Reliability and Durability of Materials & Components for SOFCs

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SECA Core Technology Program Review
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Oak Ridge National Laboratory

Outline

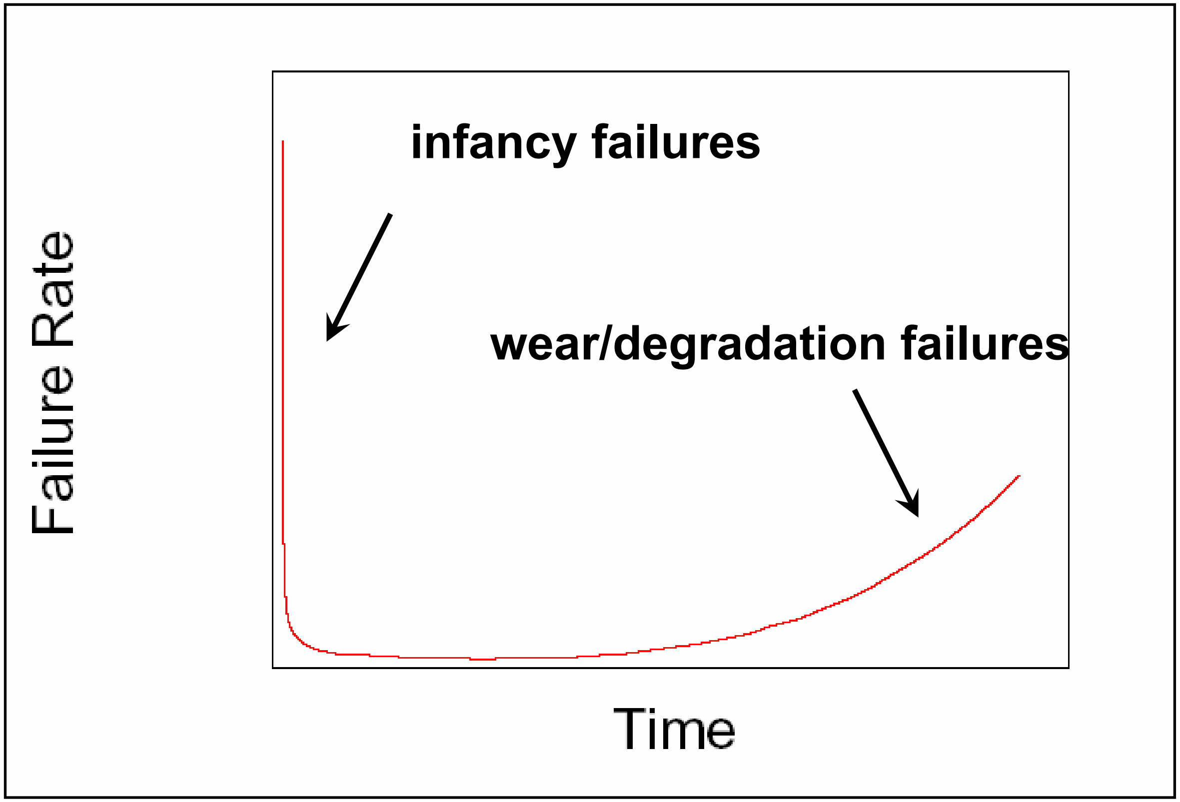
- Objectives
- Predicting Reliability
  - Infancy Failures
  - Wear/Degradation-induced Failure
- Evaluation of Material Properties
- Implications of Results for Manufacturing
- Future Work
  - Phase Identification and Micromechanical Stress Calculations

Objectives

In collaboration with industrial teams and other Core Technology Program participants,

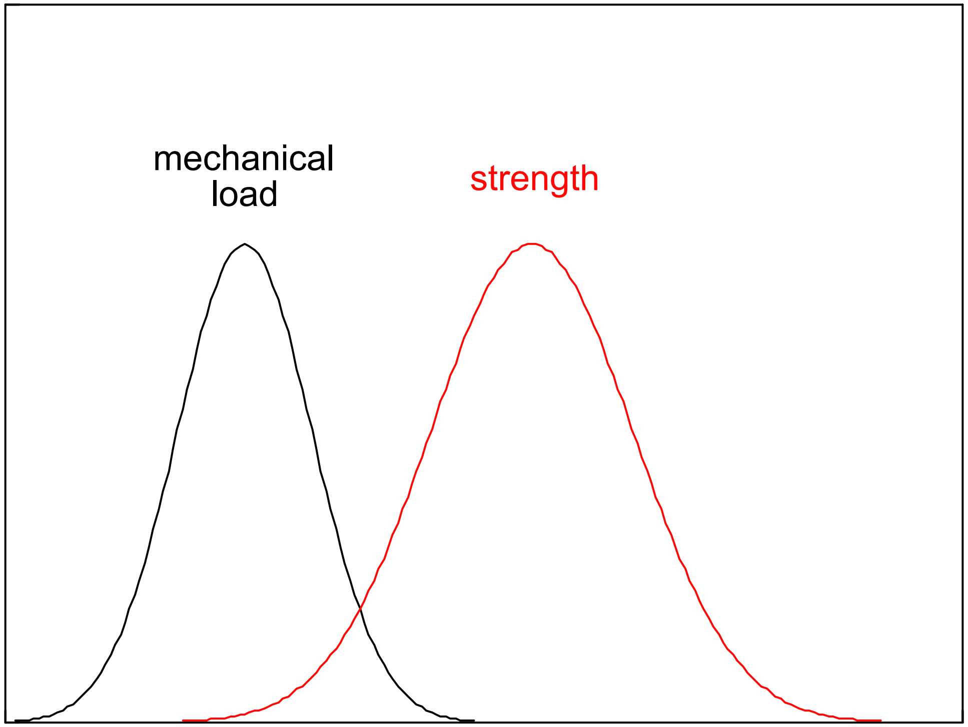
- *To develop/adapt/recommend test techniques to evaluate the properties and behavior of materials and components for SOFC.*
- *To identify and understand the mechanism responsible for the failure of materials and components for SOFCs.*
- *To develop methodologies for predicting the durability and reliability of materials and components for SOFCs.*

A bathtub curve describes the evolution of the failure rate for most complex systems



What information is needed to predict infancy failures of SOFCs?

- Stress distribution
- Distribution of strengths



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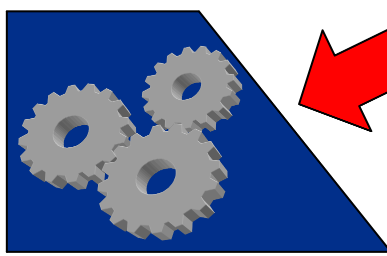
Stress Distribution

- Geometry
- Temperature Distribution
- Mechanical Loads
- Boundary Conditions
- Elastic Constants
- Volumetric Changes
- Thermal Expansion

Elastic Constants as a function of:
- porosity
- temperature

Volumetric Changes due to reduction



Distribution of Strengths

Strength as a function of:
- porosity
- temperature
- size

Toughness
- interfacial

Reliability/Probability of Failure

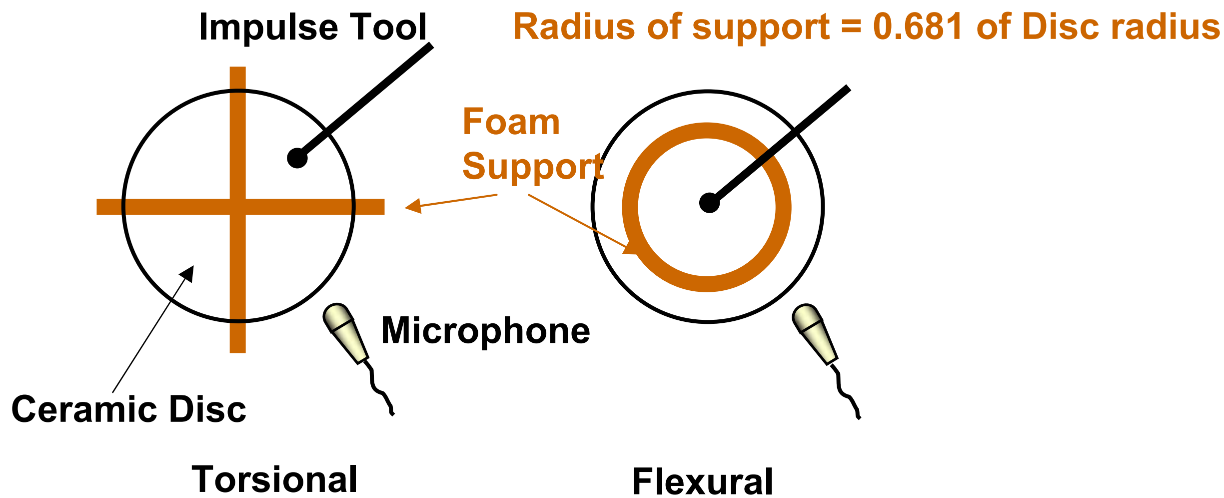
Characterized Materials

8YSZ - Zirconia stabilized with 8mol% Yttria
NiO/YSZ - 75mol%NiO/25mol%YSZ, a precursor to Ni/YSZ anode

	8YSZ			NiO/YSZ				
# of laminated layers	1	2	4	2	4	6	4	4
Nominal Thickness, mm	0.25	0.50	1.00	0.50	1.00	1.50	1.00	1.00
Pore former, vol%	0	0	0	30	30	30	25	0
Sintering conditions	1400 °C for 2 h			1400 °C for 2 h				
Measured porosity, %	6.2 ±1.0	6.3 ±1.5	5.7 ±1.2	-	22.8 ±1.1	-	19.8 ±0.9	6.8 ±0.3

Young’s and Shear Moduli

Impulse Excitation Technique (ASTM C1259-98)

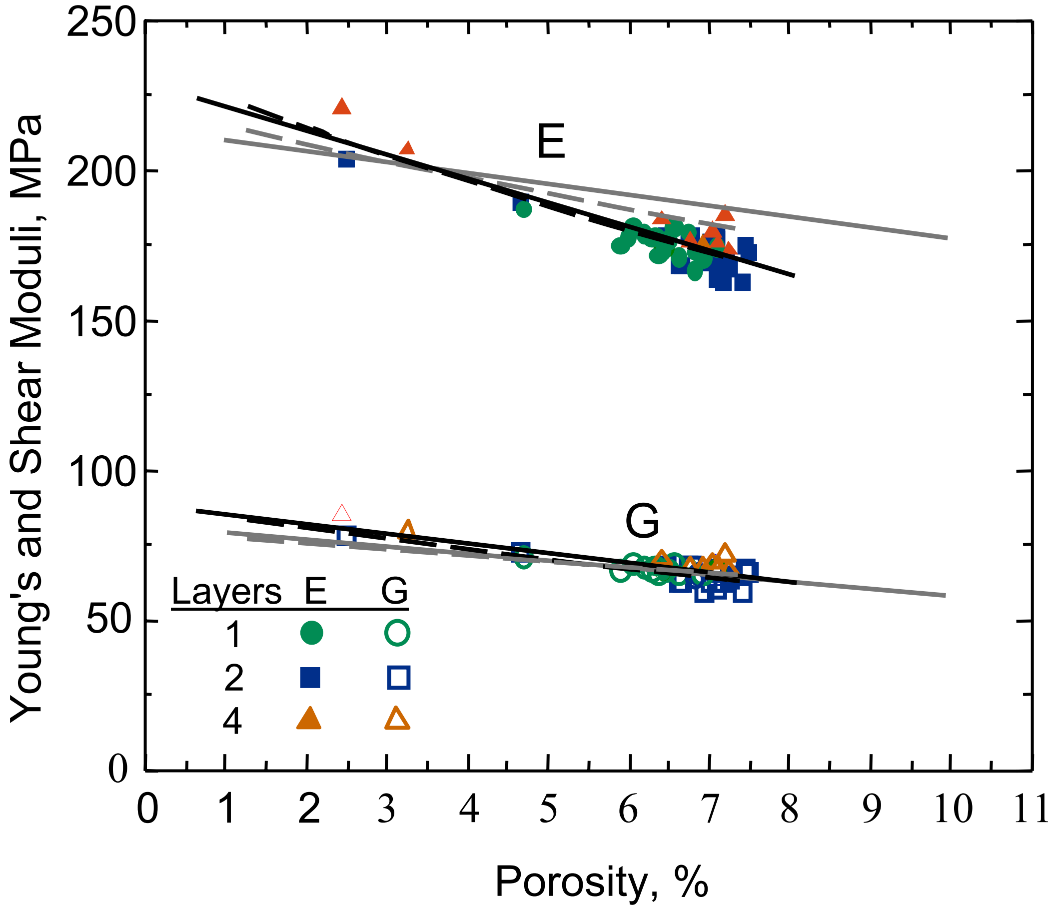


$$E_{t,f} = \frac{\left[37.699 f_{t,f}^2 D^2 m \left(1 - \mu^2\right)\right]}{K_{t,f}^2 h^3}$$

$E_{t,f}$ = Young's modulus as measured by torsional/flexural resonance
m = mass of the disc
t = height of the disc
D = diameter of the disc
$F_{t,f}$ = fundamental torsional/flexural resonant frequency of the disc
K_t = a correction factor (ASTM C1259-98)
μ = Poisson's ratio

Young's and Shear Moduli

8mol%YSZ as a function of porosity



This work:

E = 229.85 (1 - 3.80 *p*)
G = 88.24 (1 - 3.69 *p*)

E = 234.54 exp(- 4.35 *p*)
G = 90.20 exp(- 4.51 *p*)

Literature*:

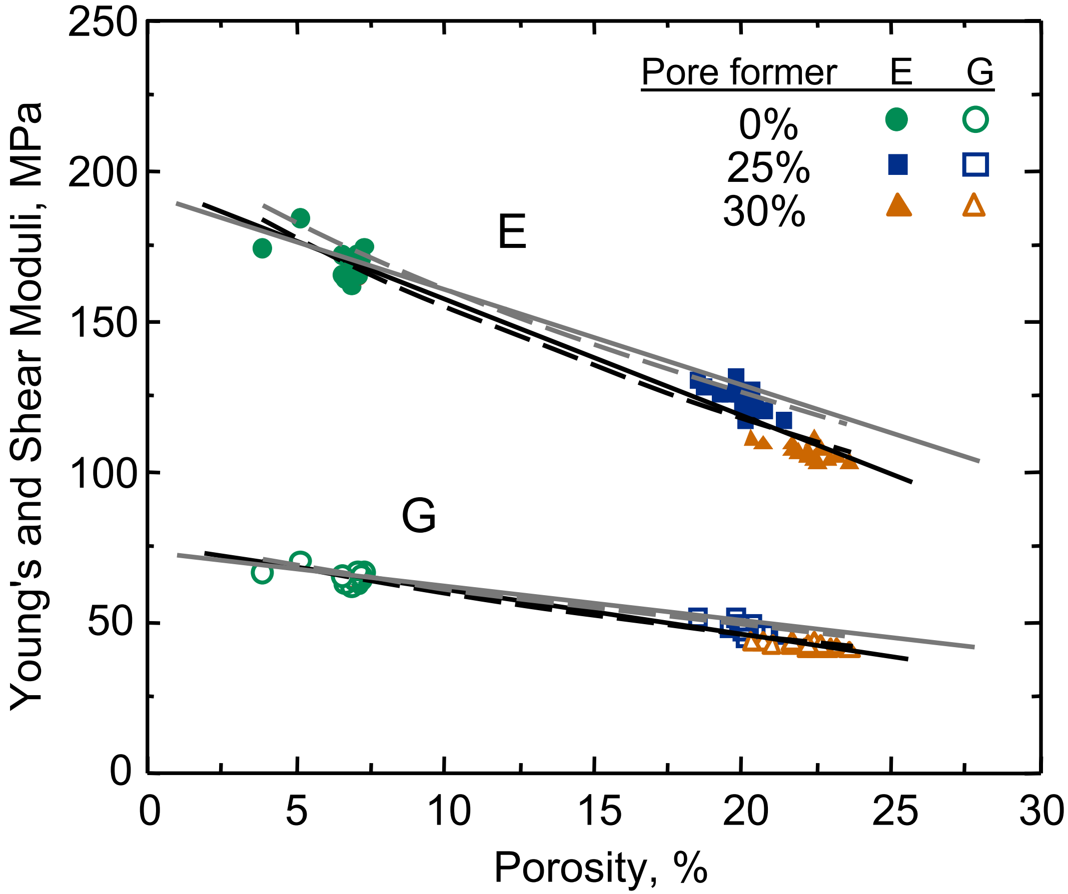
E = 219.53 (1 – 2.50 *p*)
G = 83.22 (1 – 2.39 *p*)

E = 220.27 exp(- 2.76 *p*)
G = 83.47 exp(- 2.63 *p*)

*A. Selcuk and A. Atkinson, *J. Euro. Ceram. Soc.*, **17** (1007) p.1523

Young's and Shear Moduli

75mol%NiO/YSZ as a function of porosity



This work:

E = 195.49 (1 – 1.96 *p*)
G = 75.15 (1 – 1.93 *p*)

E = 204.47 exp(- 2.76 *p*)
G = 78.09 exp(- 2.65 *p*)

Literature*:

E = 205.46 (1 – 2.10 *p*)
G = 77.04 (1 – 2.03 *p*)

E = 207.13 exp(- 2.48 *p*)
G = 78.04 exp(- 2.38 *p*)

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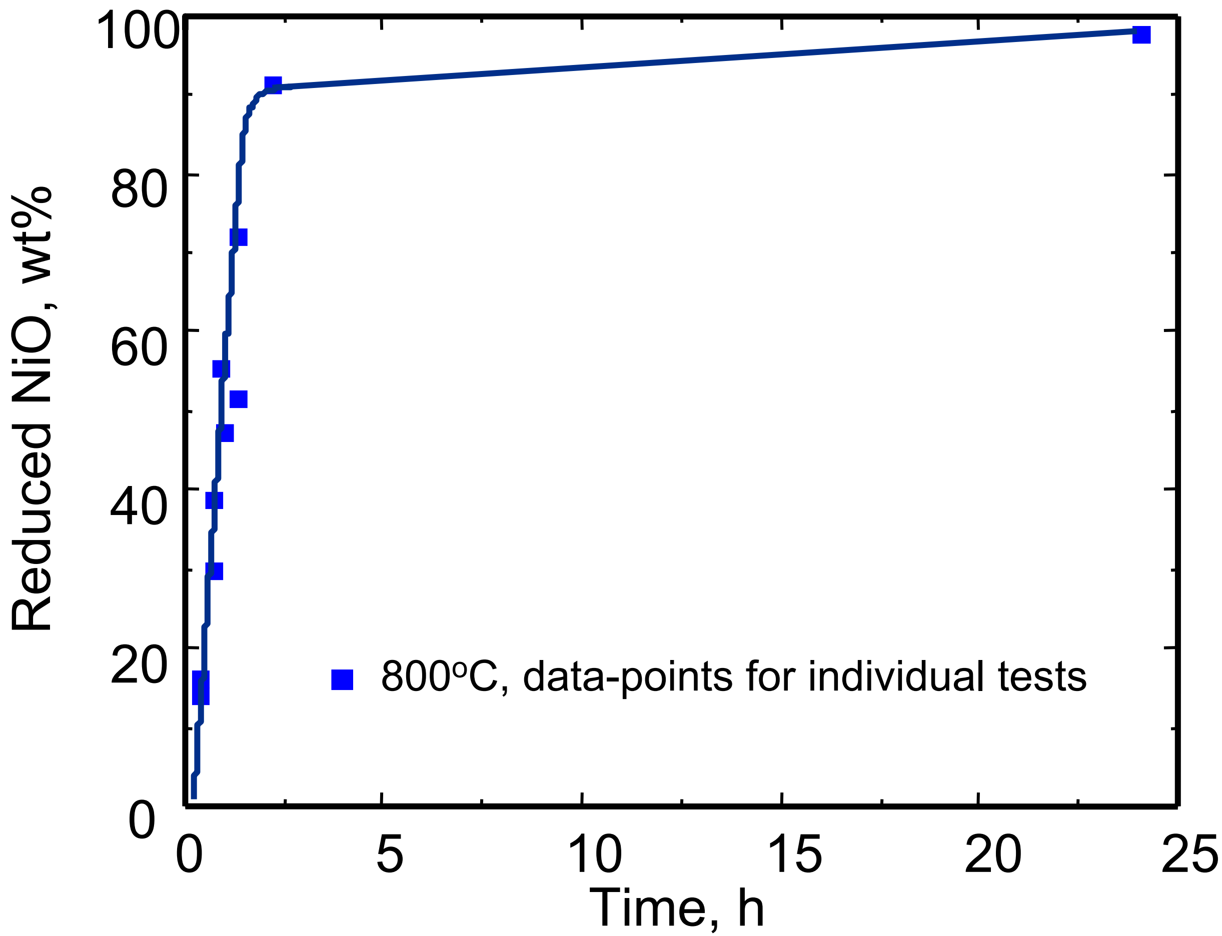
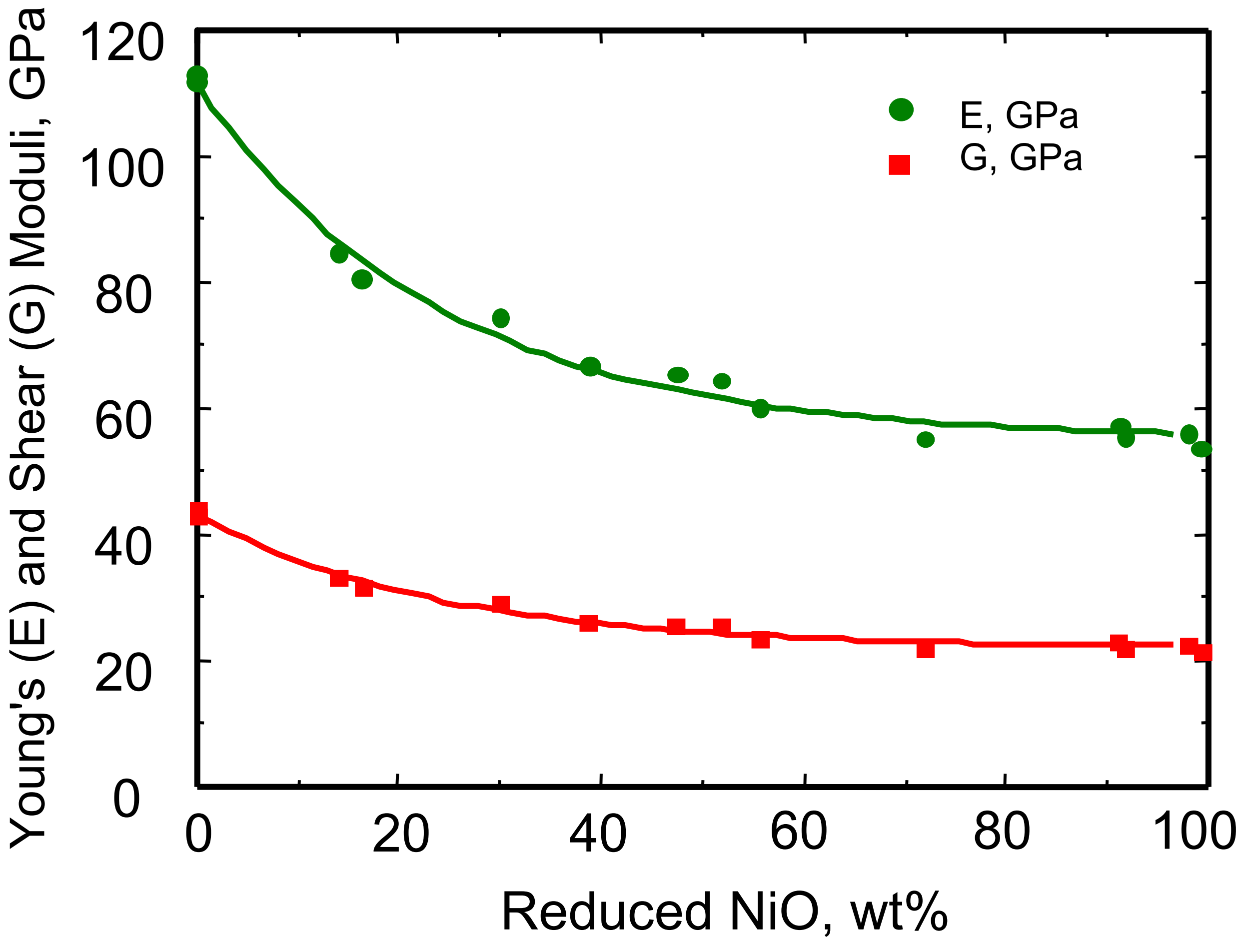
Thermogravimetric Analysis (TGA) of NiO/YSZ Reduction



Reduction of NiO measured for different samples. Samples were reduced for a different period of time at 800°C in 4%H2-96%Ar gas mixture

Young's and Shear Moduli vs. wt% of Reduced NiO in Anode



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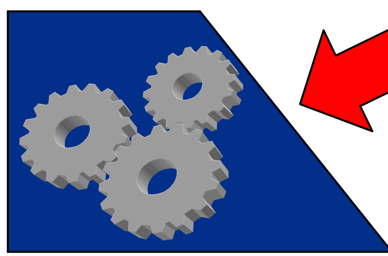

Distribution of Strengths

Strength as a function of:
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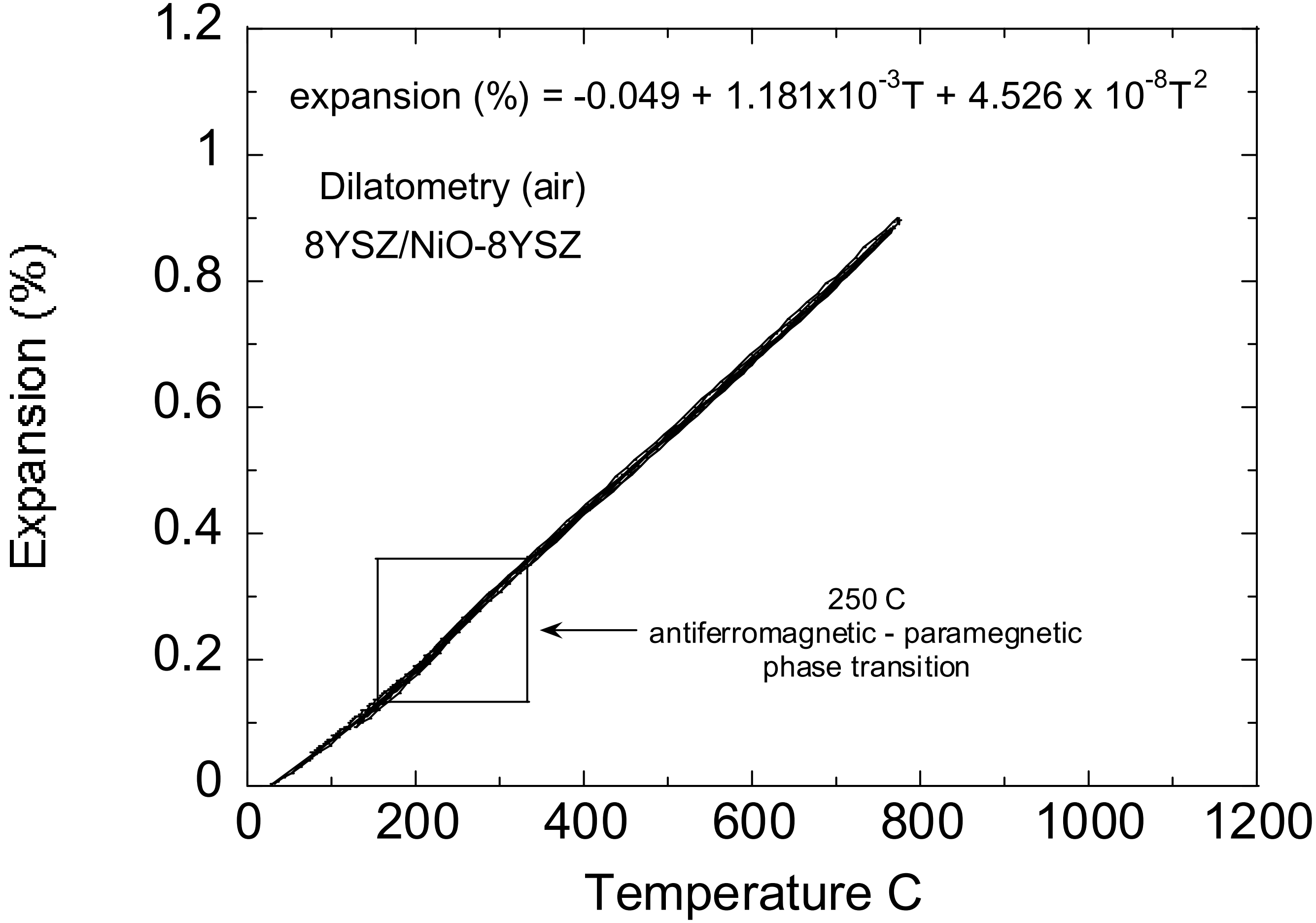
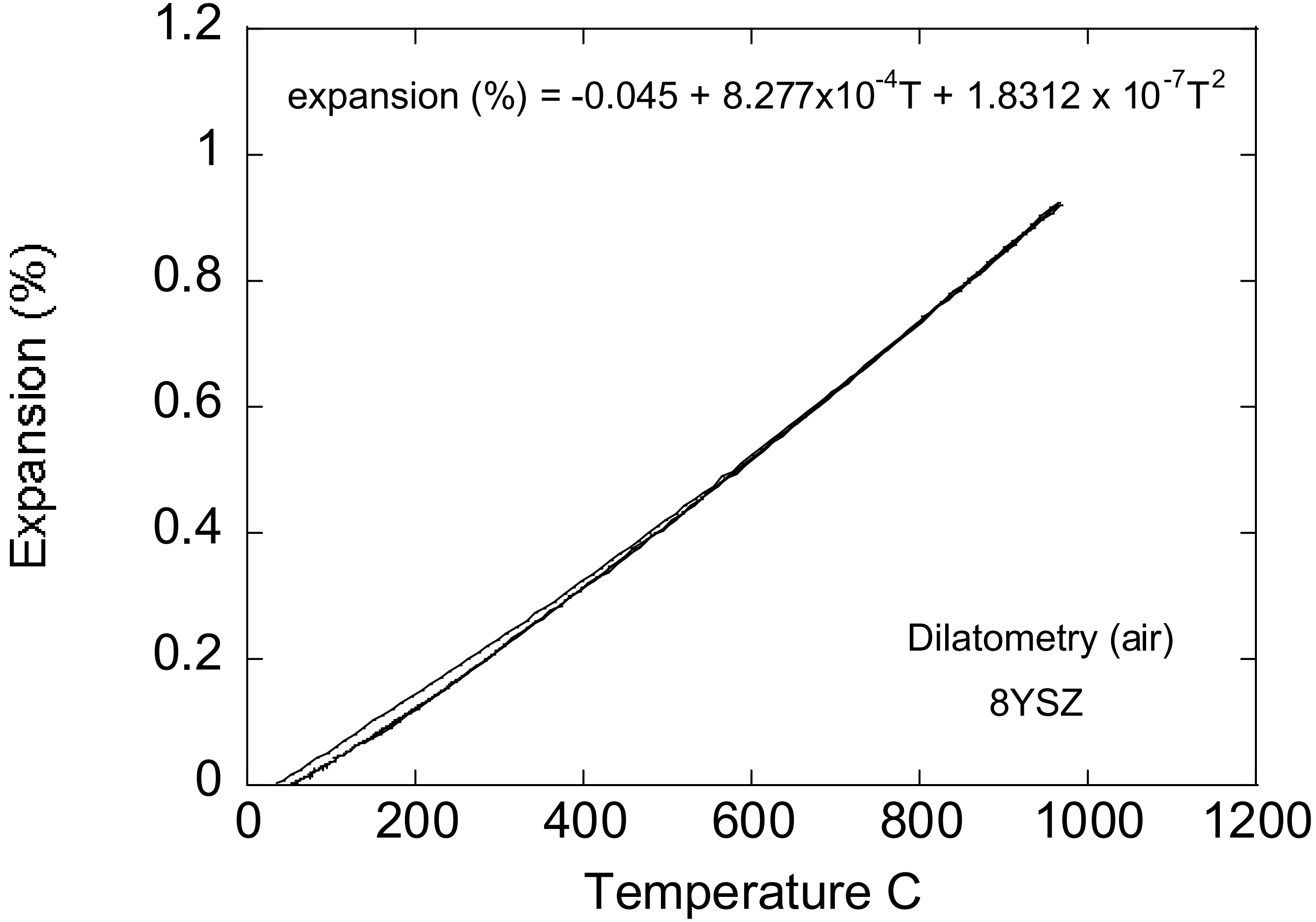
Toughness
- interfacial

Reliability/Probability of Failure

Thermal Expansion of NiO/8YSZ



Thermal Expansion of 8YSZ



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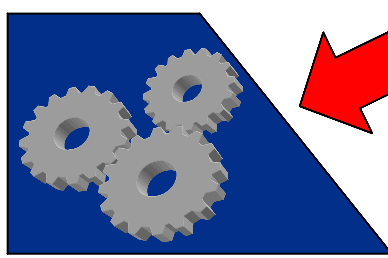

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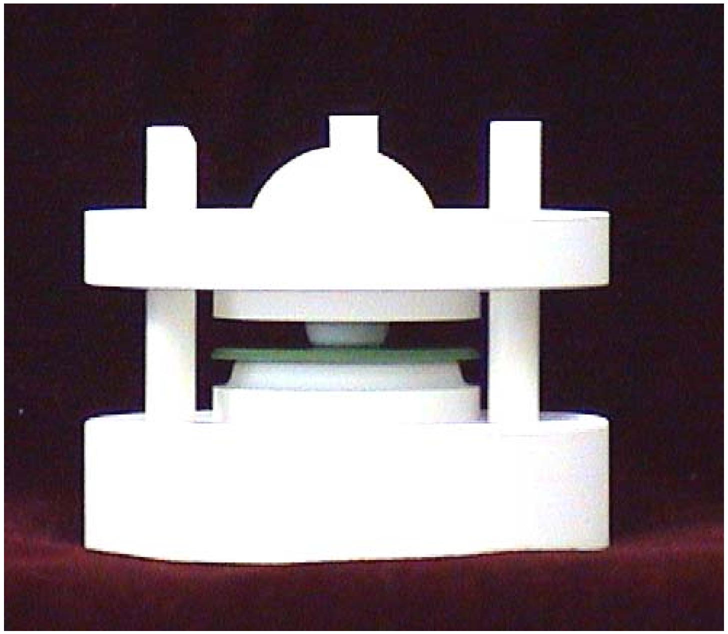
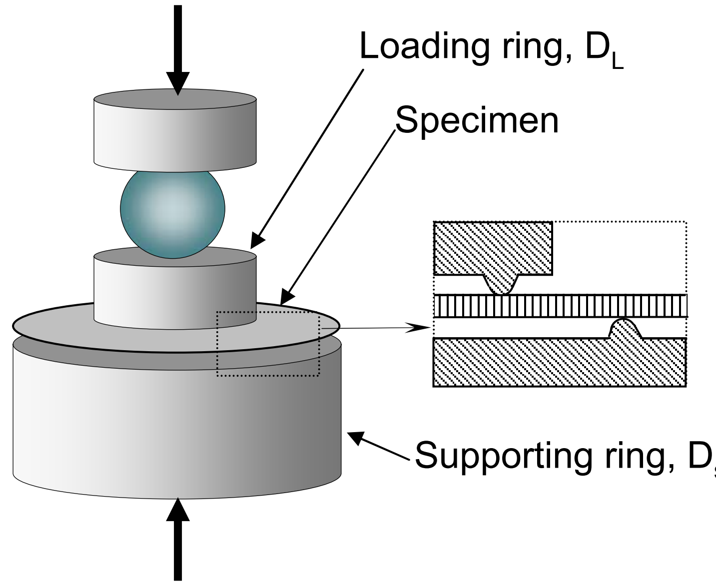
Reliability/Probability of Failure

Biaxial Strength

Ring-on-ring Testing (ASTM C1499-01)



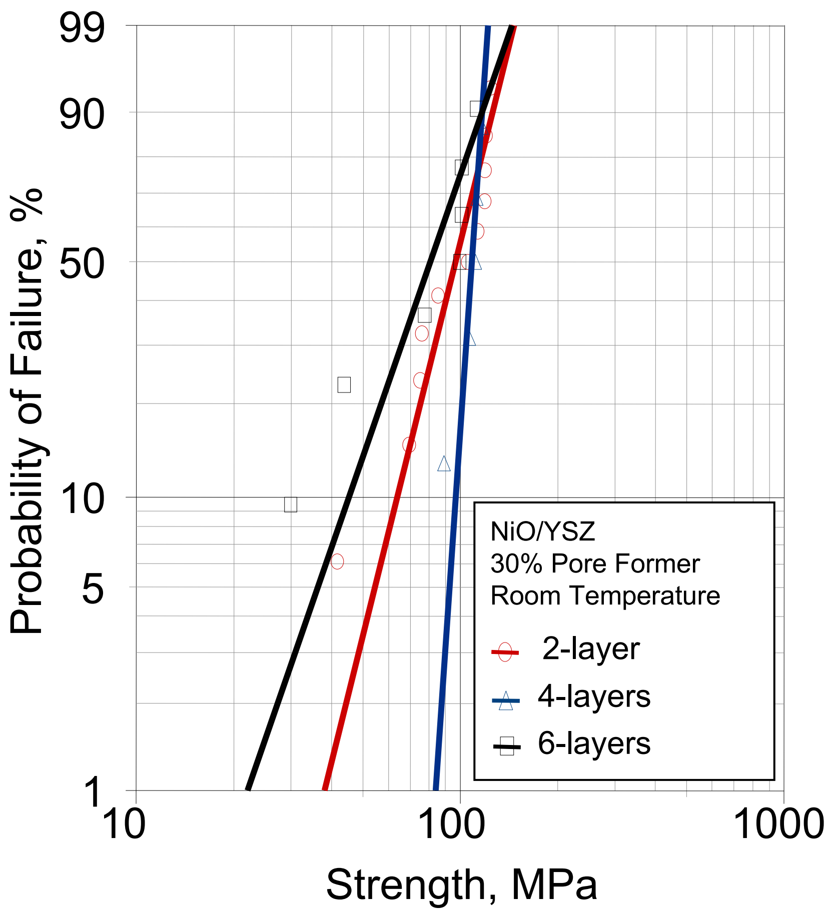


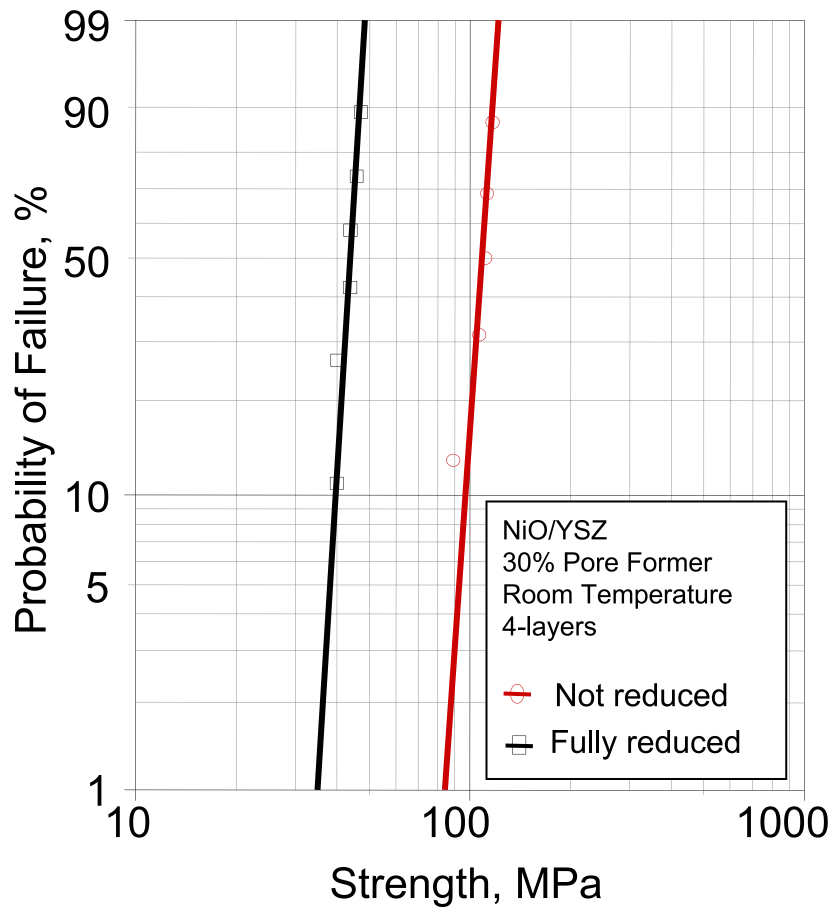
$$\sigma_f = \frac{3F}{2\pi h^2}\left[(1-\nu)\frac{D_s^2 - D_l^2}{2D^2} + (1+\nu)\ln\frac{D_s}{D_l}\right]$$

where *F* is breaking load, *h* sample thickness, ν is Poisson's ratio and *D*, *Ds* and *Dl* are diameter of sample, supporting ring and loading ring, respectively

Biaxial Strength

NiO/8YSZ – Weibull plots

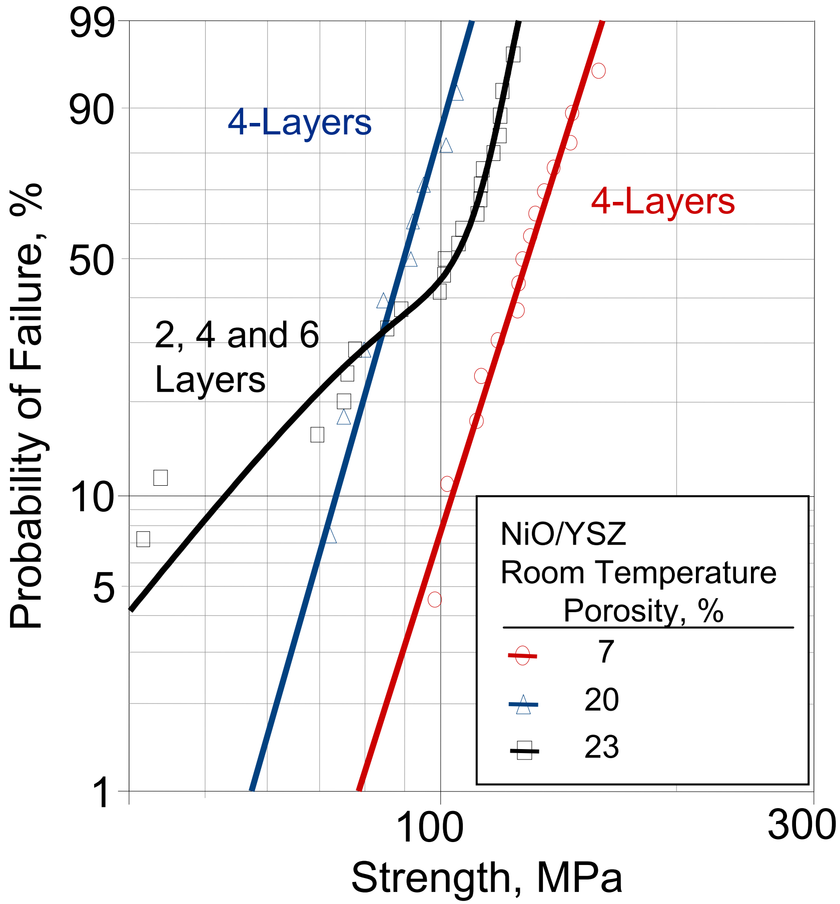


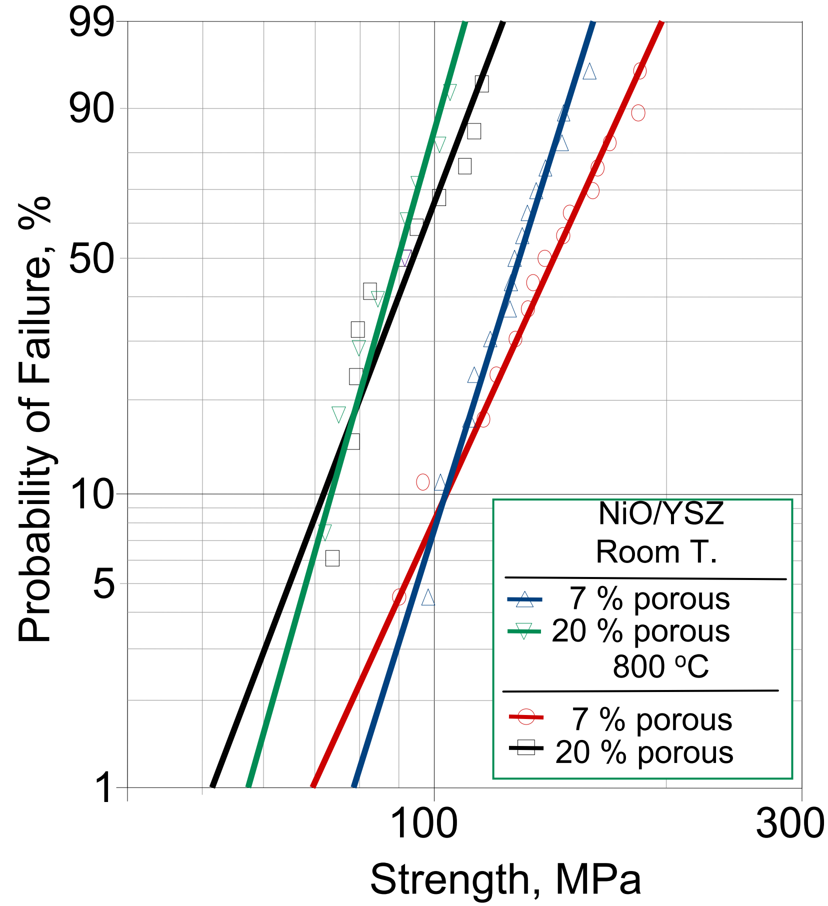


b2=18.8243, h2=44.7583

Biaxial Strength

NiO/YSZ – Weibull plots





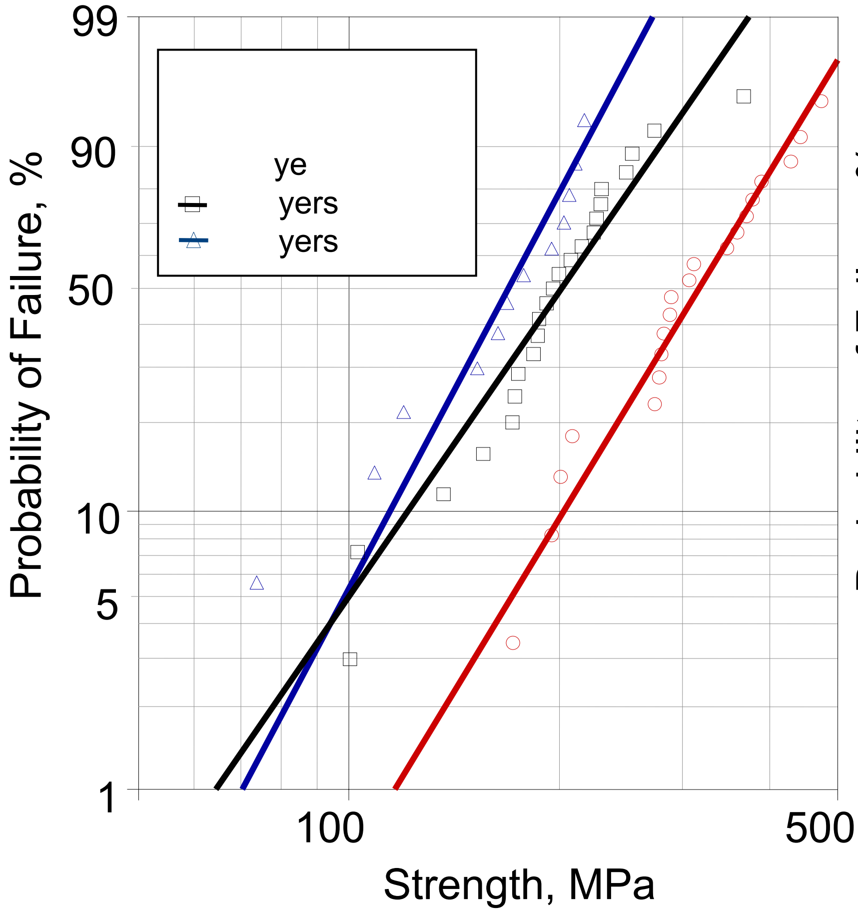
Biaxial Strength

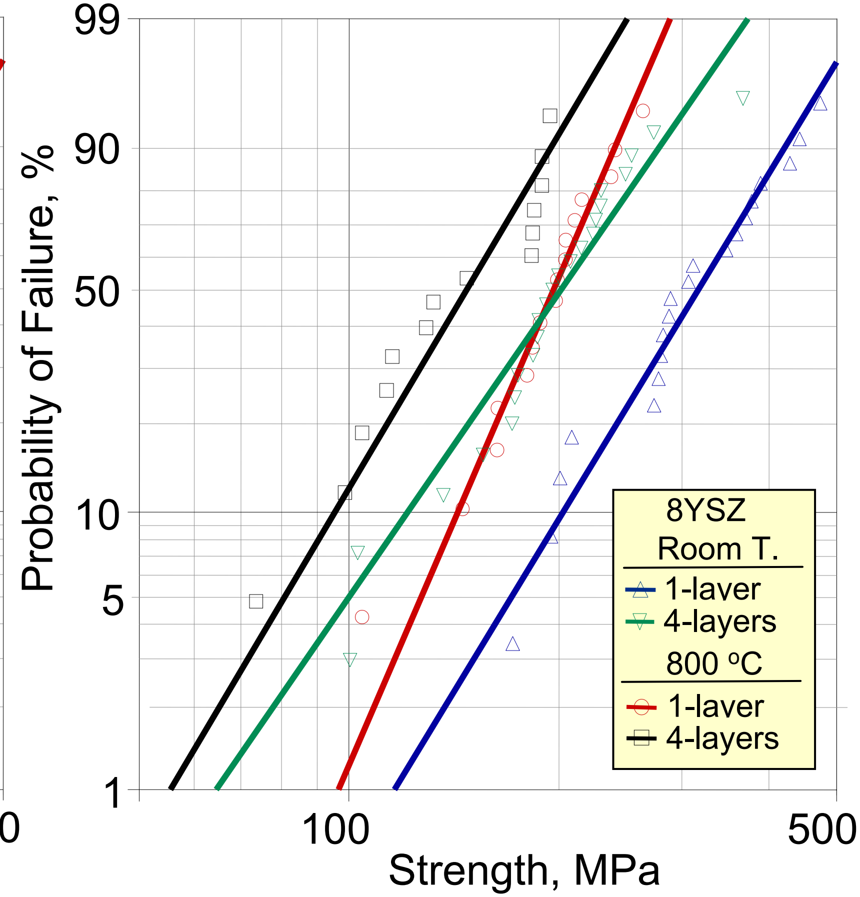
NiO/YSZ – Summary of Weibull statistics

Characteristic strength (MPa) / Weibull modulus Average strength ± Standard Deviation (MPa)			
NiO/YSZ			
# layers -Pore former/Porosity, %	2 - 30/23	4 - 30/23	6 - 30/23
Room Temperature	105.9 / 3.5 95.3 ± 27.2	111.3 / 16.5 107.3 ± 10.8	90.6 / 3.3 80.8 ± 32.1
Ni/YSZ (Fully reduced NiO/YSZ)			
# layers-Pore former/Porosity, %	-	4 - 30/41	-
Room Temperature	-	44.7 / 18.7 43.5 ± 2.9	-
NiO/YSZ			
# layers-Pore former/Porosity, %	4 - 0/7	4 - 25/20	2, 4 and 6 - 30/23
Room Temperature	134.6 / 8.6 127.4 ± 17.3	93.3 / 9.4 88.5 ± 11.4	79.6 / 3.4 - 115.4 / 17.4 65.4 ± 25.3 - 111.6 ± 7.6
800oC	152.3 / 5.8 140.9 ± 28.6	98.9 / 7.0 92.6 ± 15.1	-

Biaxial Strength

8YSZ – Weibull plots





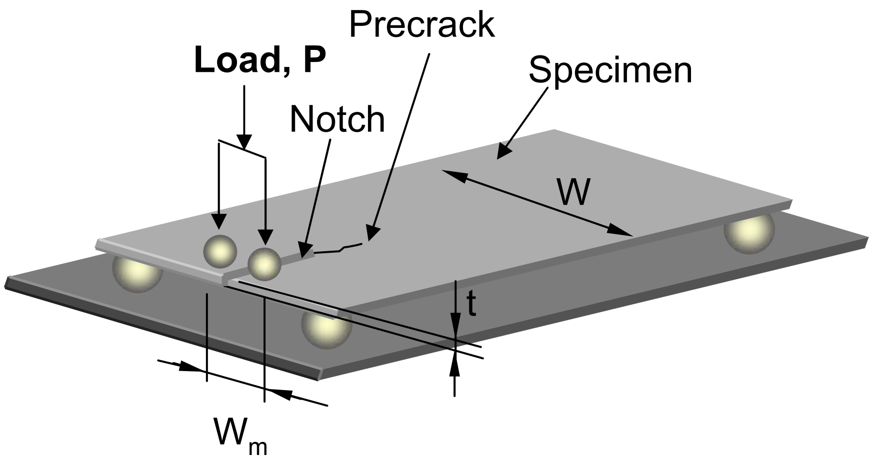
Biaxial Strength

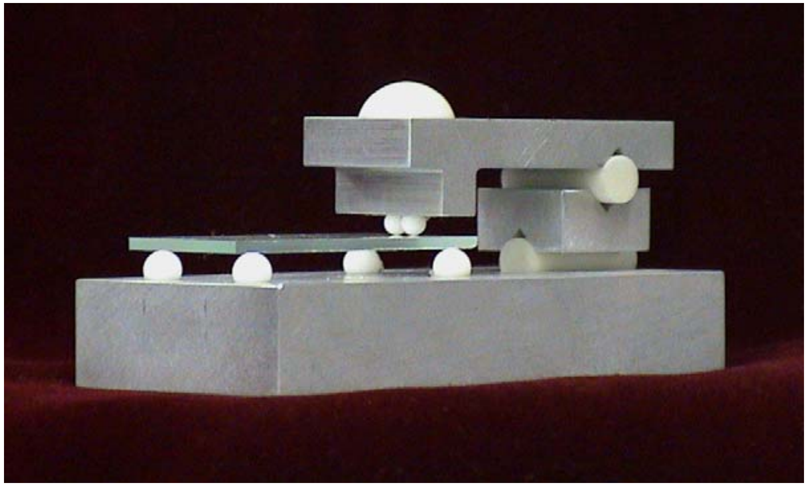
8YSZ – Summary Weibull Statistics

8mol%YSZ	Characteristic strength (MPa) / Weibull modulus Average strength ± Standard Deviation (MPa)		
Number of layers	1	2	4
Room Temperature	345.3 / 4.2 313.7 ± 84.8	182.4 / 4.8 166.4 ± 45.4	222.2 / 3.7 201.5 ± 56.5
600°C	-	-	131.5 / 4.4 127.10 ± 29.4
800°C	208.9 / 5.9 193.9 ± 38.8	175.4 / 8.2 166.2 ± 25.6	160.5 / 4.3 145.5 ± 41.1

Fracture Toughness

Double Torsion Testing





$$K_I = PW_m\left[\frac{3(1+\nu)}{Wt^4\xi}\right]^{1/2}, \xi = 1 - 1.26(t/W) + 2.4(t/W)\exp[-\pi W/(2t)]$$

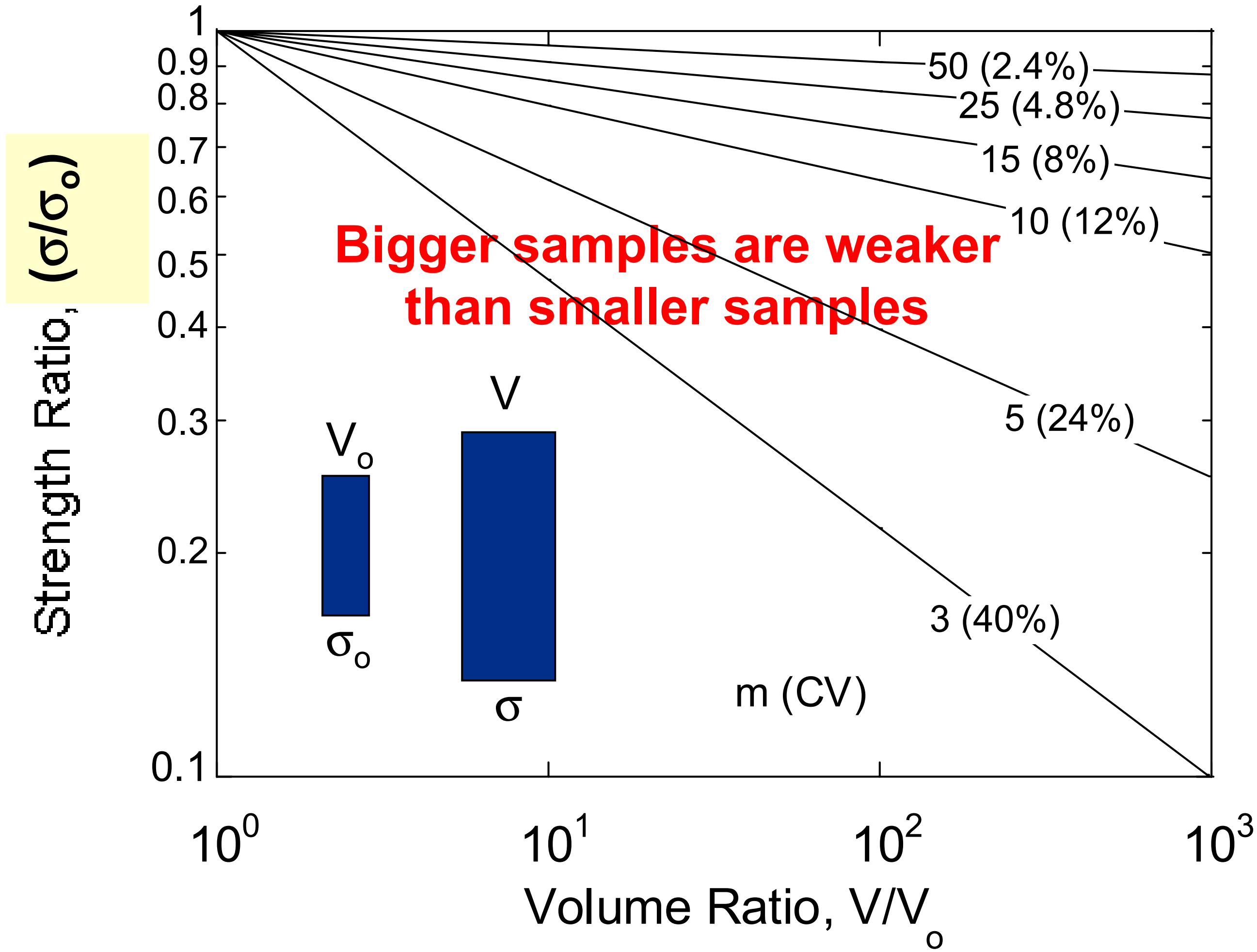
Precracked @ 0.02 mm/min and tested @ 1 mm/min

4 layers	8YSZ	NiO/YSZ
K_{IC}, MPa $m^{1/2}$	1.65 ± 0.02	1.04 ± 0.13

Implications of stochastic nature of strength

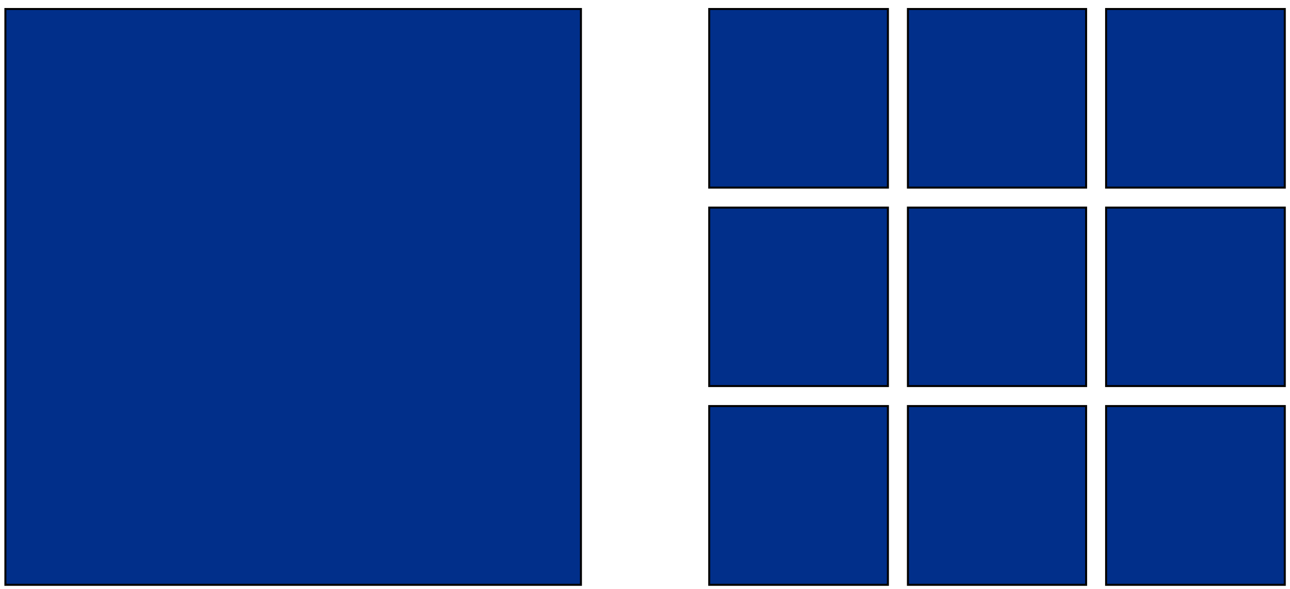
Implications of stochastic nature of strength

If a specimen of size V_o has average strength σ_o, then

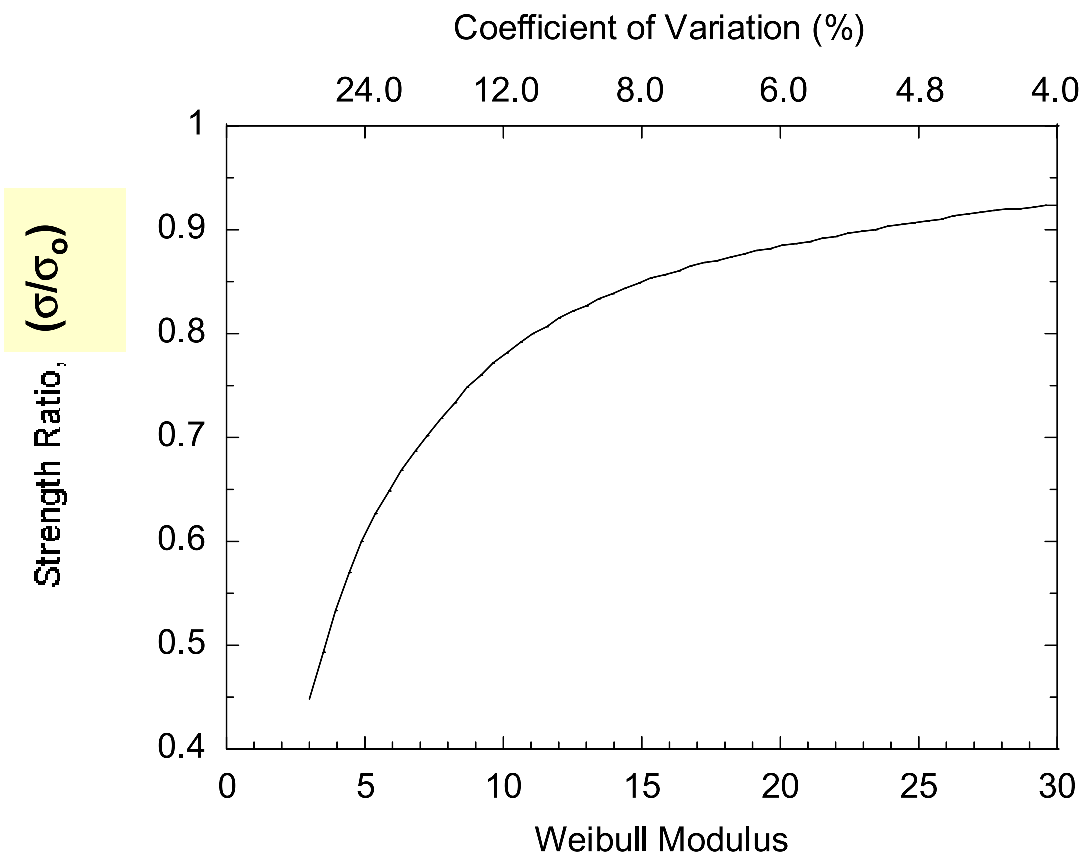


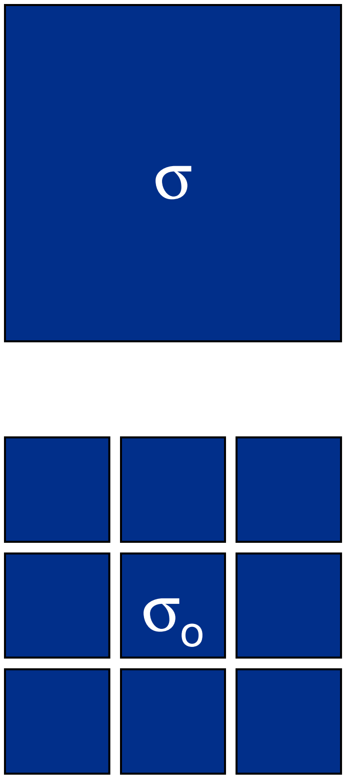
Impact of Stochastic Strength on Manufacturing Decisions

Instead of building large cells, which are weaker than smaller cells, why not using a larger number of smaller cells to cover the same surface area?



Impact of Stochastic Strength on Manufacturing Decisions



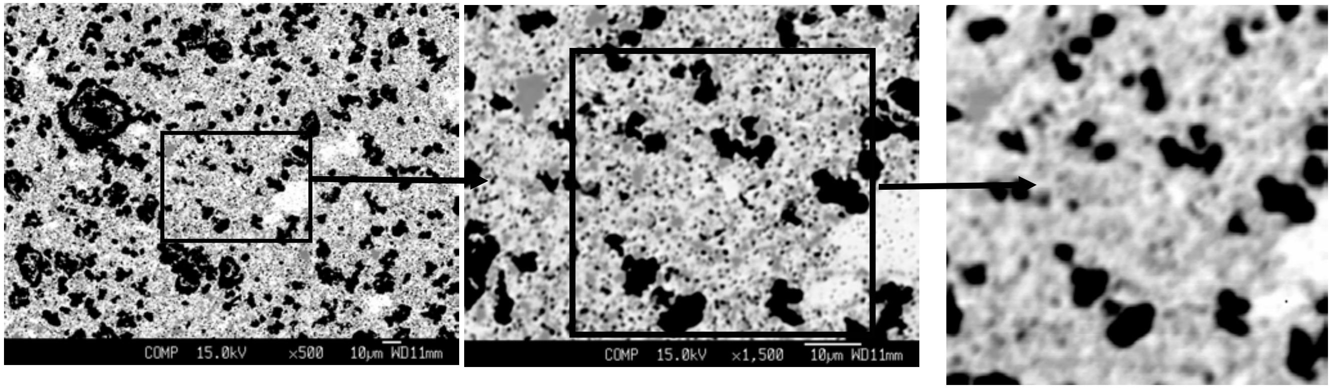


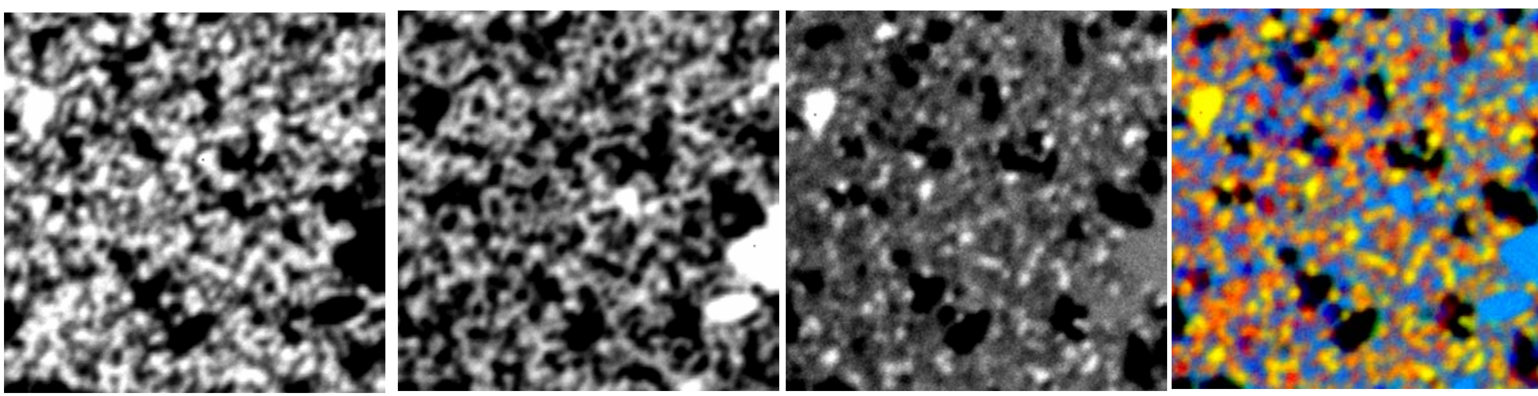
V/Vo = 9

Future Work

- Complete implementation of methodology to predict reliability of model system (geometry, materials).
- Verification of stress predictions using Raman spectroscopy.
- Determination of fracture toughness and adhesion strength of thin coatings.
- Effect of thermal cycling on reliability and durability
- Long-term reliability
- Compositional Analysis and Micromechanical Modeling

Compositional Analysis and Micromechanical Modeling





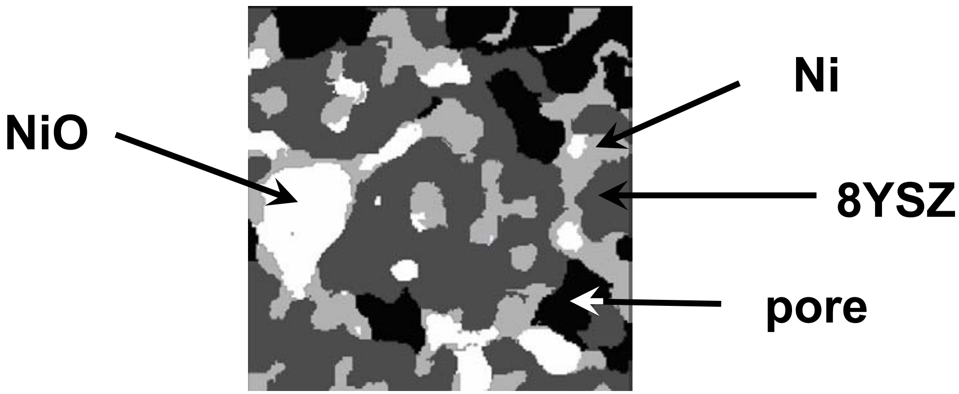
Ni
white – maximum
Black -minimum

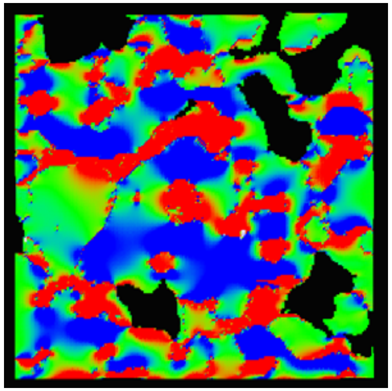
Zr
white – maximum
Black -minimum

O
white – maximum
Black -minimum

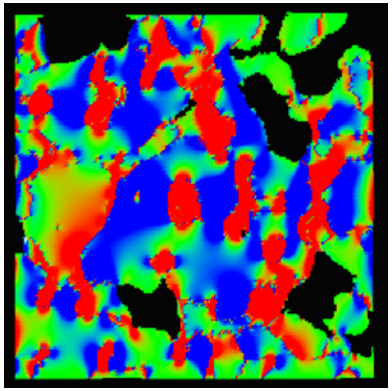
Ni, O, Zr
blue – ZrO
Red – Ni
Yellow – NiO

Compositional Analysis and Micromechanical Stress Modeling





σ_x



σ_y