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# Strength, Fracture Toughness, Fatigue, and Standardization Issues of Free-Standing Thermal Barrier Coatings

Sung R. Choi
Ohio Aerospace Institute, Brook Park, Ohio

Dongming Zhu
U.S. Army Research Laboratory, Glenn Research Center, Cleveland, Ohio

Robert A. Miller
Glenn Research Center, Cleveland, Ohio

July 2003

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This report is a formal draft or working paper, intended to solicit comments and ideas from a technical peer group.

This report contains preliminary findings, subject to revision as analysis proceeds.

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Sung R. Choi, Dongming Zhu, and Robert A. Miller
NASA Glenn Research Center, Cleveland, OH

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## Abstract

**Strength, Fracture Toughness, Fatigue, and Standardization Issues of Free-Standing Plasma-Sprayed Thermal Barrier Coatings**

**Sung R. Choi, Dongming Zhu, and Robert A. Miller**
**NASA Glenn Research Center, Cleveland, OH 44135**

**Strength, fracture toughness and fatigue behavior of free-standing thick thermal barrier coatings of plasma-sprayed $ZrO_2$-8wt% $Y_2O_3$ were determined at ambient and elevated temperatures in an attempt to establish a database for design. Strength, in conjunction with deformation (stress-strain behavior), was evaluated in tension (uniaxial and trans-thickness), compression, and uniaxial and biaxial flexure; fracture toughness was determined in various load conditions including mode I, mode II, and mixed modes I and II; fatigue or slow crack growth behavior was estimated in cyclic tension and dynamic flexure loading. Effect of sintering was quantified through approaches using strength, fracture toughness and modulus (constitutive relations) measurements. Standardization issues on test methodology also was presented with a special regard to material's unique constitutive relations.**

## Contents

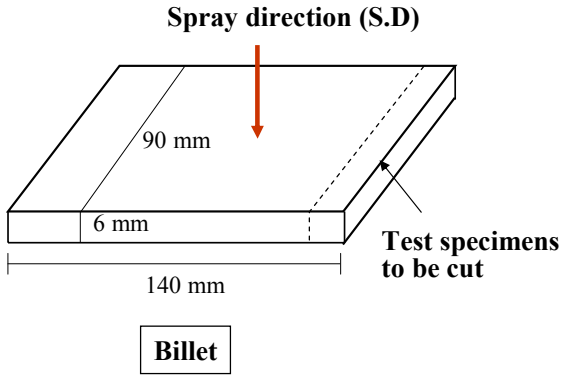

I. Background
II. Processing
III. Strength
IV. Fracture toughness
V. Fatigue/slow crack growth
VI. Deformation
VII. Sintering Effects
VIII. Summary
IX. Bibliography

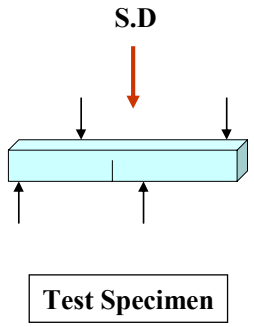

## I. Backgrounds

- Thermal Barrier coatings (TBCs), $ZrO_2$-8 wt% $Y_2O_3$ – important coating materials due to low thermal conductivity, high thermal expansivity, and unique microstructure
- Somewhat anisotropic nature of porosity, microcracks and splat structure - a challenge in routine mechanical testing and data interpretation
- Mechanical testing for TBCs performed to characterize strength, fracture toughness, fatigue, and deformation, and also to establish database
- Results of mechanical testing presented and discussed, and related issues discussed

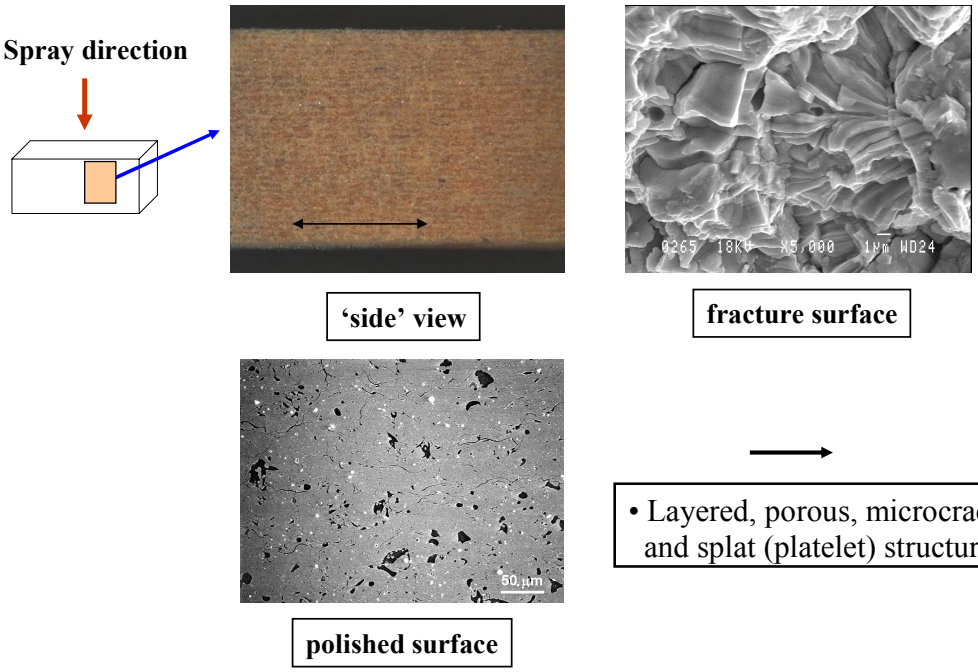
# II. Material Processing

- **$ZrO_2$-8 wt% $Y_2O_3$ powder** with an average particle size of 60 µm
- Plasma sprayed on a steel or graphite substrate
- SULZER-METCO ATC-1 plasma coating system with a 6-axes industrial robot used
- **Free standing** TBC billets fabricated
- Test specimens machined from billets with appropriate configurations
- Typical billets:



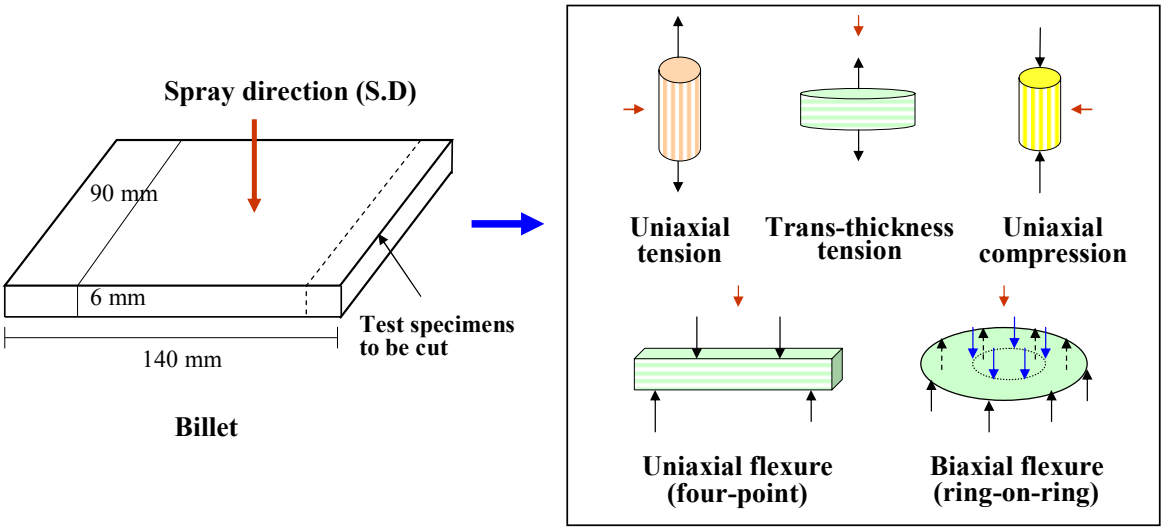

# Unique Microstructure of TBCs




• Layered, porous, microcracked, and splat (platelet) structure

# III. Strength Testing

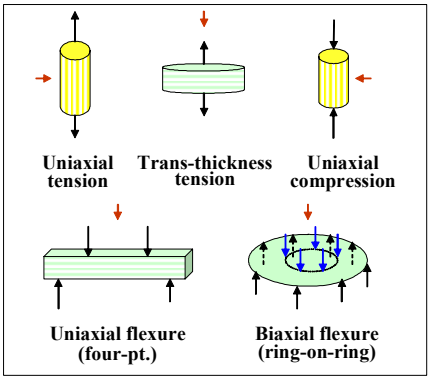
## Types of Testing/Test Specimens/Orientations




↓ indicates spray direction

# Test Matrix (strength)

| Type of tests | Specimen geometry | No. of test specimens | Direction of fracture* |
|---|---|---|---|
| Uniaxial tension | 15mm x 5mm$^{\phi}$ | 10 | P |
| Trans-thickness Tension | 15 mm x 3 mm (t) (diameter x thick.) | 10 | N |
| Uniaxial compression | 10mm x 5mm$^{\phi}$ | 10 | P |
| Uniaxial flexure (four-point) | 3mm x 4mm x 25mm [10/20 mm spans] | 30 | P |
| Biaxial flexure (ring-on-ring) | 25mm x 3mm (t) [11/22 mm rings] | 10 | P |



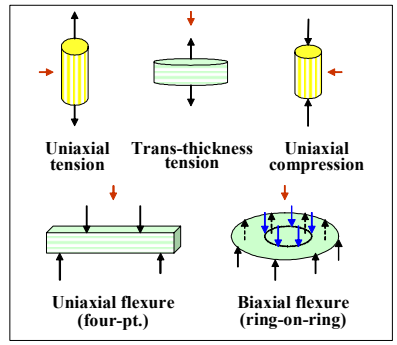

↓ indicates spray direction

* indicates the direction of fracture w.r.t plasma-spray direction.
Test temperature: ambient temperature in air.

# Experimental Results (strength)

| Type of tests | No. of test specimens valid | Direction* | Average strength (MPa)# | Weibull modulus |
|---|---|---|---|---|
| Uniaxial tension | 3 | P | 15(1) | - |
| Trans-thickness Tension | 10 | N | 11(1) | 13 |
| Uniaxial compression | 10 | P | 300(77) | 4 |
| Uniaxial flexure (four-point) | 30 | P | 33(7) | 6 |
| Biaxial flexure (ring-on-ring) | 10 | P | 40(4) | 12 |

* indicates fracture direction w.r.t plasma-spray direction:
N: normal; P: parallel
# represents ±1.0 standard deviation




↓ indicates spray direction

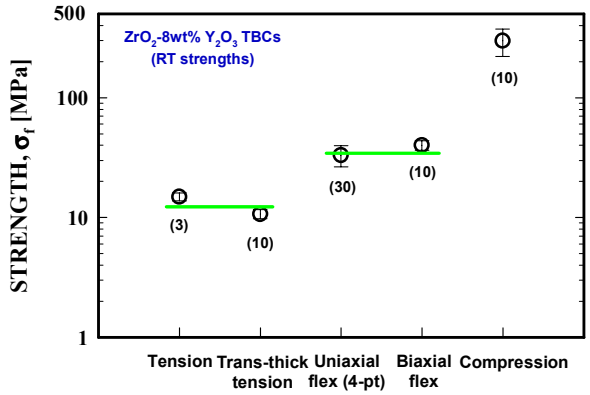
→ **A basic assumption** in strength calculation: a **continuum mechanics** (isotropic and linear-elastic)

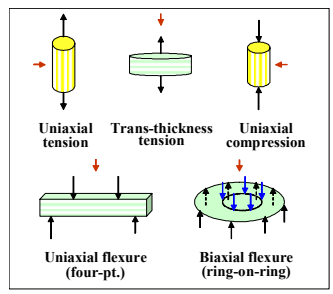
**$\sigma_f$ = 10-15 MPa in tension**
**= 30-40 MPa in flexure**
**= 300 MPa in compression**

**Choi, Zhu, and Miller ('98,'99,'00,'01)**

# Experimental Results (strength)

## Strength *vs* Type of Tests



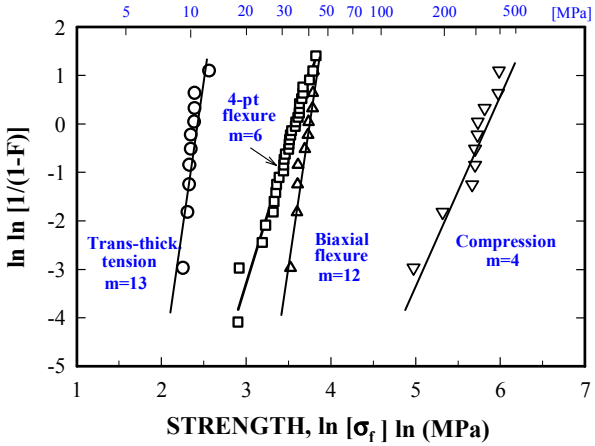

↓ indicates spray direction

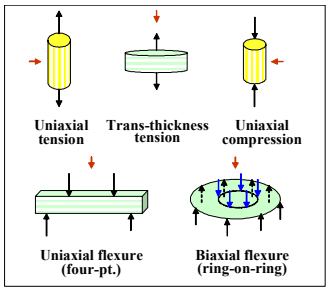
→ $\sigma_f$ = 10-15 MPa in tension
= 30-40 MPa in flexure
= 300 MPa in compression

The numbers indicates the number of specimens tested valid

# Experimental Results (strength)

## Weibull Strength Distributions



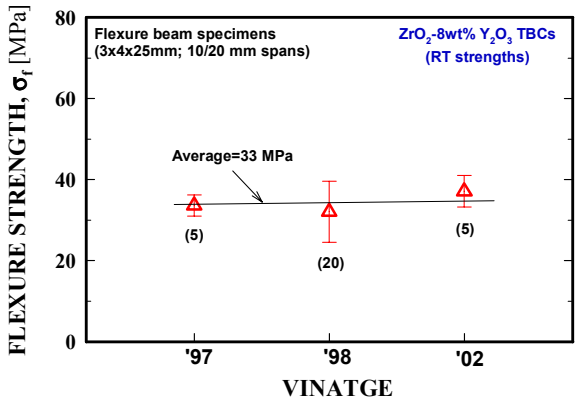

↓ indicates spray direction

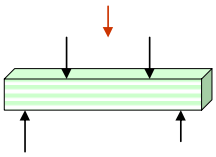
- **Weibull moduli of m=5-15, a typical range for many commercial or in-house (dense) monolithic ceramics**

**Choi, Zhu, and Miller ('98,'99,'00,'01)**

# Experimental Results (strength)

## Flexure Strength *vs* Vintage



↓ indicates spray direction

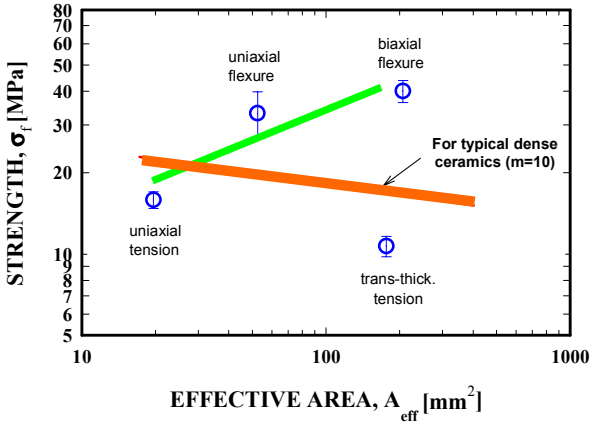
- **Flexure strength – less influence by vintage, indicating consistency in plasma-spray processing over the years**

**Choi, Zhu, and Miller ('98,'99,'03)**

The numbers indicates the number of specimens tested valid

# Strength vs. Effective Area – Size Effect

## Strength-Effective Area (Weibull PIA model)



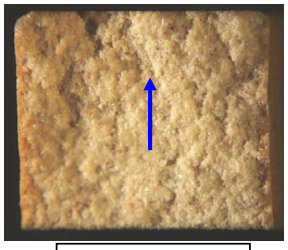

- **No reasonable agreement** in size effect between data and Weibull analysis (e.g., PIA); inconsistency in flaw populations (?)

# Fractography (strength)

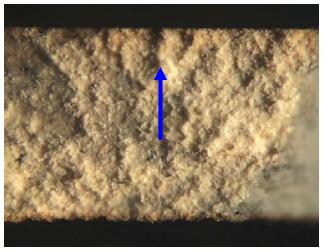
**Uniaxial tension**

**Trans-thickness tension**

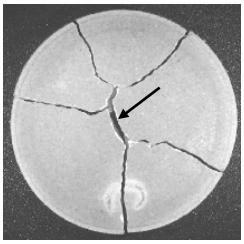
**Uniaxial compression**



**Uniaxial flexure**



Fracture surface



Top view of biaxial Flexure specimen

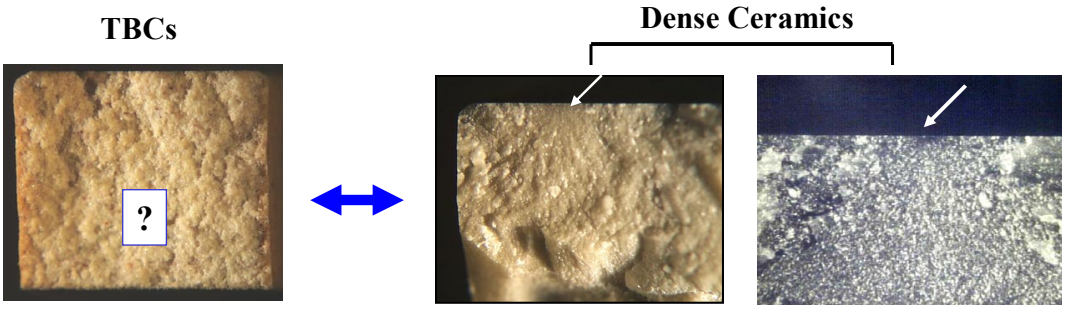
**Biaxial flexure**

- **Very difficult to locate fracture origins and to analyze their nature**

**Choi, Zhu, and Miller ('98,'99,'00,'01)**

# Fractography – A Great Challenge

## Four-point flexure




$ZrO_2$-8wt% $Y_2O_3$ | Toughened $Si_3N_4$ | $ZrO_2$-10 mol% $Y_2O_3$

**Fracture mirror size ($r_m$):**
- **TBCs - 3-40 mm (estimated)**
- **Dense ceramics – 50-500 µm**

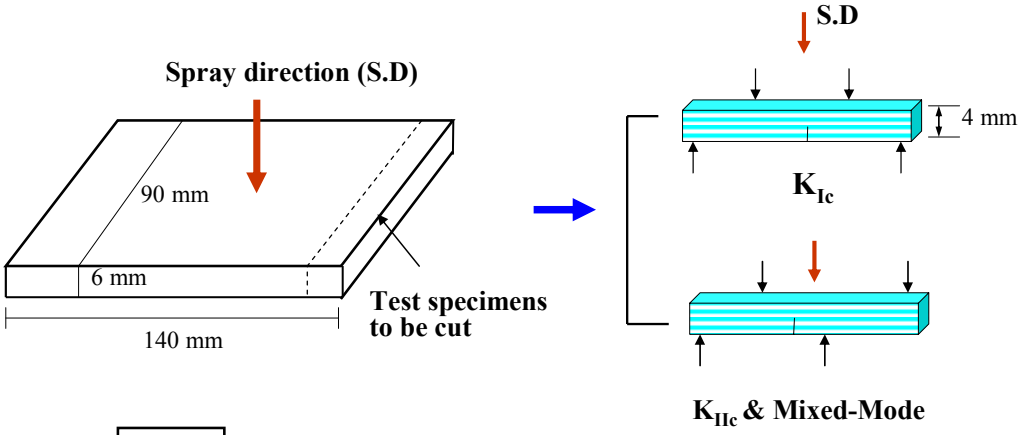
$\sigma_f r_m^{1/2}=A$

• Big mirror size & porous/microcracked nature of TBCs
→ An enormous challenge in fractogrphy

Choi, Zhu, and Miller ('00,'01)
Choi ('02); Choi and Narottam ('02)

# IV. Fracture Toughness Testing
## (Mode I, Mode II and Mixed Mode)

## Types of Testing/Test Specimens/Orientations



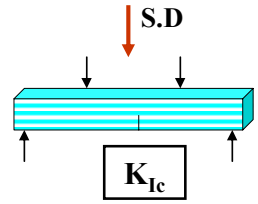

**Billet**

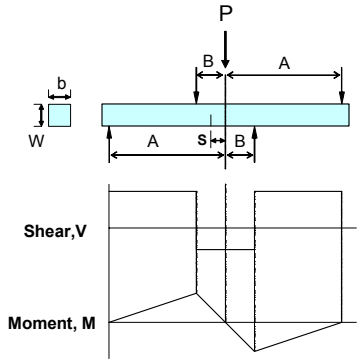
**Test specimens**
**(4mm x 3 mm x 25-50 mm)**

# Experimental (fracture toughness)
## (Mode I, Mode II and Mixed Mode)

### Types & Procedures

- **Sharp precracks generated**
  - Single edge v-notched beam (**SEVNB**) method: Saw-notched → a sharp V-notch generated with a razor blade with diamond paste, $a/W \approx 0.5$
- **Test fixture configurations**
  - A/B=10/5 (typical); s=0-3.6 mm in mixed mode
  - 10/20 or 20/40 mm spans in $K_{Ic}$
- **Test temperatures**
  25 and 1316 °C in air
- **Number of test specimens**: typically ≥4



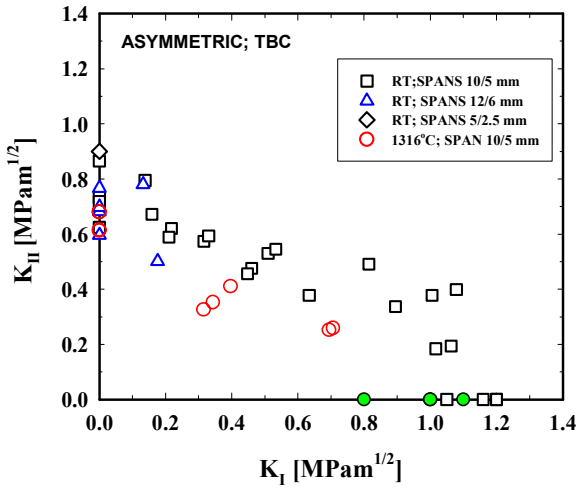



A typical, sharp SEVNB precrack (root radius ≤30 µm)

$K_{IIc}$ & Mixed-Mode -asymmetric flexure-

# Experimental Results (fracture toughness)

- **Mode I, Mode II, and Mixed Mode (25 and 1316 °C)**



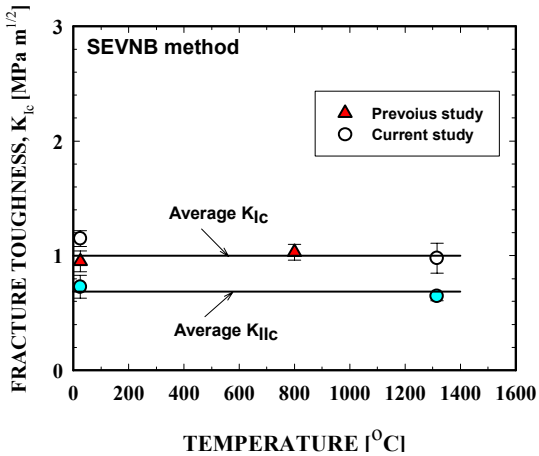

| Test Temp(°C) | No. of specimens used | $K_{Ic}$ (MPa√m) | $K_{IIc}$ (MPa√m) |
|---|---|---|---|
| 25 | 4 in $K_{Ic}$<br>9 in $K_{IIc}$ | 1.15(0.07) | 0.73(0.10) |
| 1316 | 4 each | 0.98(0.13) | 0.65(0.04) |

- **$K_{Ic} > K_{IIc}$** → $K_{IIc}/K_{Ic}$=0.64 & 0.66 (at 25 & 1316 °C)
- $K_{Ic}$ and $K_{IIc}$ at 25 °C ≥ $K_{IC}$ and $K_{IIc}$ at 1316 °C
- **Elliptical relation** between $K_I$ and $K_{II}$
- Test spans **independent**

Choi, Zhu, and Miller ('03)

# Experimental Results (fracture toughness)

- **Fracture Toughness vs. Temperature**

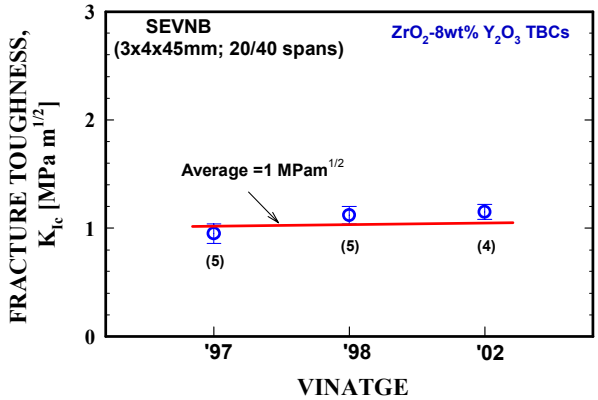



- **Temperature insensitive in $K_{Ic}$ and $K_{IIc}$**
  **$\rightarrow K_{Ic} \approx 1$ and $K_{IIc} \approx 0.65$ MPa$\sqrt{m}$**
- **$K_{IIc} / K_{IC} \approx 0.65$**

Choi, Zhu, and Miller ('98,'03)

# Experimental Results (fracture toughness)

- **Fracture Toughness (RT) vs. Vintage**




- **Fracture toughness ($K_{Ic}$) – less influence by vintage (similar to strength), indicating consistency in plasma-spray processing over the years**

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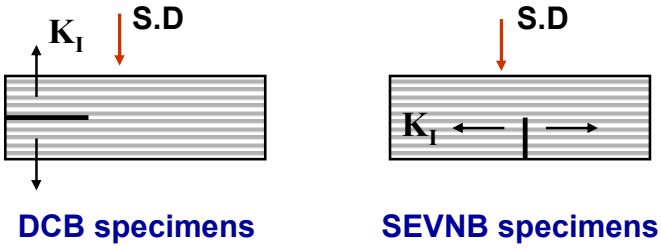
# Experimental Results (fracture toughness)

## Fracture Toughness vs. Orientation



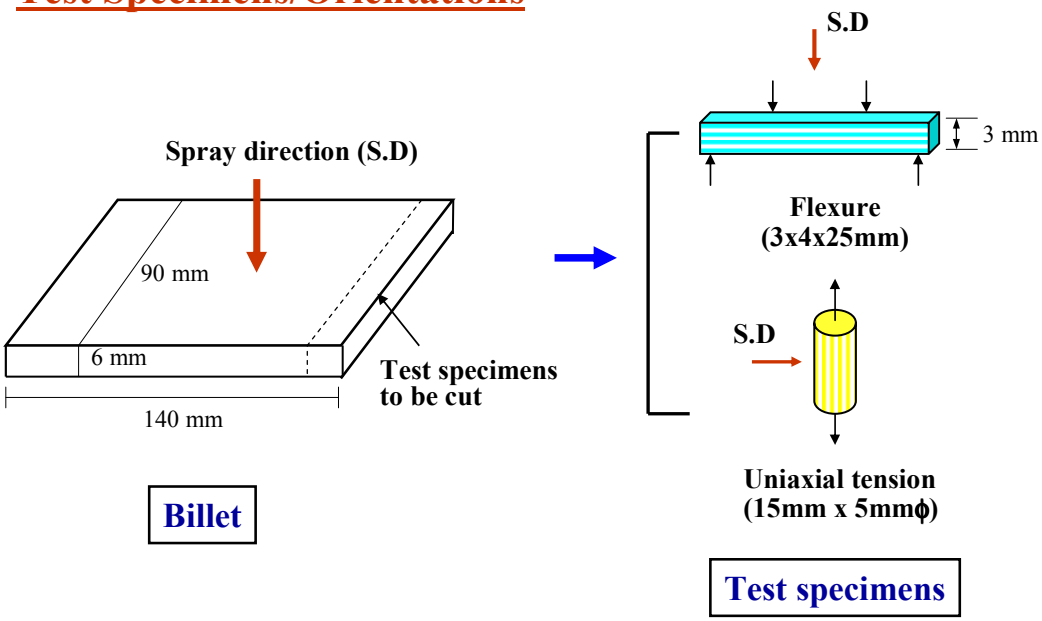

| Direction of crack | Fracture Toughness $K_{IC}$ (MPa√m) | Method |
|---|---|---|
| Parallel to plasma spray direction | **1.15±0.07** | SEVNB (regular method) |
| Normal to plasma spray direction | **1.04±0.05** | DCB (Double Cantilever Beam) |

→ • **No significant difference in $K_{Ic}$-- Little directionality effect on $K_{Ic}$**

Choi, Zhu, and Miller ('98,'03)

# V. Fatigue/Slow Crack Growth

## Test Specimens/Orientations

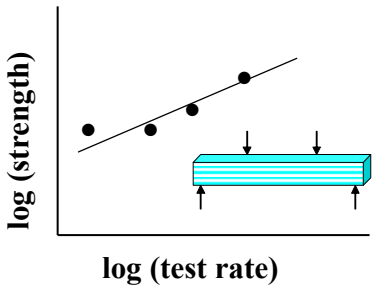


# Experimental (fatigue)

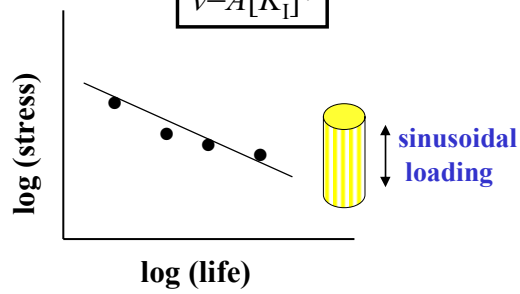
## Test Types and Conditions

- **Dynamic fatigue (ASTM C1425)**
  800 °C in air; 3 test rates in flexure
- **Tensile cyclic fatigue**
  RT in air; sinusoidal; R= 0.1; f=10 Hz

$v=A[K_I]^n$



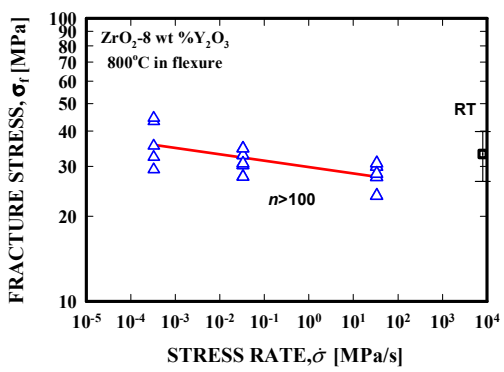

Dynamic fatigue



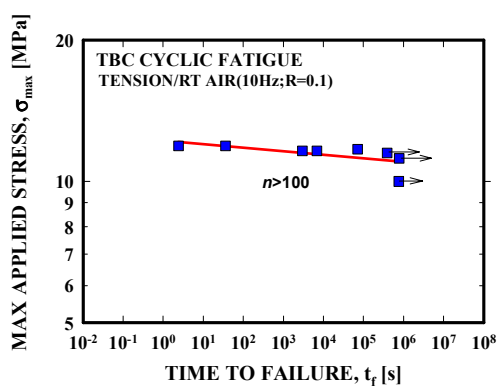

Tensile cyclic fatigue

# Experimental Results (fatigue/SCG)

**Dynamic fatigue**




**Tensile cyclic fatigue**



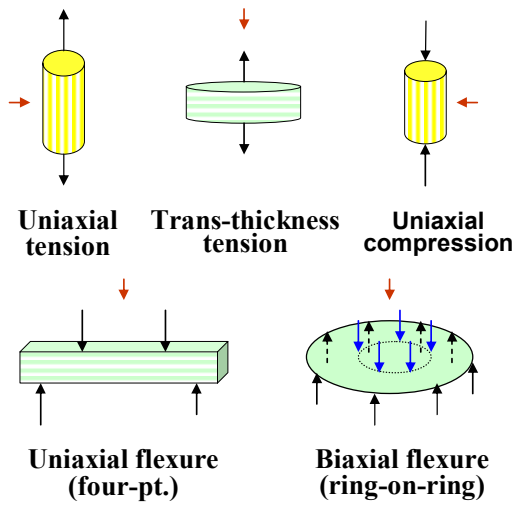

$v=A[K_I]^n$

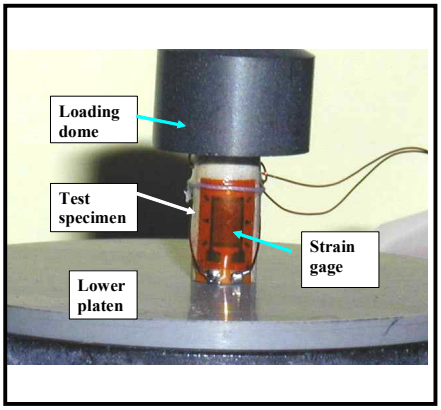
→ • Slow crack growth (SCG) parameter $n$:
$n > 100$ in both dynamic and tensile cyclic fatigue –
**Little susceptibility to SCG (fatigue)**

Choi, Zhu, and Miller ('98,'99,'01)

# VI. Deformation (Stress-Strain) Behavior

## 5 Specimen/Loading Conditions Considered



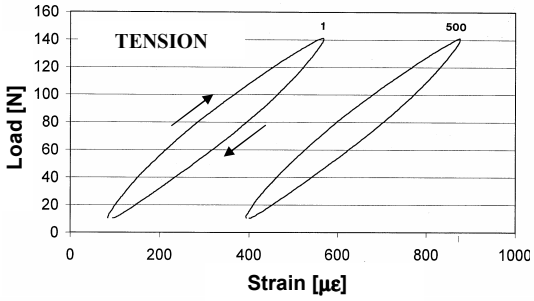



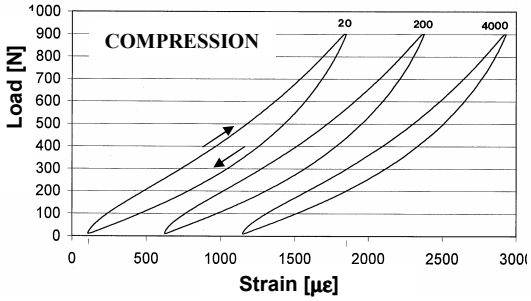

Test Setup (strain gaging)

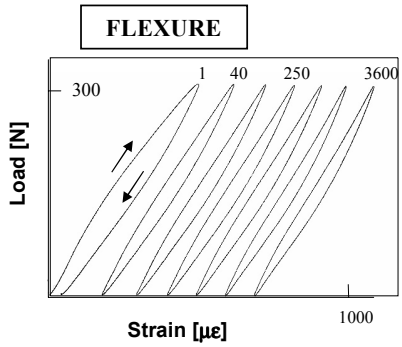
↓ indicates spray direction

# Experimental Results (deformation)

## Typical Load-Strain Curves



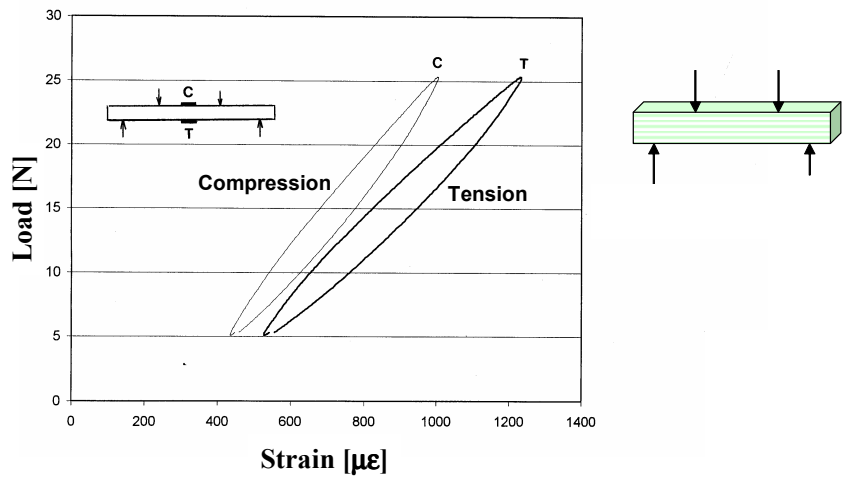




- **Non-linearity** with **hysteresis** but **elastic**
  -desirable in TBCs but difficulty in analysis
- Independent of the number of cycles and test rate (not-viscoelastic)

Choi, Zhu, and Miller ('00,'01)

# Experimental Results (deformation)

## Four-Point Flexure

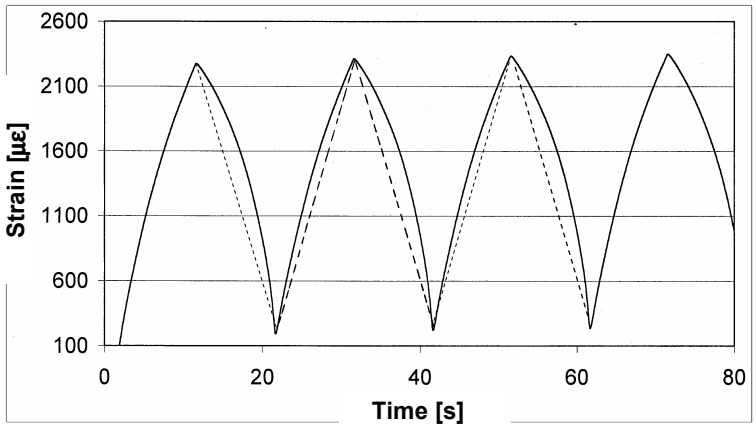


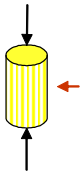

- **Different response of strain** in compression and tension
- A possible **neutral axis shift** due to different elastic modulus
- Flexure stress calculation – complex

Choi, Zhu, and Miller ('01)

# Experimental Results (deformation)

## Response of Output Wave Form to Cyclic Compression Loading



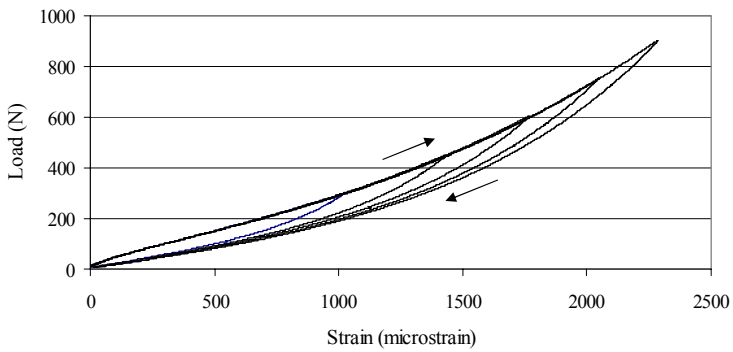
**Triangular cyclic loading**

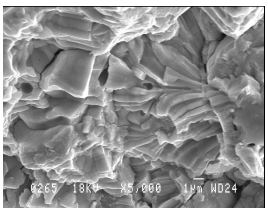
The output wave form - **distorted** from the input triangular wave form

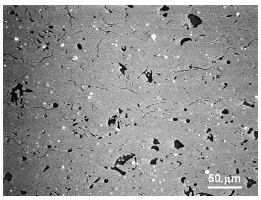
Choi, Zhu, and Miller ('01)

# Deformation (Stress-Strain) Behavior

## What is the cause of nonlinearity and hysteresis?



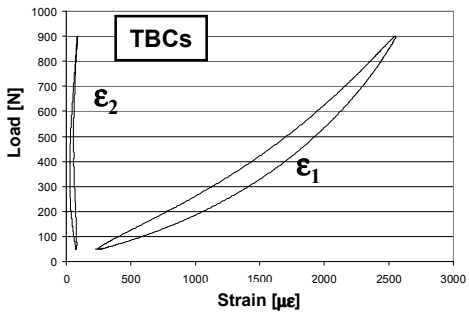


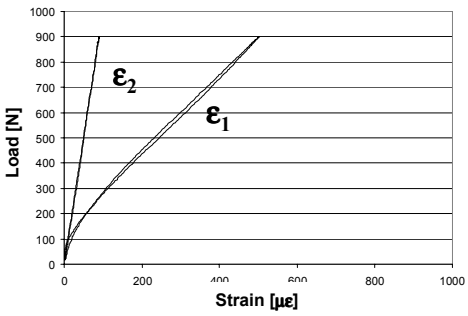

Major reason – **'loosely' connected open structure** due to pores and microcracks
- Internal friction and densification
- Still overall elastic behavior

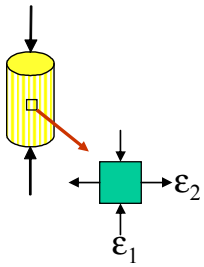
Eldridge, Morscher, and Choi ('02)

# Deformation (Stress-Strain) Behavior

## 'Loosely-Connected Open' Structure – Poisson's Response




**Poisson's ratio**
**$\nu = |\varepsilon_2/\varepsilon_1|$**

Exhibits **no or little change** in lateral strain: Poisson's ratio not well defined

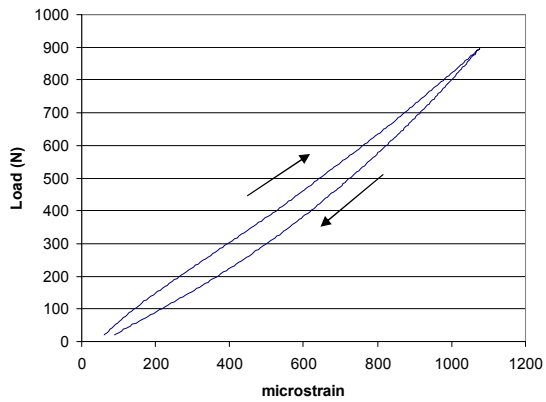
**Open, loose structure**

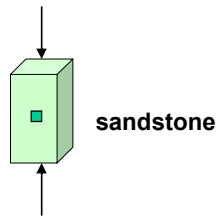
Exhibits a **linearly increasing** lateral strain: Poisson's ratio well defined

**Dense structure**

# Experimental Results (deformation)

## Sandstone – Another Example of Open Structure



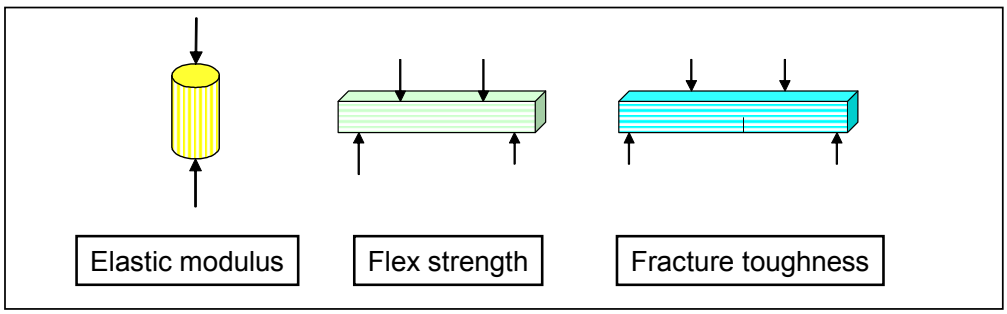




Open, loose structure: non-linearity with hysteresis
-- Similarity to TBCs

# VII. Sintering – A Changer of Structure

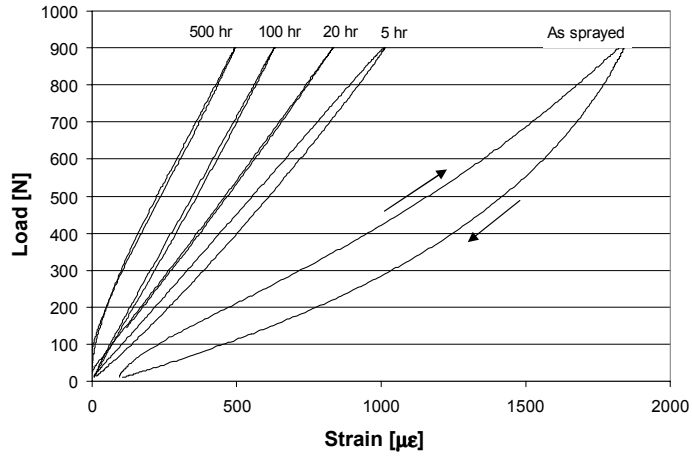
## Sintering conditions:

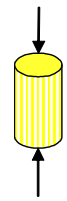
- Temperature/environment: 1316 $^{o}$C/air
- Annealing time: 0, 5, 20, 100, and 500 h
- Determine as a function of anneal time:
  - Elastic modulus
  - Fracture toughness ($K_{Ic}$)
  - Flexure strength
  - Thermal conductivity



# Experimental Results (sintering)

## Elastic Modulus



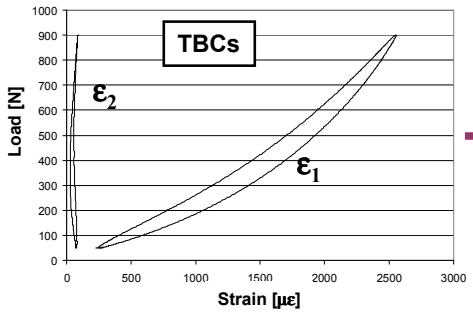
Compression

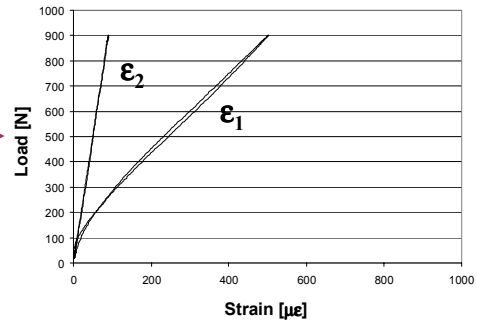
- **Slope (elastic modulus) increases** with anneal time
- **Linearity increases** with anneal time
- **Hysteresis decreases** with anneal time

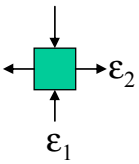
→ Implies a change of microstructure from **'loosely'** connected to '**closely**' connected

# Experimental Results (sintering)

## Well-Developed Poisson's (Lateral Strain) Response



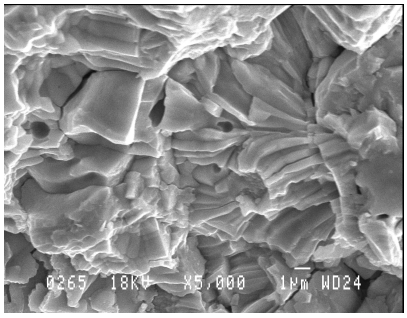

**Poisson's ratio**
**$\nu = |\varepsilon_1/\varepsilon_2|$**

**500 h annealing**

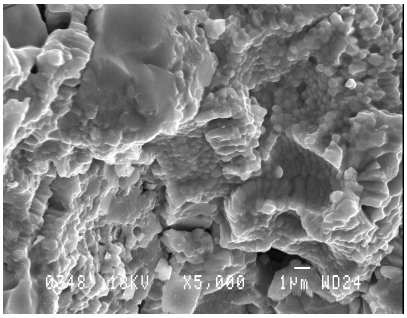
**Open structure → More closely-connected structure**

# Experimental Results (sintering)

## Microstructure




As-sprayed

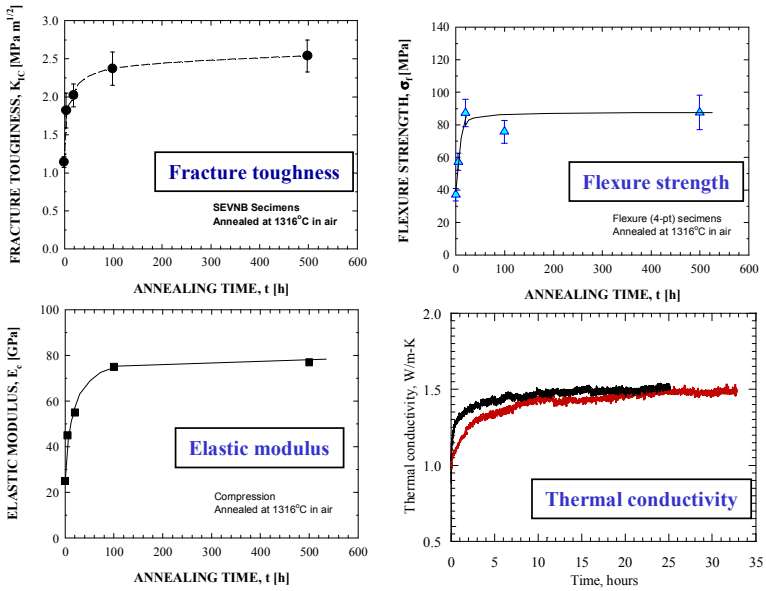



100 h annealed

- **As-sprayed - Large amounts of microcracks and pores with a unique platelet (splat) structure presented**
- **100 h annealing - Increased grain growth at longer annealing time**

# Experimental Results (sintering)

- **Summary on elastic modulus, flexure strength, fracture toughness and thermal conductivity**




→ **Properties change exponentially with sintering time**

Choi, Zhu, and Miller ('03)

## Standardization Issues

- **The most hindering factor** in establishing test methods for as-sprayed TBCs: **non-linearity** & **hysteresis** in the constitutive relations
  - Flexure testing (uniaxial and biaxial) maybe inappropriate due to difference in modulus between tension and compression
  - Poisson's ratio not well-defined
  - Impulse excitation technique maybe inappropriate
- Pure tension and compression testing – impose less problems
- Fracture toughness testing – maybe OK in view of low fracture loads
- Fractography – challenging
- Properties change with sintering/service conditions
  - requires to evaluate based on sinter/service conditions

## Summary

- **Strength:**
  **tension: 10-15 MPa; flexure: 30-40 MPa; compression: 300 MPa**
  **Weibull modulus: 5-15**
- **Fatigue/Slow Crack Growth:**
  **SCG parameter *n*>100**
- **Fracture Toughness:**
  **$K_{Ic}$=1.0 MPa√m up to 1316 °C**
  **$K_{IIc}$=0.7 MPa√m up to 1316 °C**
- **Deformation:**
  **nonlinear elasticity with hysteresis;**
  **imposes problems in continuum approach (*test standards*)**
- **Sintering:**
  **significant influence - a changer of most properties!**

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**13. ABSTRACT** ***(Maximum 200 words)***

Strength, fracture toughness and fatigue behavior of free-standing thick thermal barrier coatings of plasma-sprayed $ZrO_2$-8wt % $Y_2O_3$ were determined at ambient and elevated temperatures in an attempt to establish a database for design. Strength, in conjunction with deformation (stress-strain behavior), was evaluated in tension (uniaxial and trans-thickness), compression, and uniaxial and biaxial flexure; fracture toughness was determined in various load conditions including mode I, mode II, and mixed modes I and II; fatigue or slow crack growth behavior was estimated in cyclic tension and dynamic flexure loading. Effect of sintering was quantified through approaches using strength, fracture toughness, and modulus (constitutive relations) measurements. Standardization issues on test methodology also was presented with a special regard to material's unique constitutive relations.

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