effects and Gage Section Geometry**
  - **Surface Condition**
  - **Controlled and Uniform Temperature**
  - **Atmosphere Monitoring and Control**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

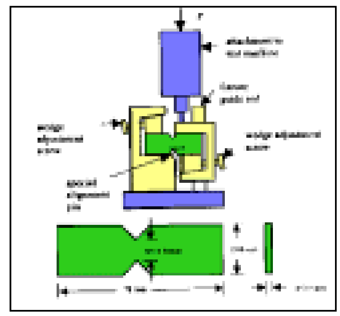


CMC Shear Tests

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

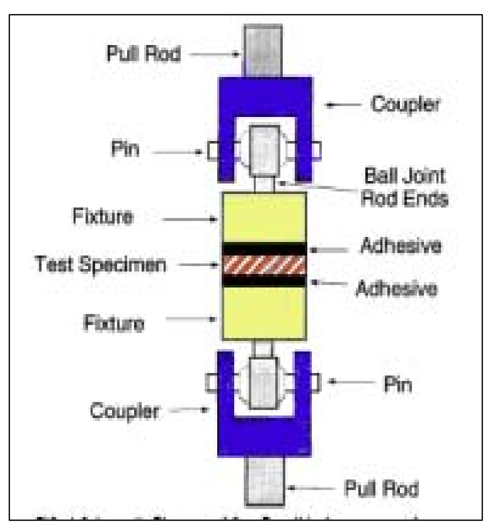
- Round and Square

Key Variables

- Specimen Size

Interferences and Challenges

- Epoxy Failure
- Epoxy Limits the HT testing
- Edge Effects
- Bending Stresses
- No Easy Strain Measurement



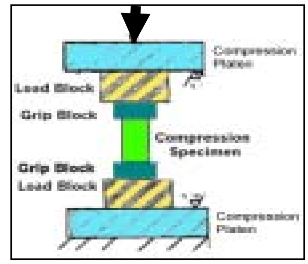
CMC Compression Tests

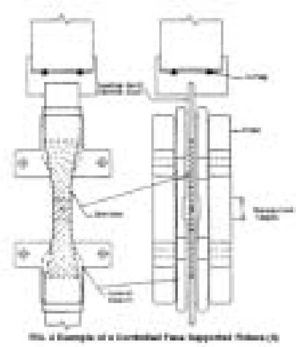
ASTM C1358 -- RT Compression – CMC Flat Specimens

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





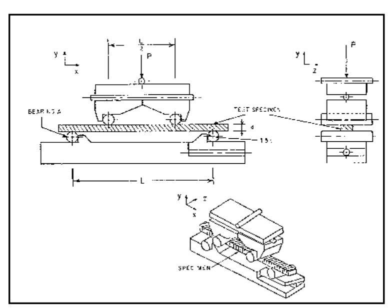
CMC Flexure Tests

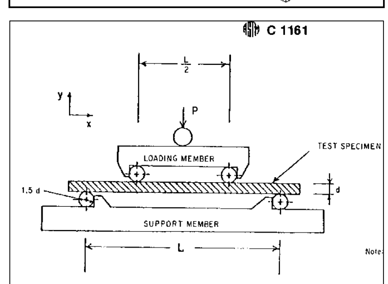
ASTM C1341 -- RT and HT Flexure – CMC Bars

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**





Filament Testing

ASTM C 1557 - RT Tensile Strength and Young's Modulus of Fibers

Specimen Geometries

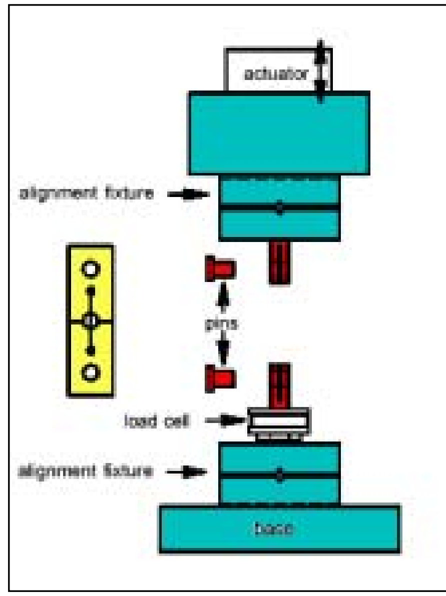
- **Single Filament in Paper Tabs**

Key Variables

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

Interferences and Challenges

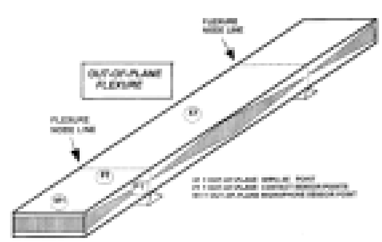
- **Alignment and Bending Stresses**
- **Test Rate and SC Growth from Moisture**
- **Surface Damage**
- **Measurement of Filament Cross-Section Area**



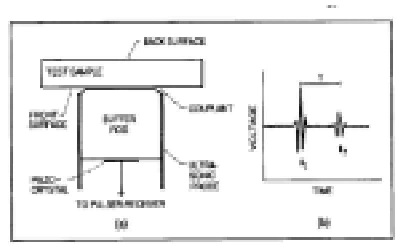
Other Possible C28 Test Standards

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

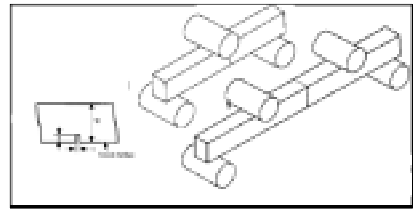


CMC Issue – Anisotropic Properties and Heterogenous Structure

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

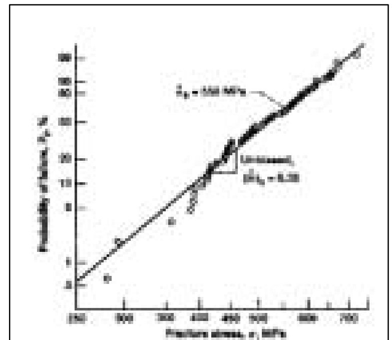


FIG. 1 Example of Weibull Plot of Strength Data

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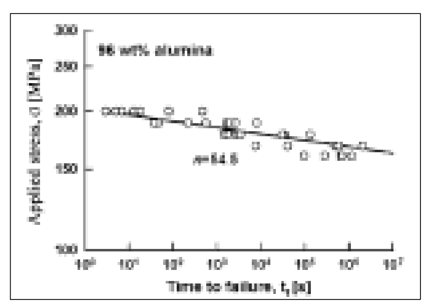
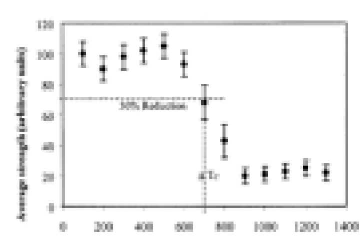
Other Possible C28 Test Standards

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

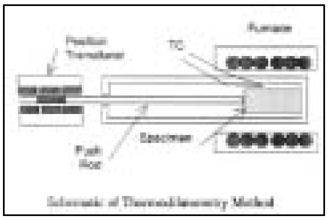


Other Possible C28 Test Standards

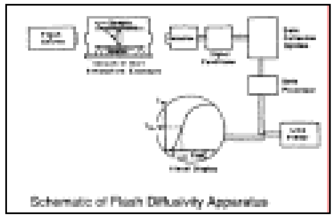
THERMAL AND PHYSICAL TESTS (Monolithic)

- **C1470** Guide for Testing the **Thermal Properties** of Advanced Ceramics

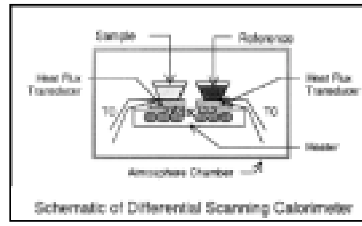
Coef. Thermal Expansion



Thermal Diffusivity/Conductivity



Specific Heat



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

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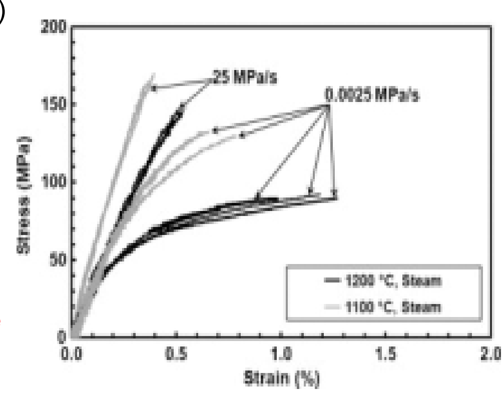
CMC Testing Challenges

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)

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CMC Testing Challenges

High Temperature (>800C) 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)
- Thermal Shock Effects versus Thermal Degradation

NASA Burner Rig
6 atm, 200m/s, 2500F

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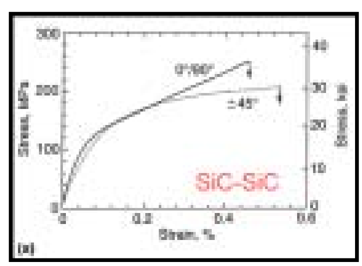
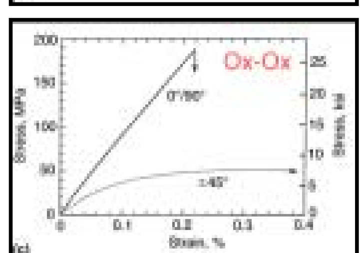
Precision and Repeatability

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?

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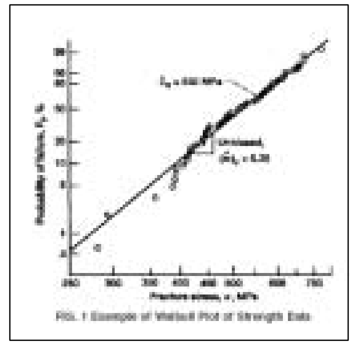
Precision and Repeatability

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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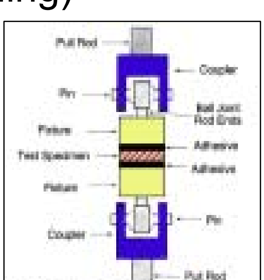
Needs for New CMC Test Standards

High Temp CMC Tests

- Interlaminar Tension
- Compression
- Thermal Gradient Stresses
- Fatigue (Tensile and Bending)



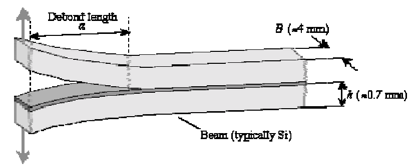
Complex High Temp Test

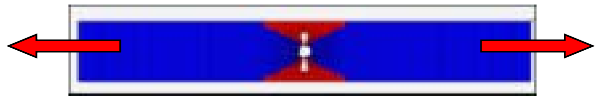
- Thermocyclic Tests
- Oxidation and Corrosion (Stressed)
- Thermal Shock Damage
- Burner Rig Tests

Needs for New CMC Test Standards

Level 2 CMC Tests

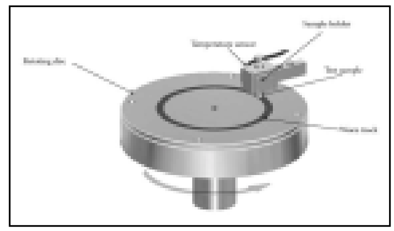
- Crack Growth Resistance (Work of Fracture??)
- Open Hole and Notch Effects
- Mixed Stress State Tests





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*