Reliability & Durability of Materials & Components For Solid Oxide Fuel Cells

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SECA Core Technology Program

Modeling Task

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Outline

- Objectives
- Approach
- Evaluation of Materials & Components
- Summary
- Future Work

Objectives

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

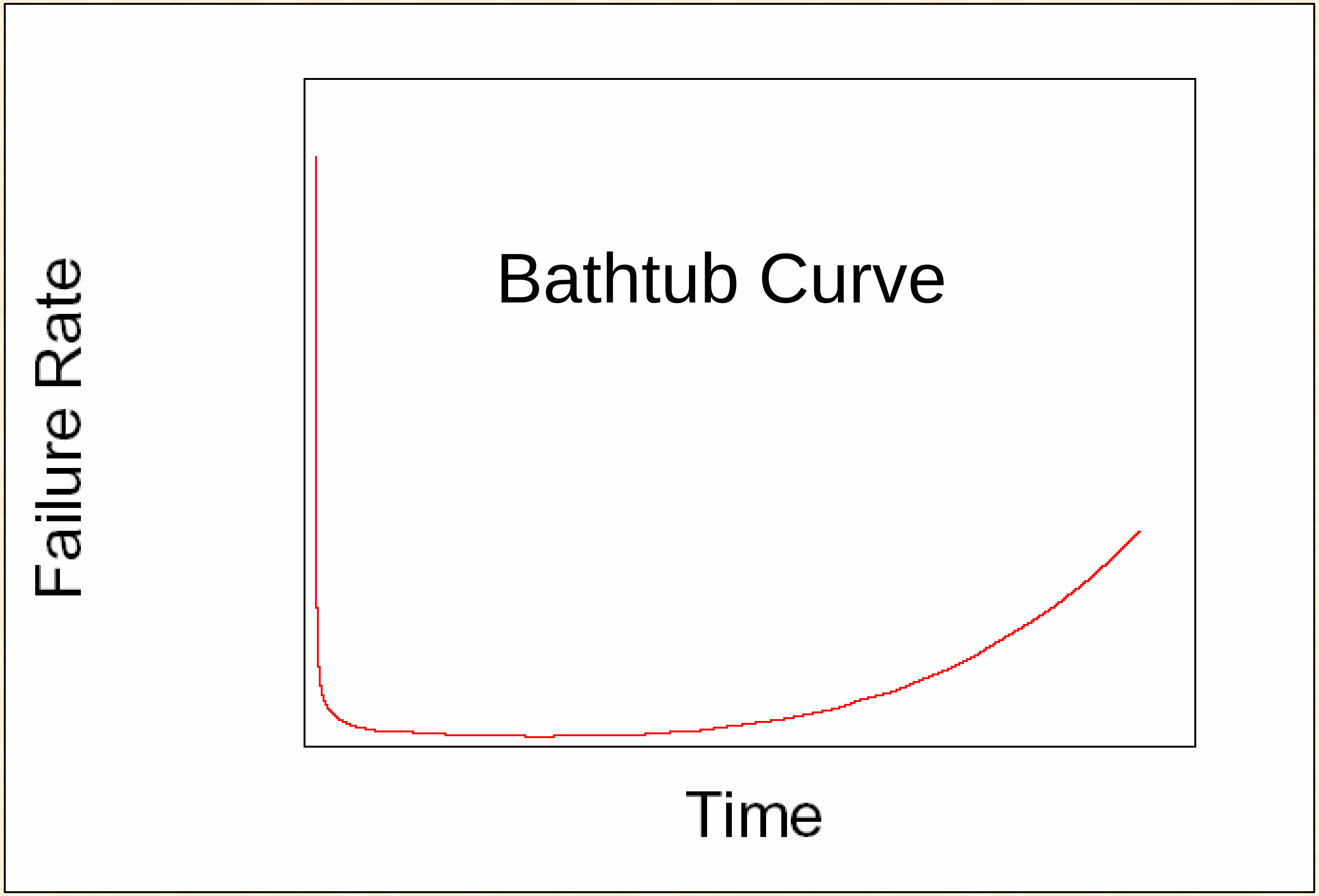
- *To develop/adapt/recommend test techniques to evaluate the properties and behavior of materials and components for SOFC.*
- *To identify and understand the mechanism responsible for the failure of materials and components for SOFCs.*
- *To develop methodologies for predicting the durability and reliability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

Background

The failure rate in complex systems usually follows three stages

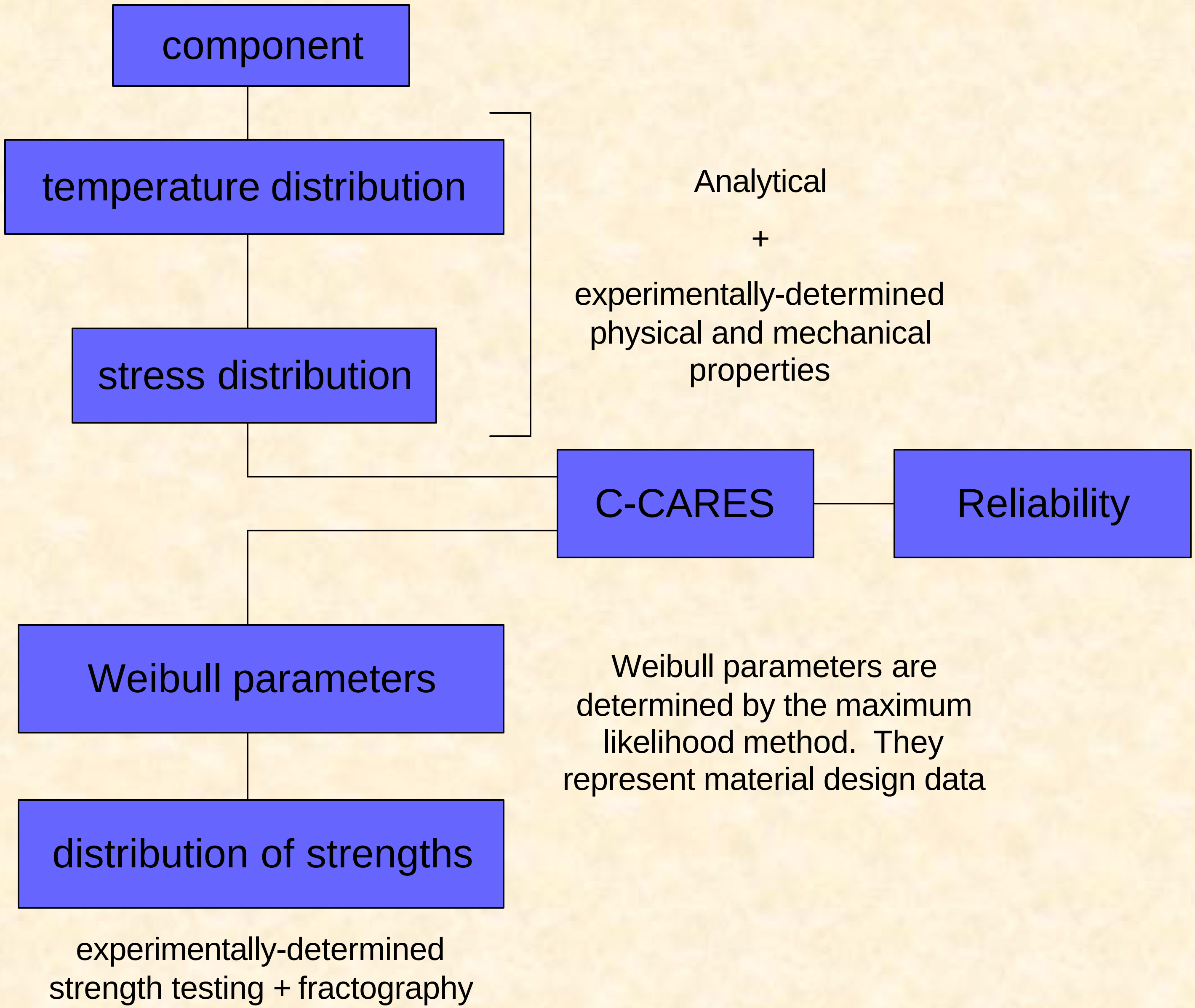
- ***a period with decreasing failure rate at the beginning of service life***
- ***a period with constant failure rate***
- ***Increase of the failure rate at the later part of the life cycle.***

Approach: Background

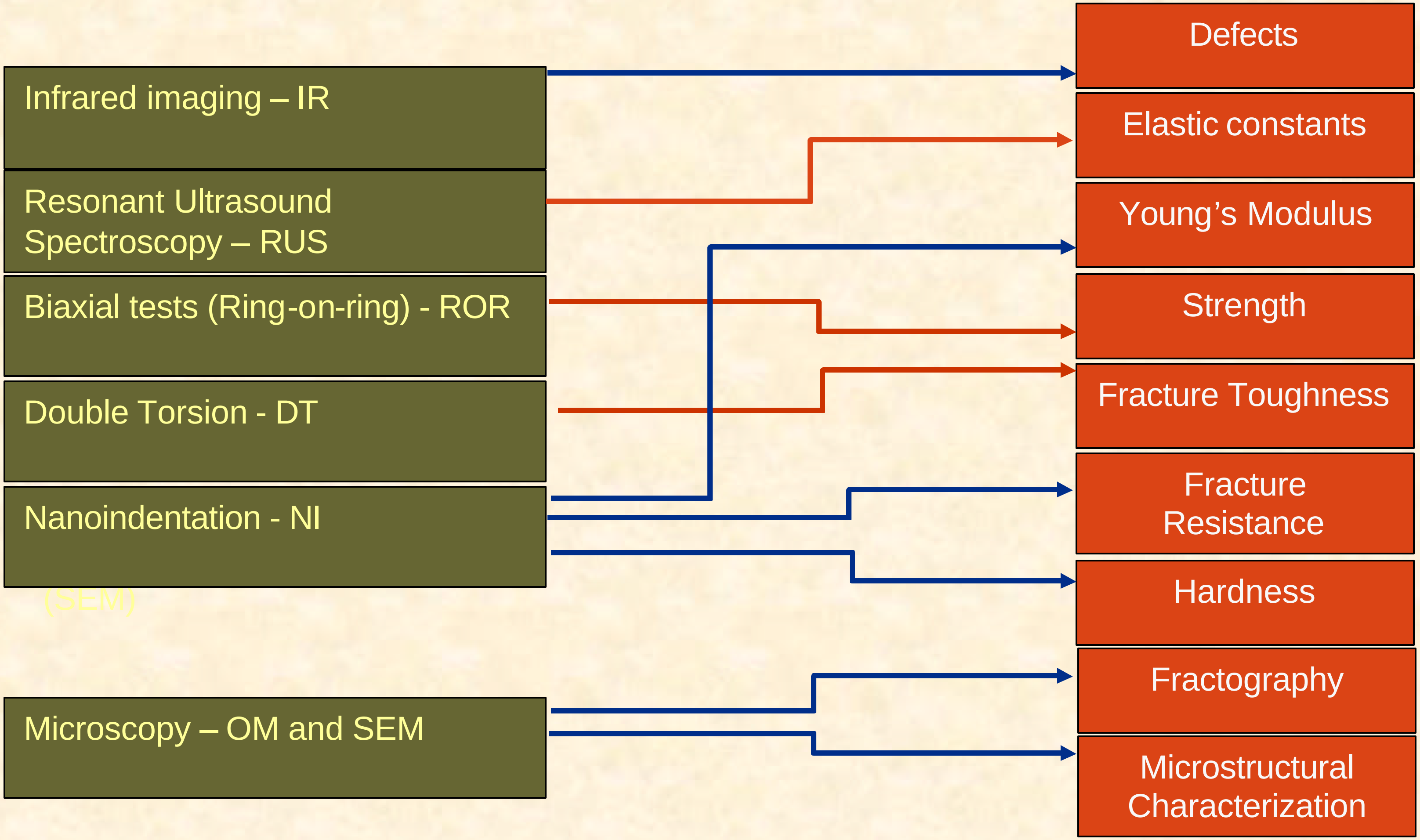


Approach

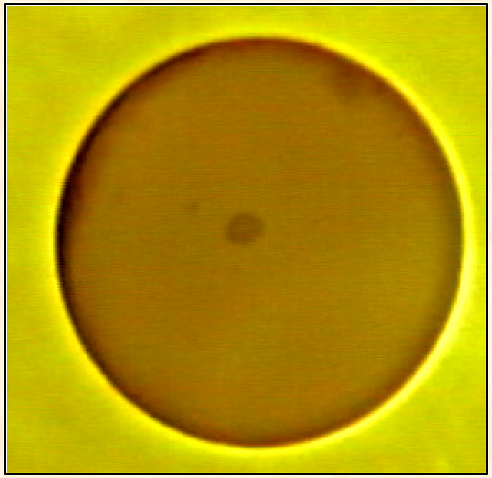
- ***Identification of mechanism that dominate the failure of SOFC materials and components at short times.***
- ***Identification of mechanisms that dominate the failure of SOFC materials and components at long service times/cycles.***
- ***Integrate information into life-prediction methodologies.***

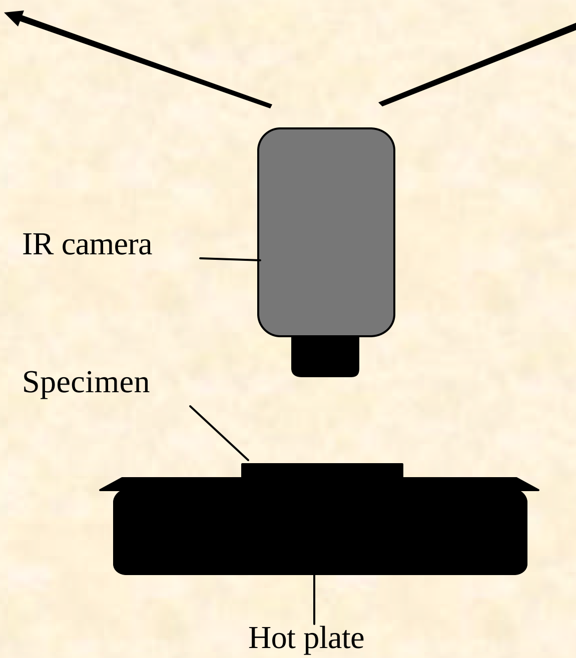

component
temperature distribution
stress distribution
Analytical
+
experimentally-determined physical and mechanical properties
C-CARES
Reliability
Weibull parameters
Weibull parameters are determined by the maximum likelihood method. They represent material design data
distribution of strengths
experimentally-determined strength testing + fractography

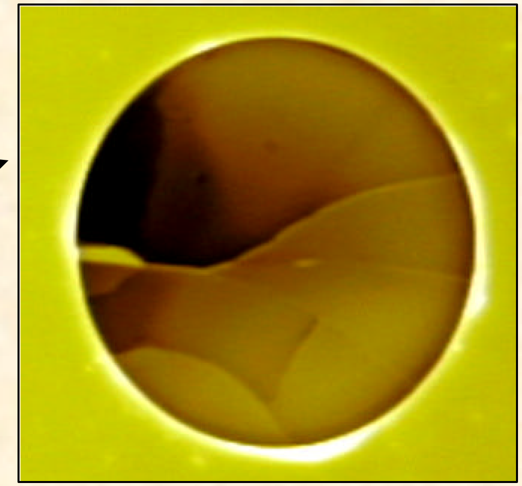
Experimental Techniques for Mechanical Characterization of Materials for SOFC



Infrared Imaging

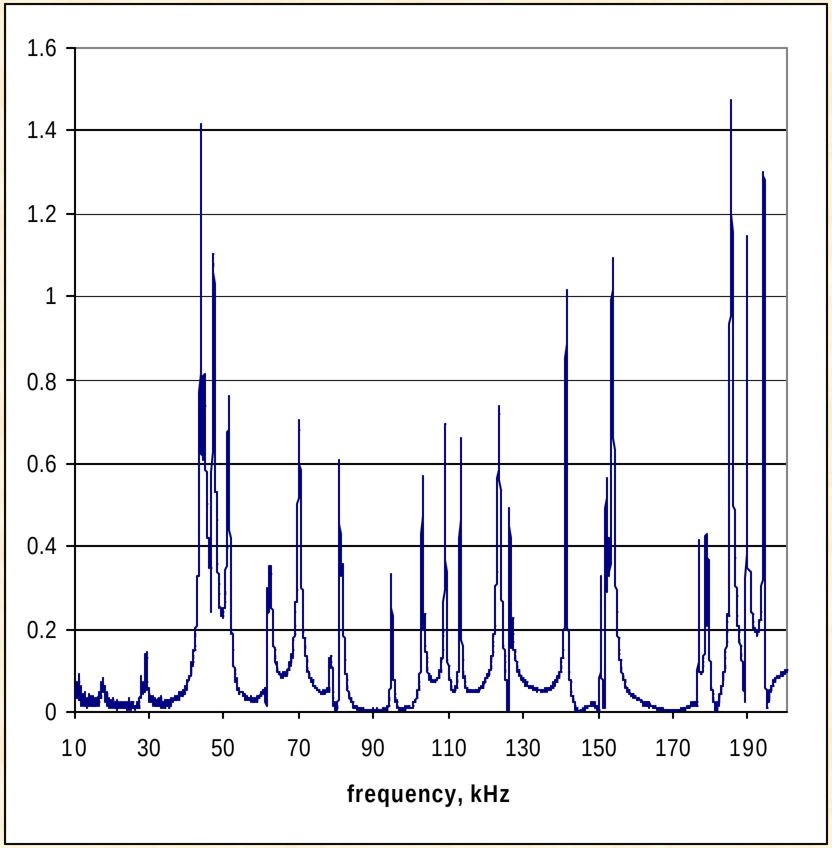






- **Characterization of Defects**
- **Crack propagation Study**

Resonant Ultrasound Spectroscopy



Resonant Ultrasound Spectrum

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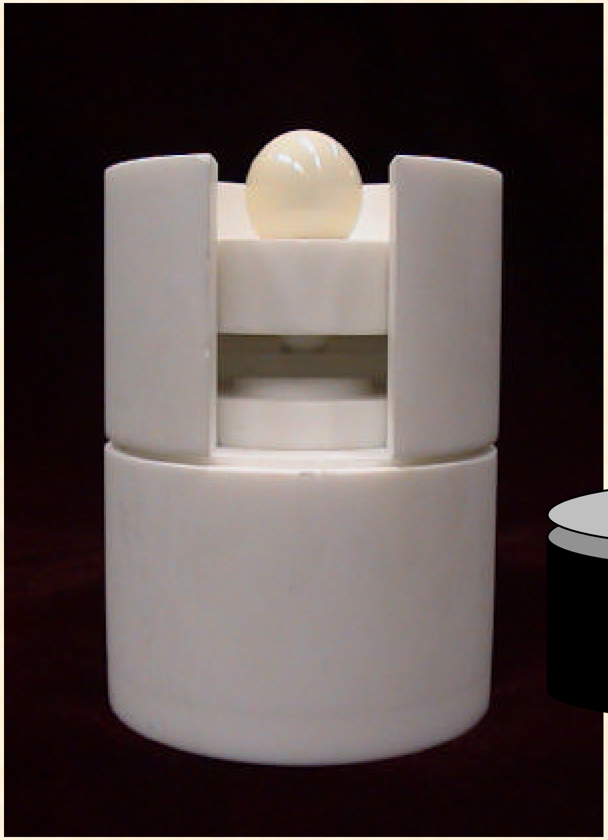
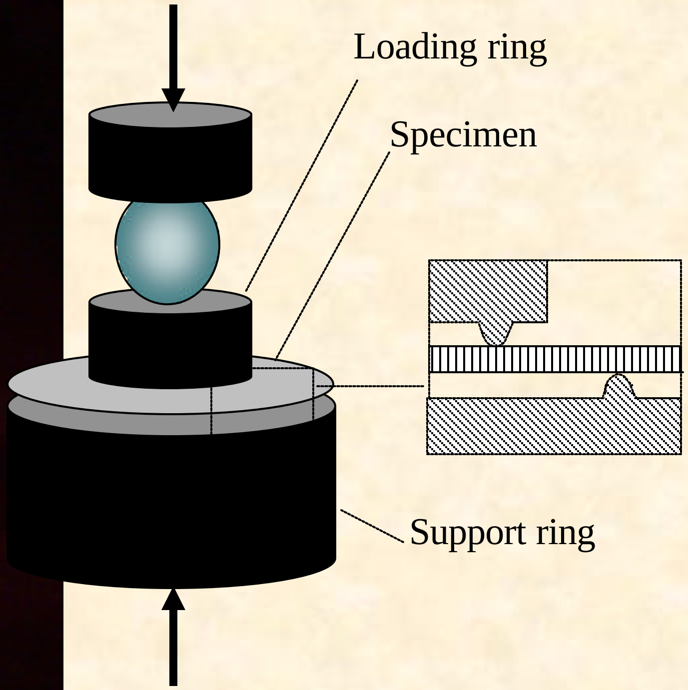
Unique "fingerprint" of each sample.

Depends on:

- ***Geometry (size and shape)***
- ***Elastic properties of the material***
- ***Defects***

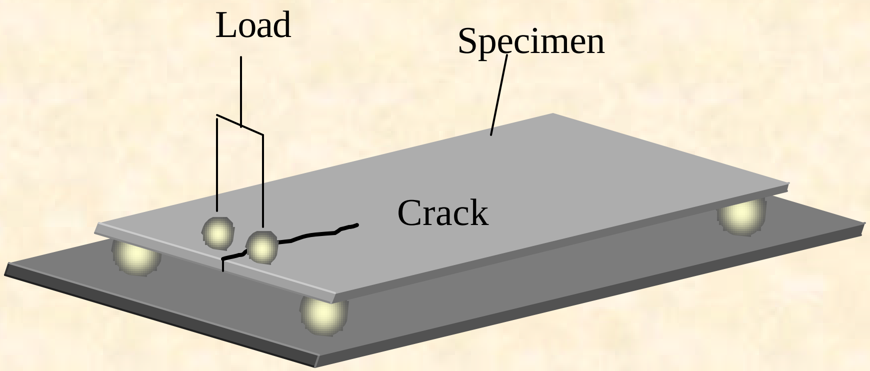
Biaxial Testing – Ring-on-Ring





- Biaxial Strength
- Effect of defects, temperature and environment on strength.

Double torsion test

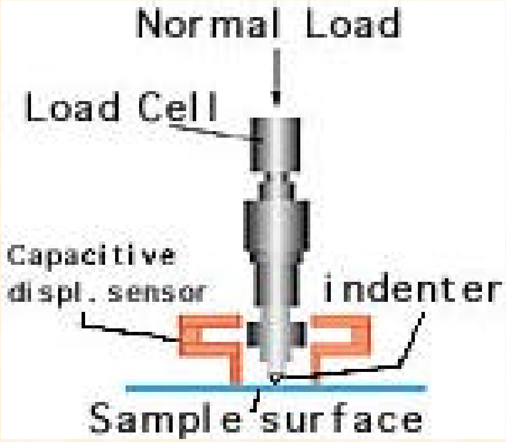


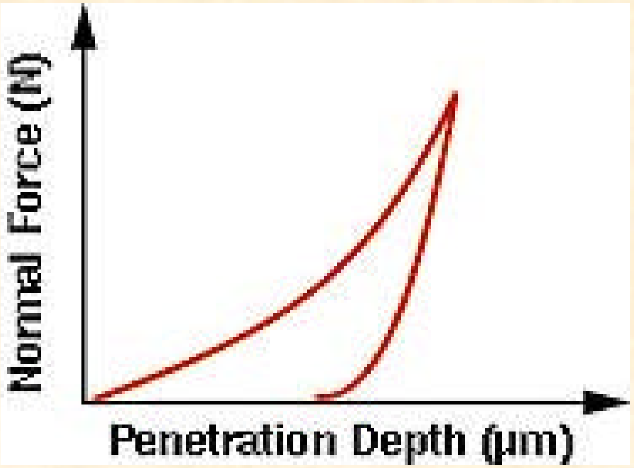
- **Fracture toughness, K_{IC}**
- **Crack Growth**

Indentation Method

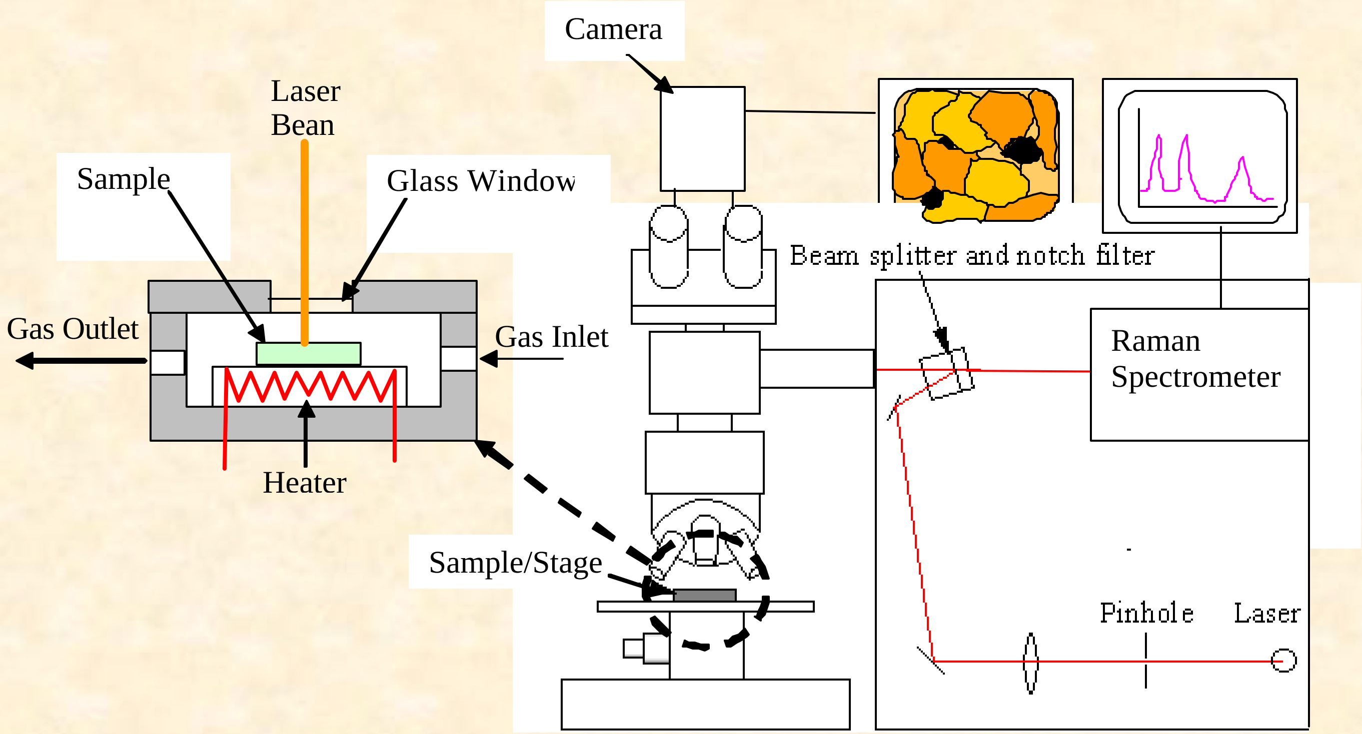
Nano-indentation

- Nanohardness
- Young's Modulus
- Fracture Resistance





Raman Spectroscopy



Characterized Materials

- **Electrolyte:**
  8mol% YSZ

- **Anode:**
  NiO/8mol% YSZ Cermet
  ***Ni/8mol% YSZ Cermet**

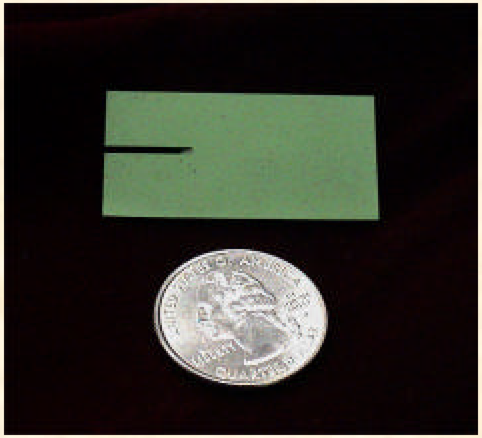
  *** reduced in hydrogen**

- **Cathode:**
  LSM

Characterized Materials

- **Disks 1” Æ**
  - **Resonant Ultrasound Spectroscopiy**
  - **Infrared Imaging**
  - **Biaxial strength**
  - **Nanoindentation**
  - **Raman Spectroscopy**

- **Notched Plates**
  Fracture toughness



Characterization of Electrolyte Materials

8%mol YSZ - porosity: 8%

<u>Elastic Properties at Room Temperature:</u>

RUS: E =175±8 GPa

G = 67± 3 GPa

ν =0.32 ± 0.01

Nanoindentation: displacement »800 nm

surface	E =196±6 GPa	H =13±0.5 GPa
cross-section	E =176±4 GPa	H =12.6 ±0.5 GPa

A. Selcuk & A. Atkinson, J.Euro.Ceram.Soc. 17 (1997)

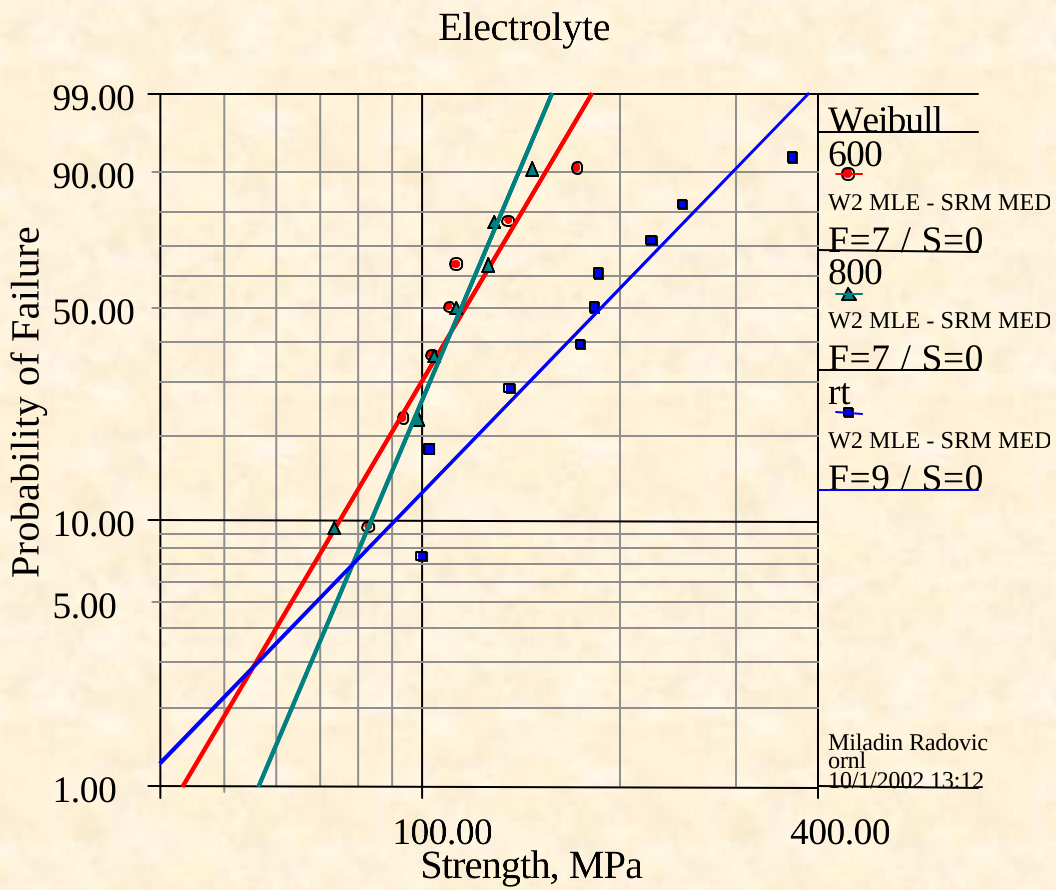
8% porosity E= 176 GPa, G=67 GPa

fully dense: E=220 GPa, G=83 GPa

Impulse excitation technique

Characterization of Electrolyte Materials

Biaxial Strength



600°C $\beta1=4.2723$, $\eta1=127.5248$ $\sigma = 116.385 \pm 27.682$
800°C $\beta2=5.9528$, $\eta2=122.3148$ $\sigma = 113.289 \pm 22.242$
25°C $\beta3=2.6050$, $\eta3=216.2506$ $\sigma = 191.64 \pm 78.024$

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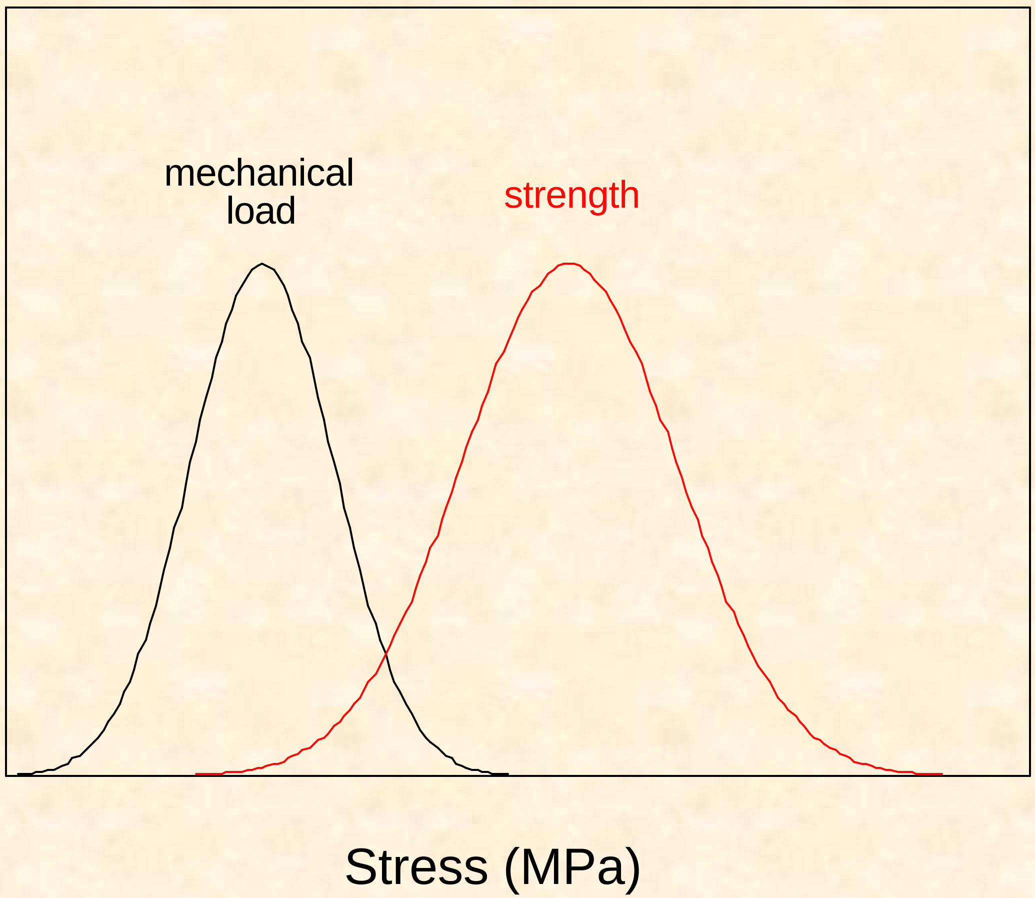
Distribution of Strengths

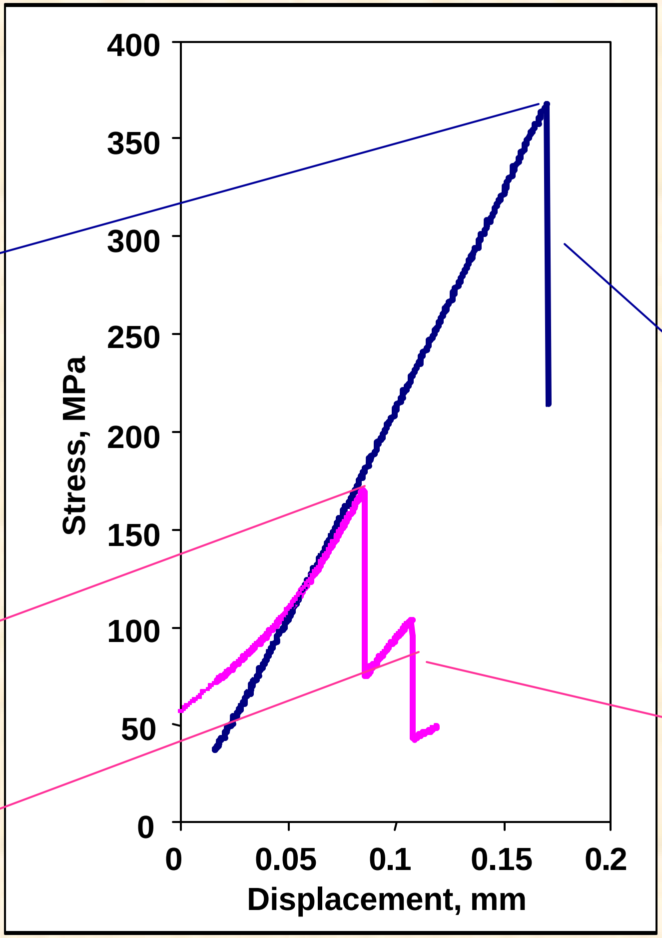


Characterization of Electrolyte Materials

Failure with branched crack formation from the loading ring area

Primary crack formation

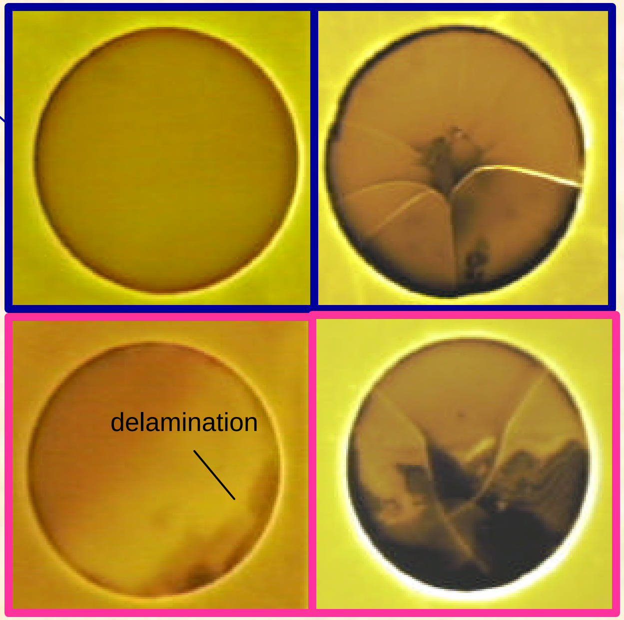
Secondary crack formation and delamination



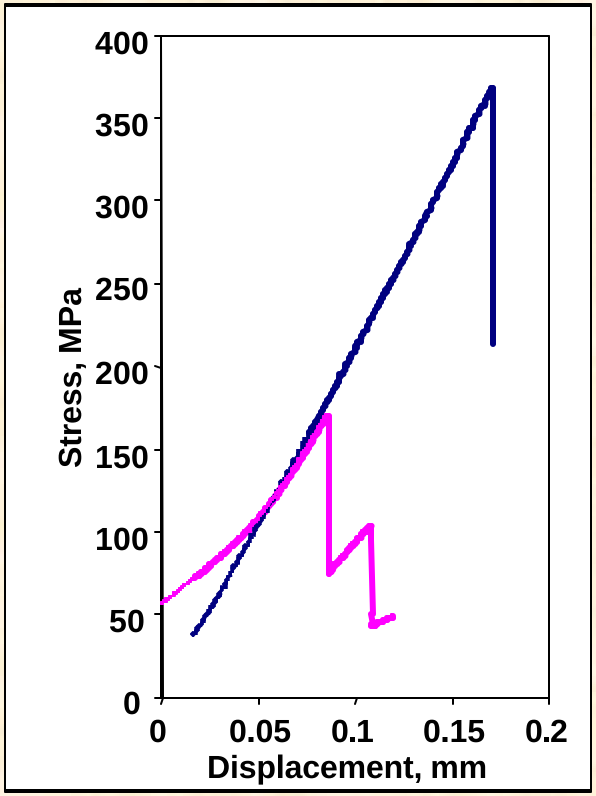
Infrared images

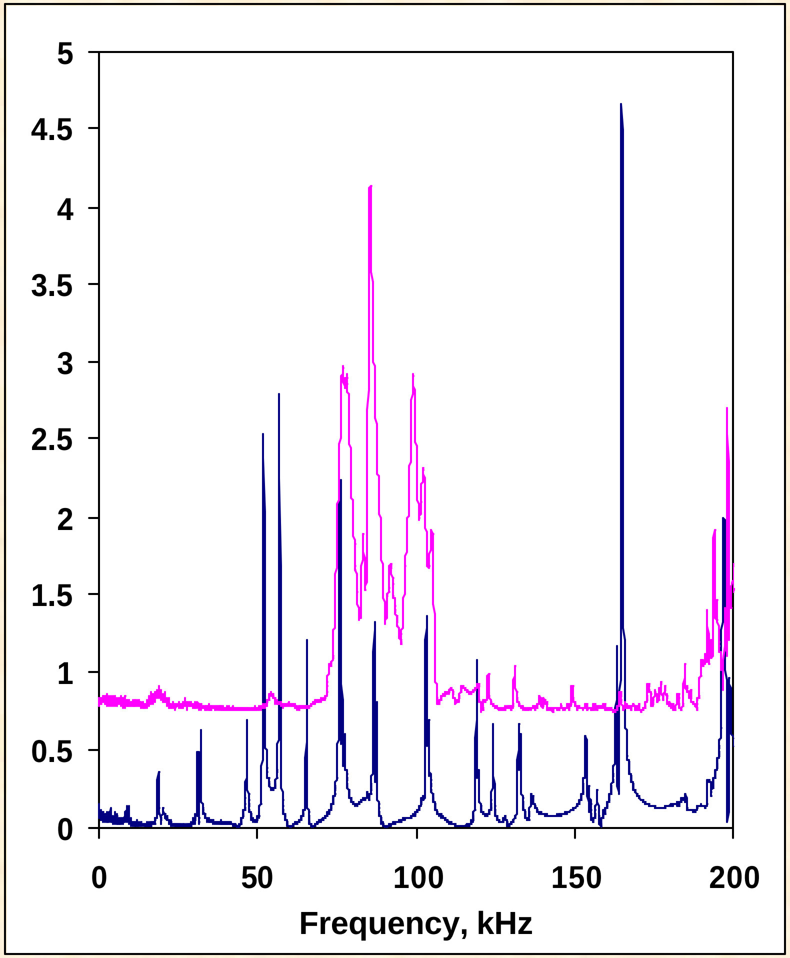
Before testing

After testing



Characterization of Electrolyte Materials

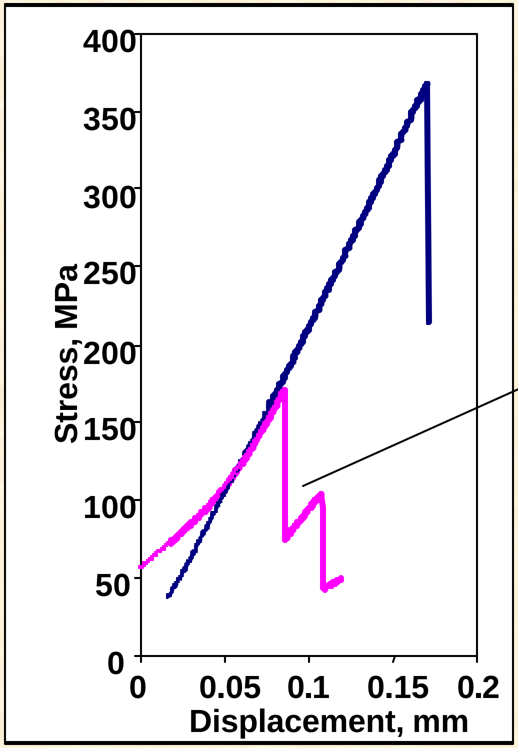


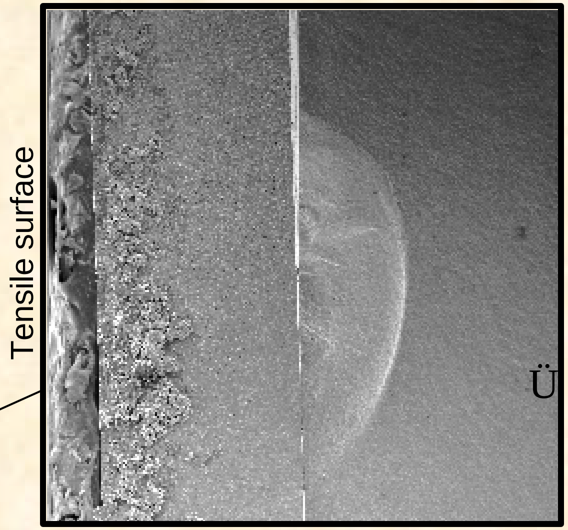


Blue: “good” spectra –sharp peaks

Pink: spectra with peak splitting and shifting associated with defects in sample

Characterization of Electrolyte Materials





SEM - Fractography

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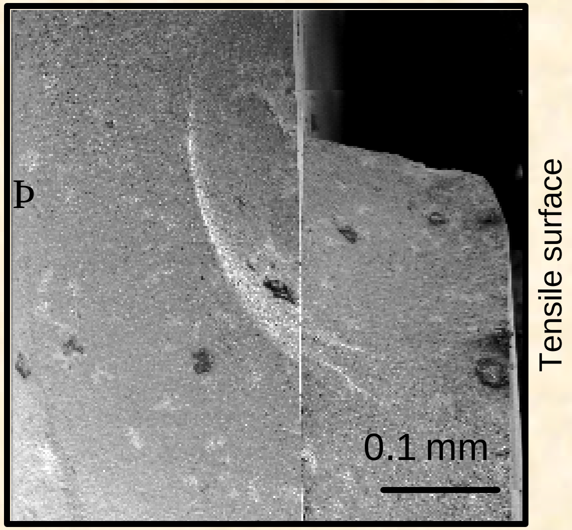


Characterization of Anode Materials

NiO/YSZ Cermet– ORNL (2, 4 and 6 layers; 0.5, 1 and 1.5 mm thick) – 30% porosity

NiO/YSZ Cermet – NexTech (multilayer, 1mm thick)

Elastic Properties at Room Temperature:

RUS*:

ORNL:	E =103±6 GPa	G = 40± 2.5 GPa	ν =0.29 ± 0.03
NexTech:	E =106±6 GPa	G = 41± 2.4 GPa	ν =0.29 ± 0.01

A. Selcuk & A. Atkinson, J.Euro.Ceram.Soc. 17 (1997)

Impulse excitation technique – characterized anode 75mol%NiO/YSZ materials up to 14% porosity

Extrapolated data for 30 % porosity:

Exponential law $M=M_o exp(-bP)$: E= 99 GPa, G=38 GPa

Linear law $M=M_o(1-bP)$: E= 76 GPa, G=30 GPa

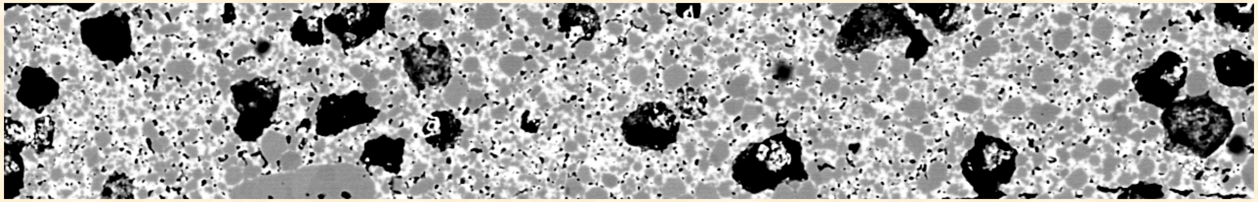
Non-linear law $M=M_o(1-(bP)/(1+(b-1)P)$: E= 99 GPa, G=38 GPa

Composite Sphere Method (CSM) $M=M_o(1-P^2)/(1+bP)$: E= 83 GPa, G=32 GPa

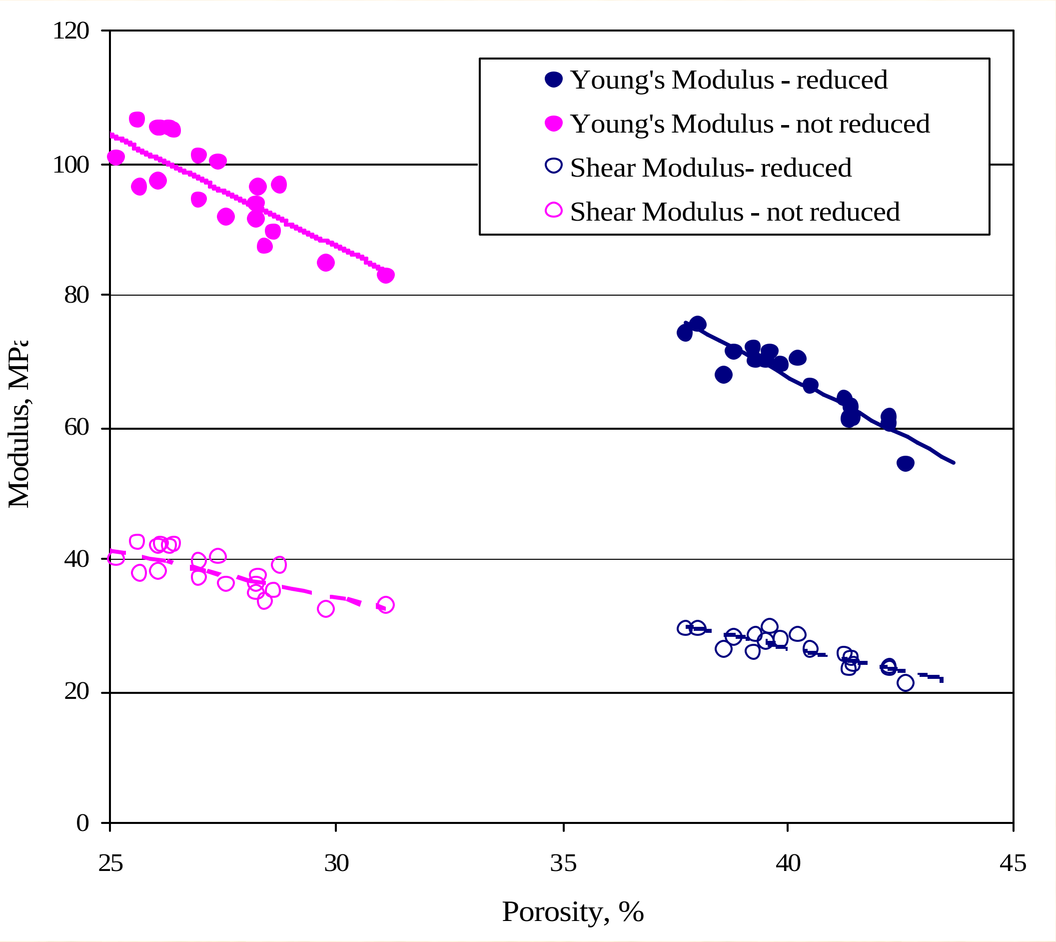
Microstructural modeling

Reduced anode, SEM BSE

unreduced anode, SEM BSE



Characterization of Anode Materials



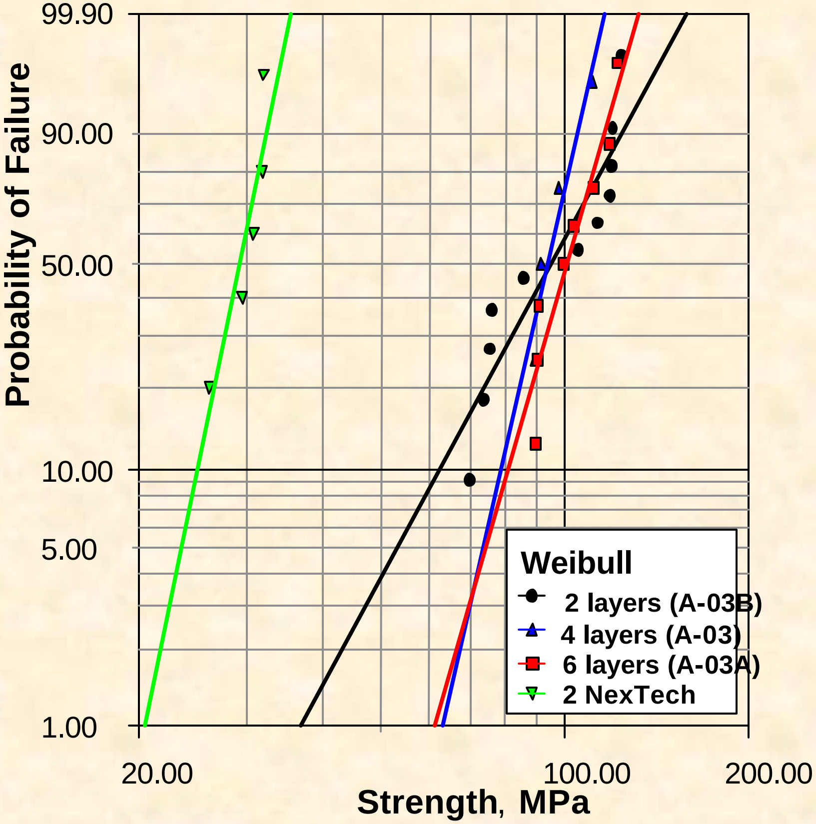
Characterization of Anode Materials

Biaxial Strength at Room Temperature:

NiO/8mol% YSZ Cermet– ORNL reduced in 4% H_2 at 600^{o}C for 4 h

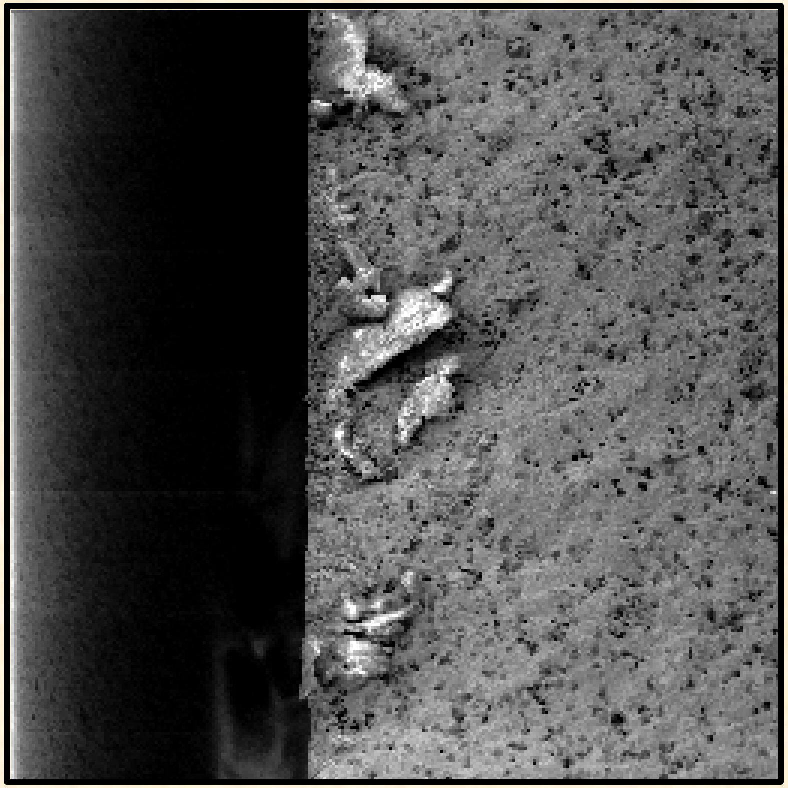
NiO/8mol% YSZ Cermet – NexTech reduced in hydrogen

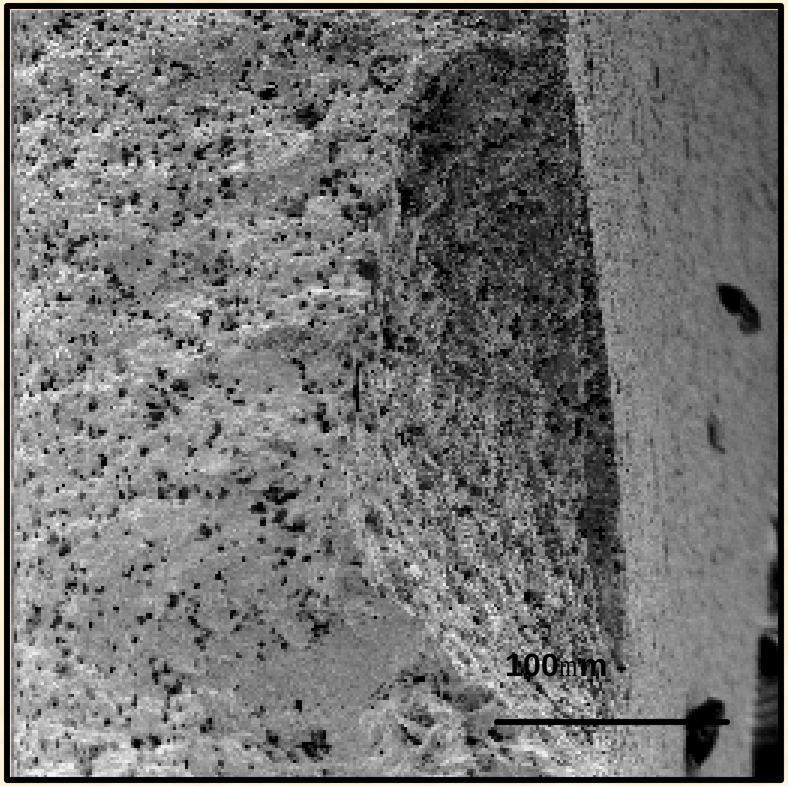
Sample	s_{ave}, MPa	Weibull Distribution	
		s_0, MPa	m
NexTech	24.5±5.9	27.03	3.96
ORNL 2 layers	109.9±14.0	122.3	5.51
ORNL 4 layers	98.5±12.7	104.7	7.25
ORNL 6 layers	102.9±12	119.9	6.16



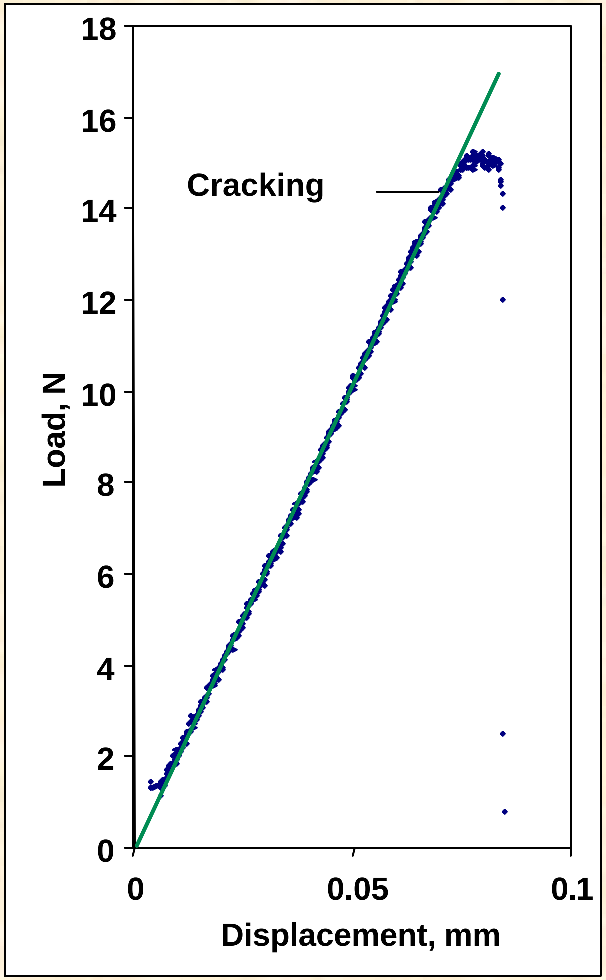
Characterization of Anode Materials

NiO/YSZ Cermet– ORNL (4 layers; 1 mm thick) – 30% porosity



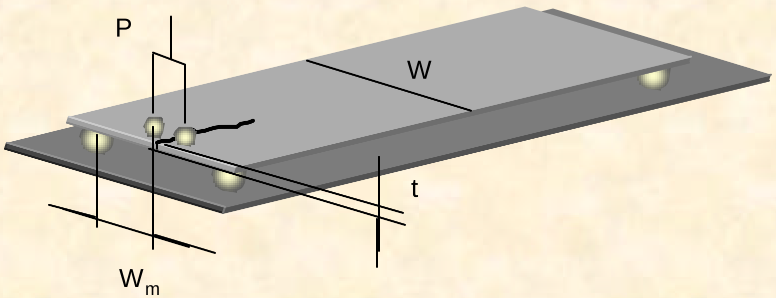


Characterization of Anode Materials



Double Torsion Testing at Room Temperature:

NiO/YSZ Cermet– ORNL



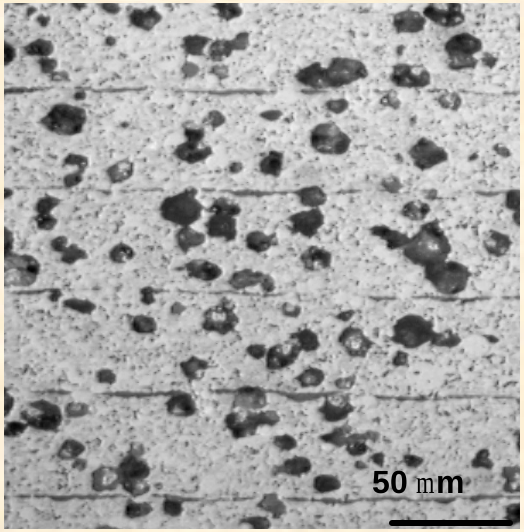
$$K_I = PW_m\left[\frac{3(1+\nu)}{Wt^4\xi}\right]^{1/2}, \xi = 1 - 1.26(t/W) + 2.4(t/W)\exp[-\pi W/(2t)]$$

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

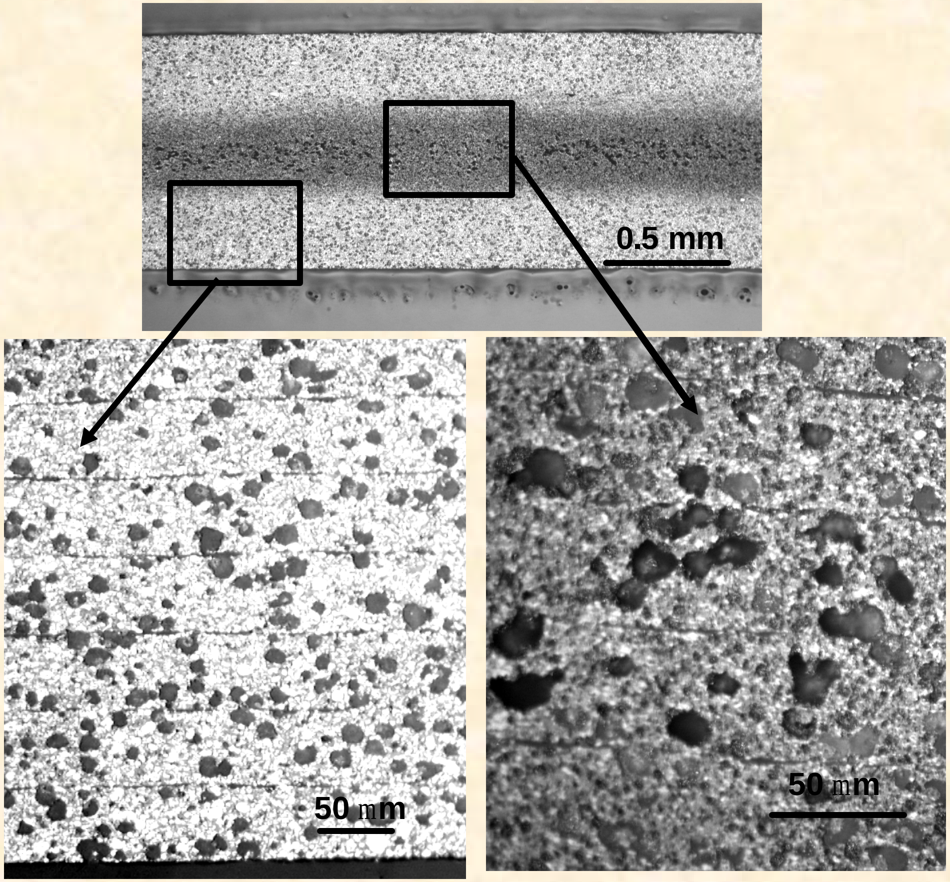
K_{IC}= 1.05±0.14 MPam$^{1/2}$

Characterization of Anode Materials

Optical Microscopy

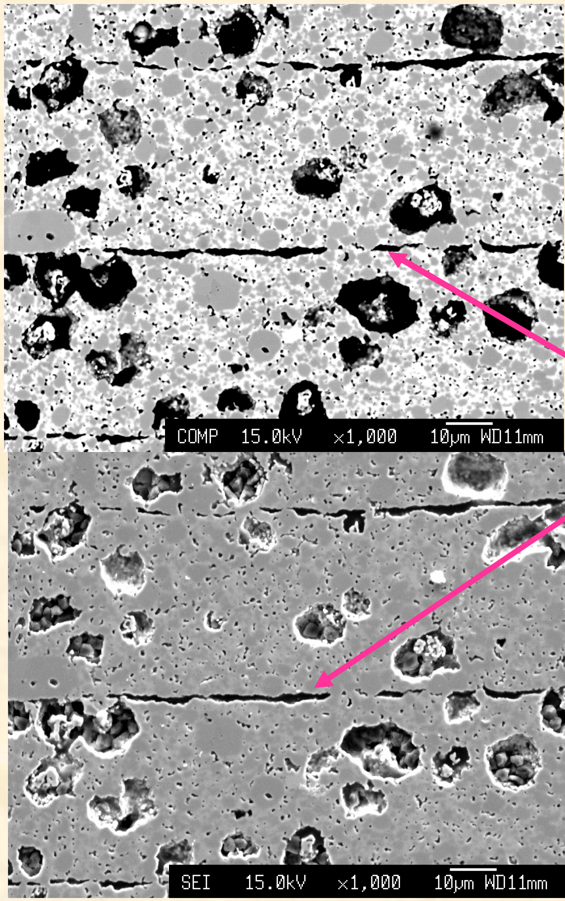


NiO/8mol% YSZ Cermet – NexTech



NiO/8mol% YSZ Cermet– NexTech
<u>reduced in hydrogen</u>

Characterization of Anode Materials



NiO/8mol% YSZ Cermet – NexTech

SEM

Back Scattered

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Interlaminar porosity

SE

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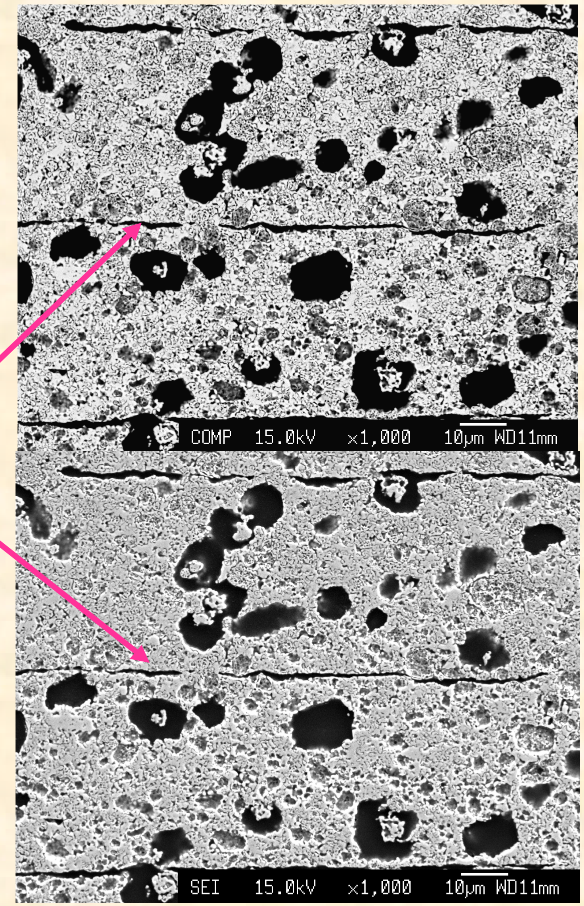
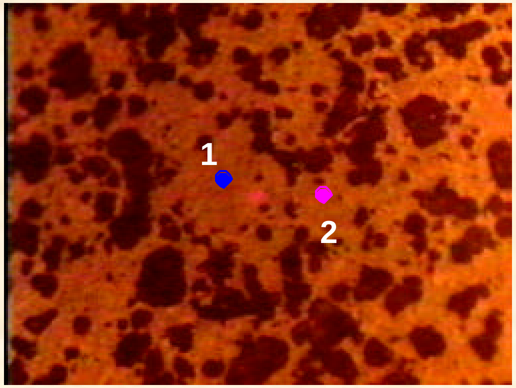
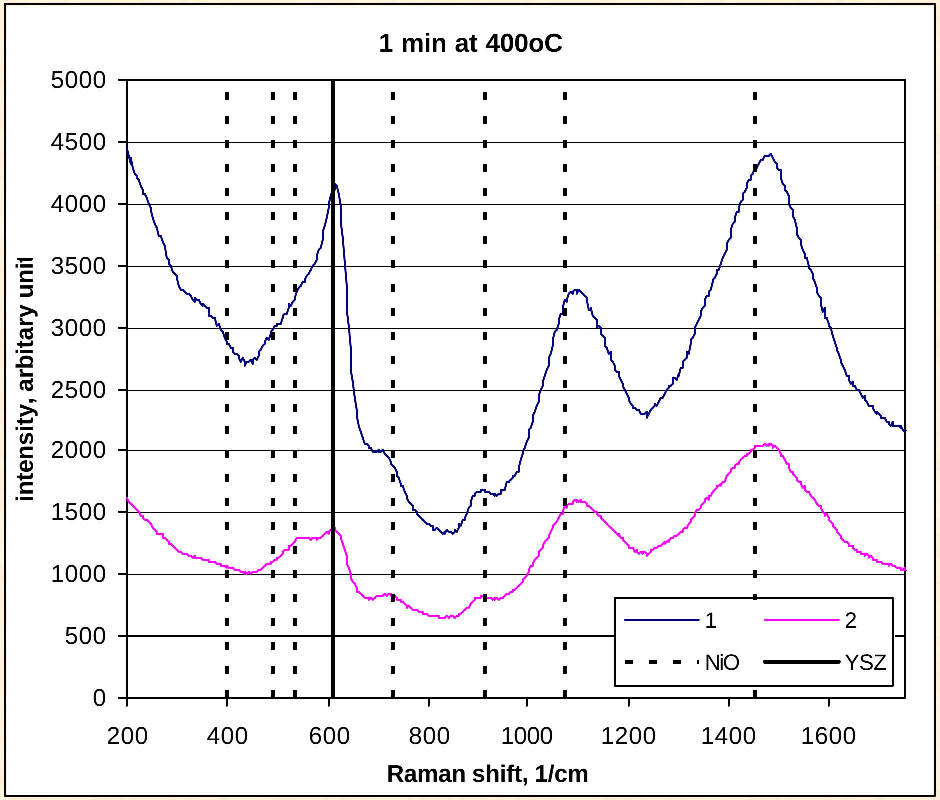


NiO/8mol% YSZ Cermet– NexTech
<u>reduced in hydrogen</u>

Microstress measurements





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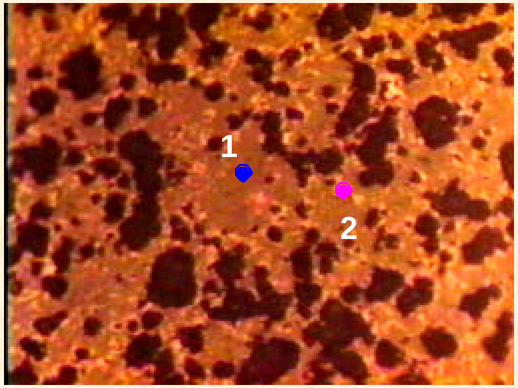

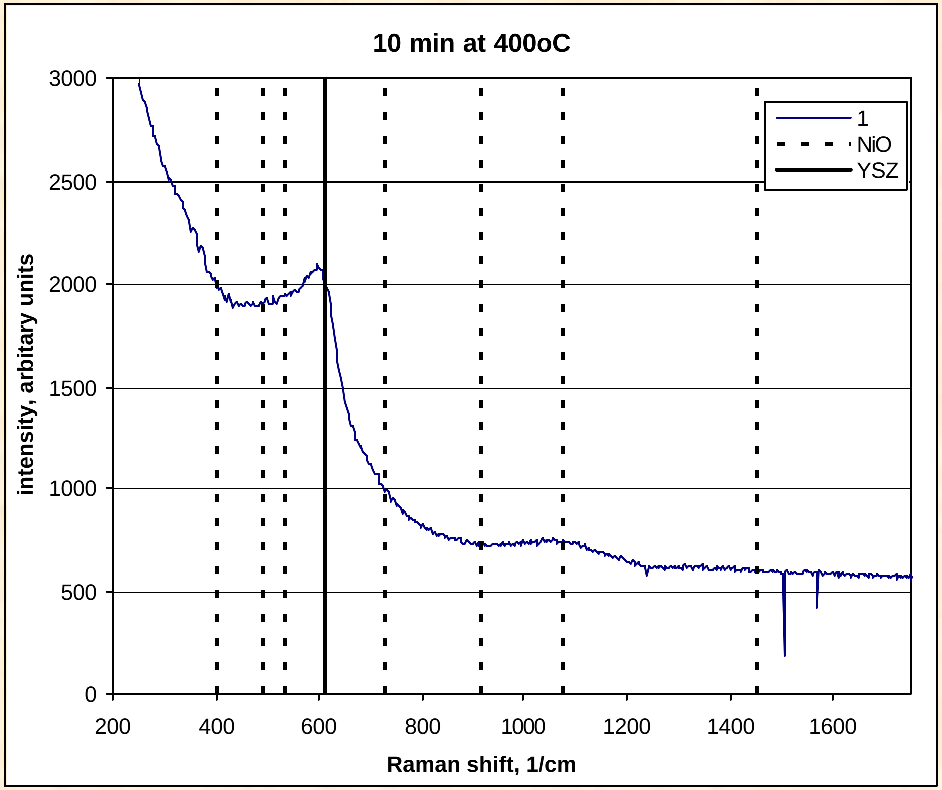

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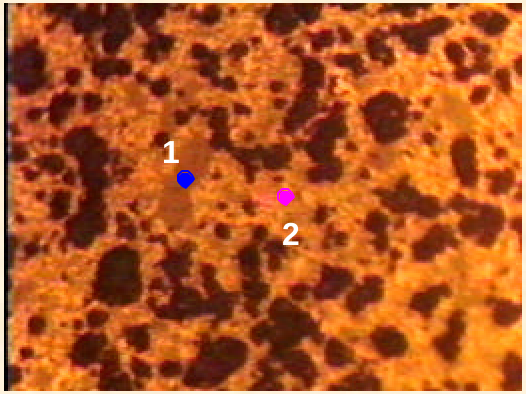
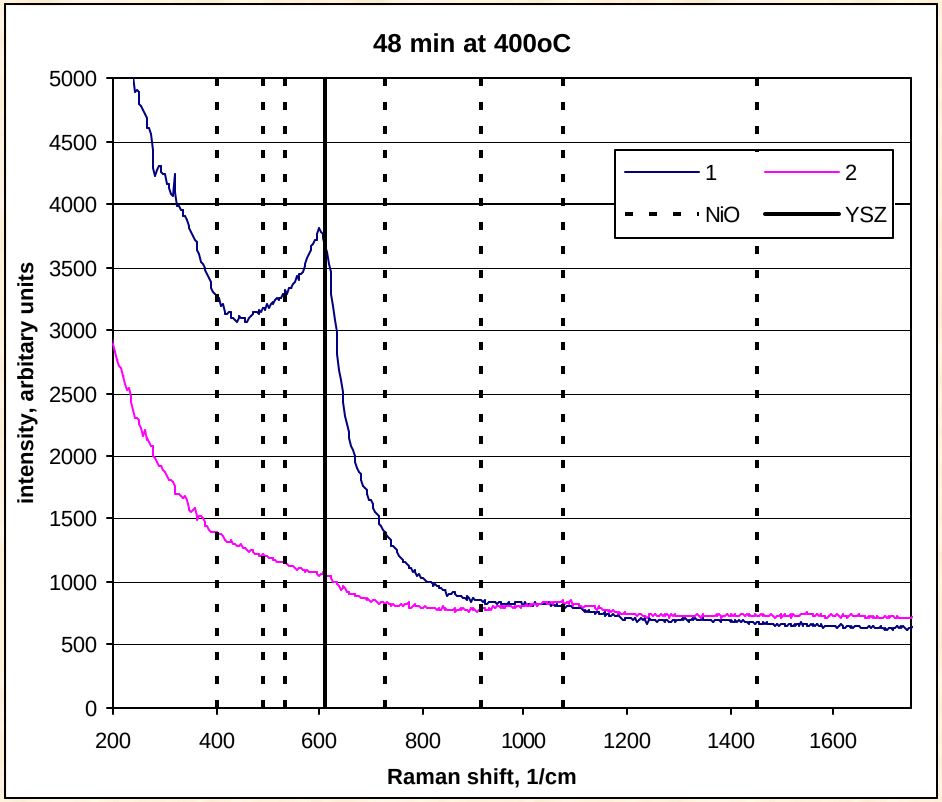
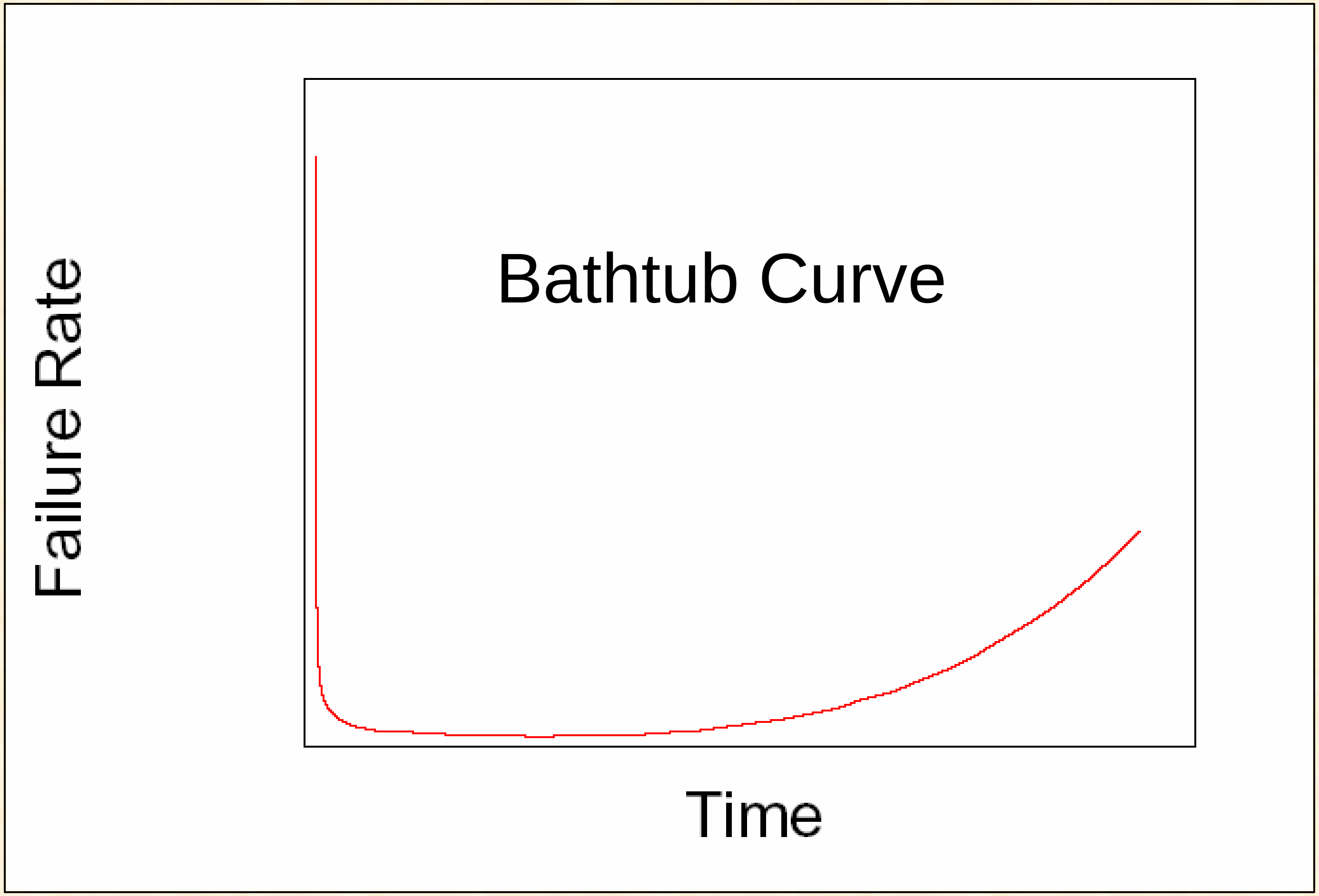




Summary

- NDE techniques (infrared imaging, RUS) have been adapted/used to detect defects (e.g. delamination, voids…) in SOFC materials. Powerful tools for quality control.
- Test methods have been adapted to determine elastic properties, in-plane biaxial strength and fracture toughness of SOFC at RT and elevated temperatures, in air or controlled environments.
- Fractographic analysis were used to identify defects and mechanisms responsible for failure of SOFC materials.
- Methodology can help industrial teams address short term failures to increase reliability of SOFCs. It also constitutes the basis for the evaluation of long-term behavior of these materials.

Summary

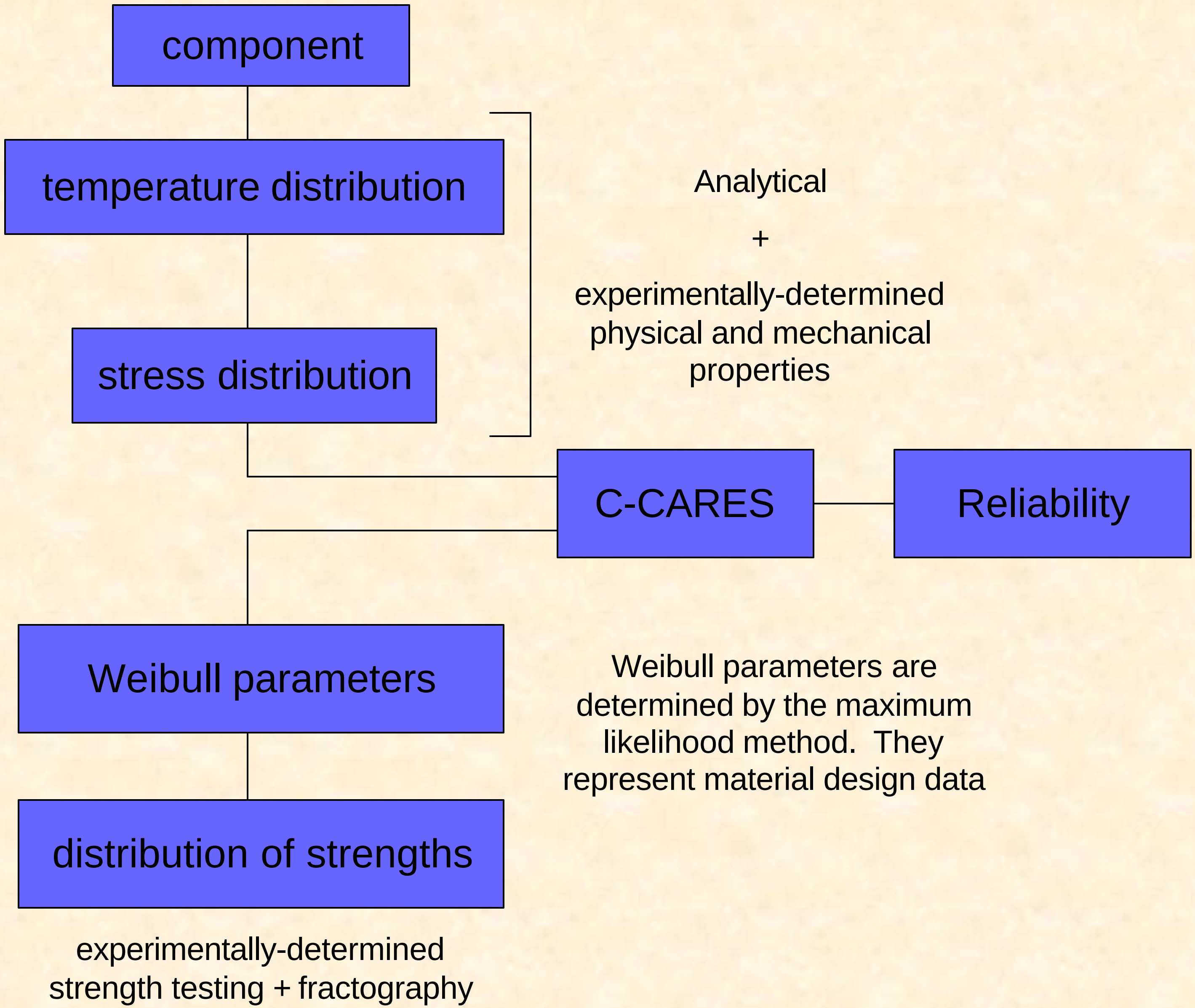


Current and Future Work

- Characterization of SOFC materials at high temperatures (strength, fracture toughness, elastic properties) in air/controlled environments.
- Effect of porosity and pore size on elastic properties, strength and fracture toughness. OOF modeling.
- Identification of defects and microstructural features responsible for failure.
- Long term reliability, transient, time-dependent phenomena.

Interaction with Task and Program Members

- Integration of interfacial modes of failure in probabilistic life prediction framework.
- Effect of chemical gradients on physical properties (e.g. – elastic constants, strength).
- Integration with PNNL modeling work (temperature and stress distributions).


component
temperature distribution
stress distribution
Analytical
+
experimentally-determined physical and mechanical properties
C-CARES
Reliability
Weibull parameters
Weibull parameters are determined by the maximum likelihood method. They represent material design data
distribution of strengths
experimentally-determined strength testing + fractography