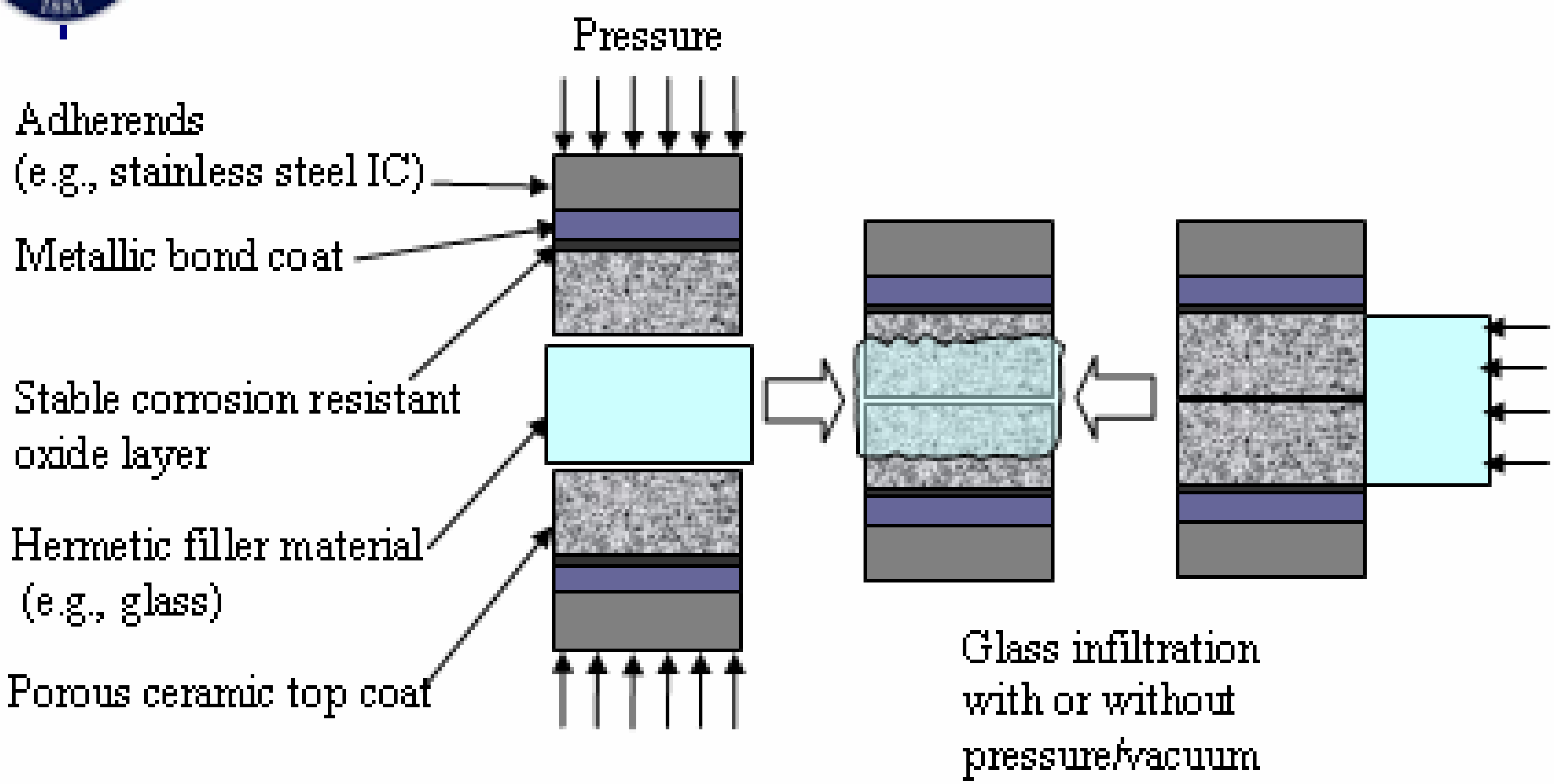
Integrated Composite Seal Concept



Potential Advantages

- Gradual transition of CTE and elastic properties
  - Higher resistance to thermal mechanical failure
- Corrosion resistance interfacial reaction layers (chromia or alumina)
  - Better bonding strength
  - Long-term oxidation resistance
  - Low electrical conductivity
- Near hermetic seal (with the help of glass)
- Larger design space (substrate + bond coat + top coat + glass)
- Integrated design & low cost fabrication

Objectives

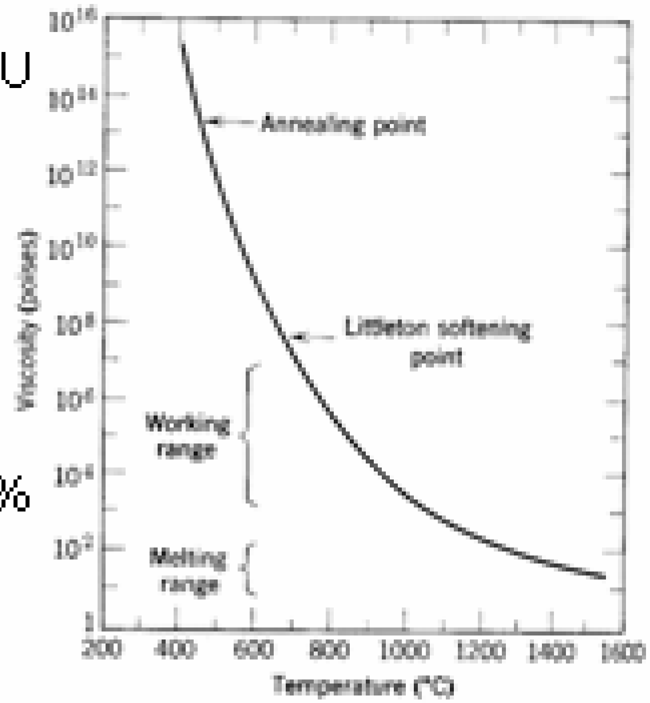
- The vision for this two-phase effort is to create a unique high-temperature composite solid oxide fuel cell (SOFC) seal and the associated design methodologies to support the SECA Industrial Teams in their efforts to design, manufacture, and market reliable SOFC power generation systems. To that end, the objectives of the program are to prove a conceived composite structure and to demonstrate a design methodology.

- Phase I objective: to demonstrate the viability of the composite seal concept using small scale coupon and single cell experiments

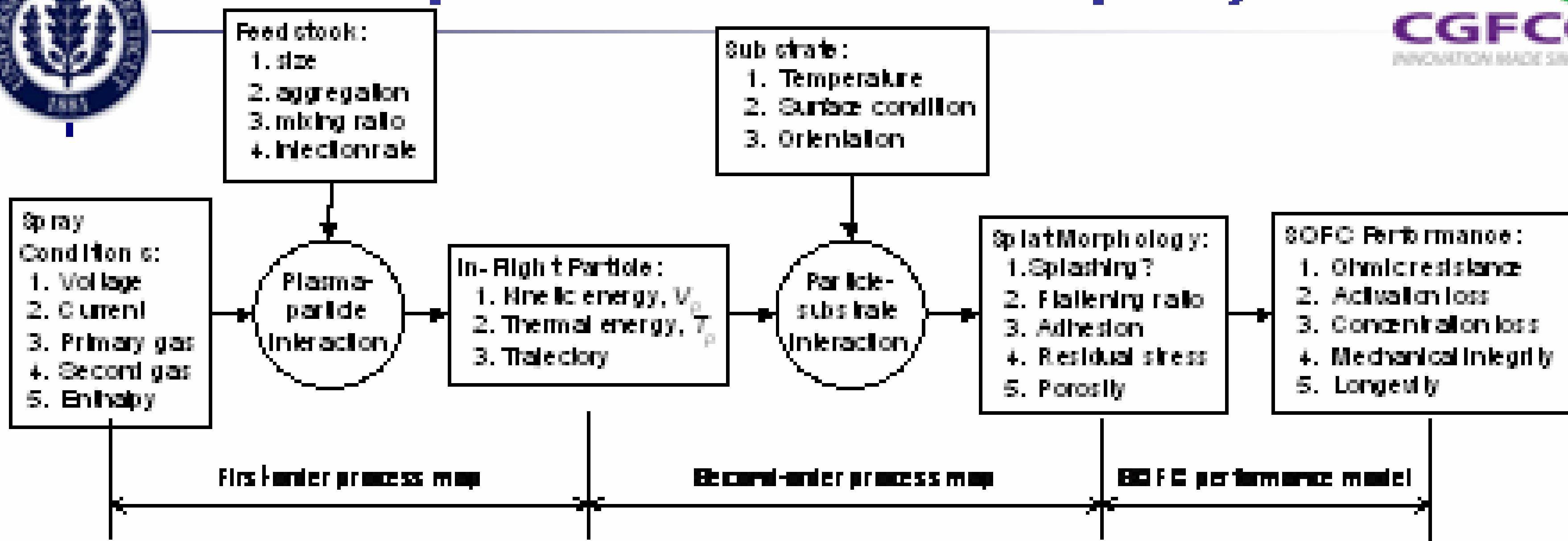
Materials Selection

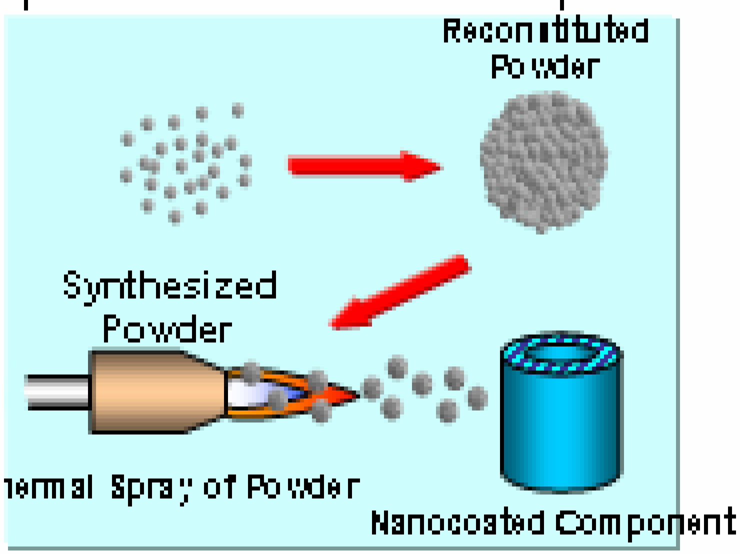
- Interconnect coupon and SOFC single cell from commercial sources
  - Allegheny Ludlum AL453, Crofer 22 APU
  - InDEC/ECN cell
- Ceramic coating material and structures
  - Bond coat (MCrAlY, or lower cost substitutes)
  - Top coat layer 1 (Al_2O_3-75vol% ZrO_2-20vol%) base layer
  - Top coat layer 2 (Al_2O_3-ZrO_2 + 30vol% glass)
- Glass composition and properties
  - Detailing glass property requirements:

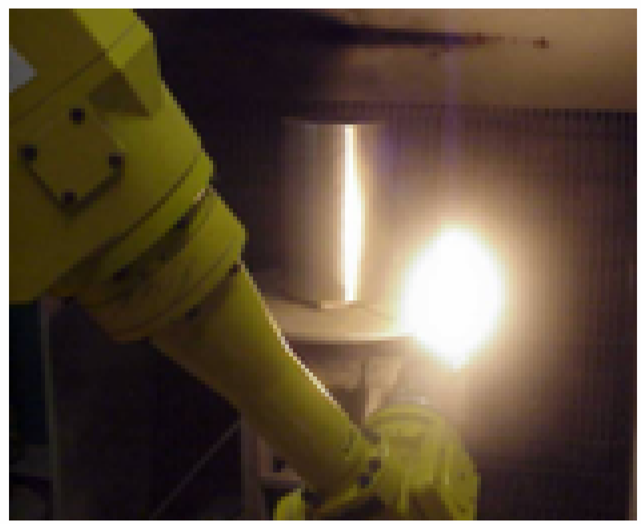
    CTE, softening point, strain point, wetability, chemical stability, etc.
  - Coordinates with other CTP efforts on glass formulation: U. Missouri Rolla, U. Cincinnati, and Sandia National Lab.



Ceramic Coating Produced via Atmospheric Plasma Spray





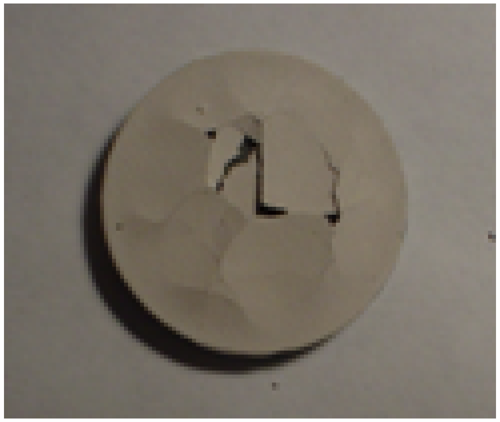


Glass Infiltration and Curing Studies

- Natural wicking or vacuum/pressure assisted infiltration
  - Porosity before and after glass infiltration
  - Interface and remaining pore morphology
- Curing schedule
  - Maximum processing temperature
  - Temperature profile, heating and cooling rate
  - Pressure profile

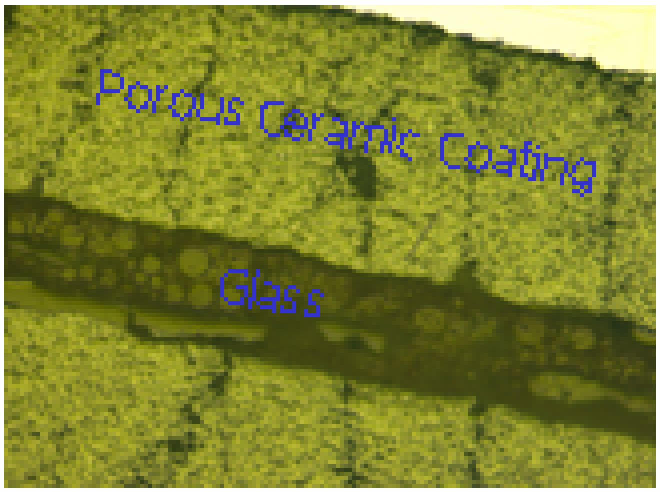
Material Screening Test using Coated Button Samples

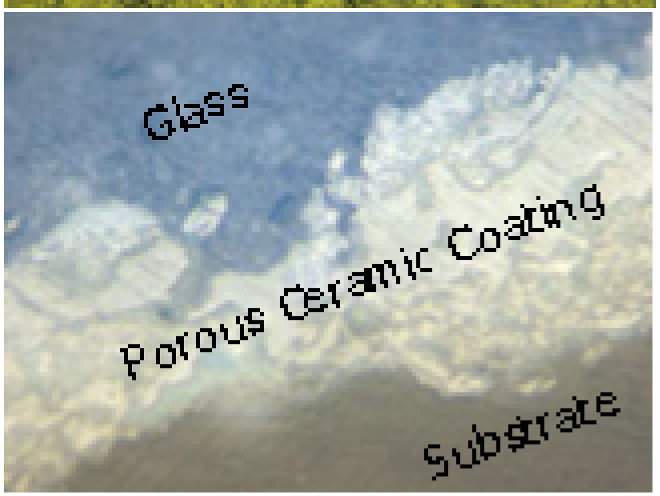
- Evaluate wetting behavior of selected glass and ceramic compositions
- Basic thermal cycle resistance & high temp aging test to evaluate thermo-mechanical robustness of coating
- Electrical resistance test of coating/glass/coating structure using DC or AC method



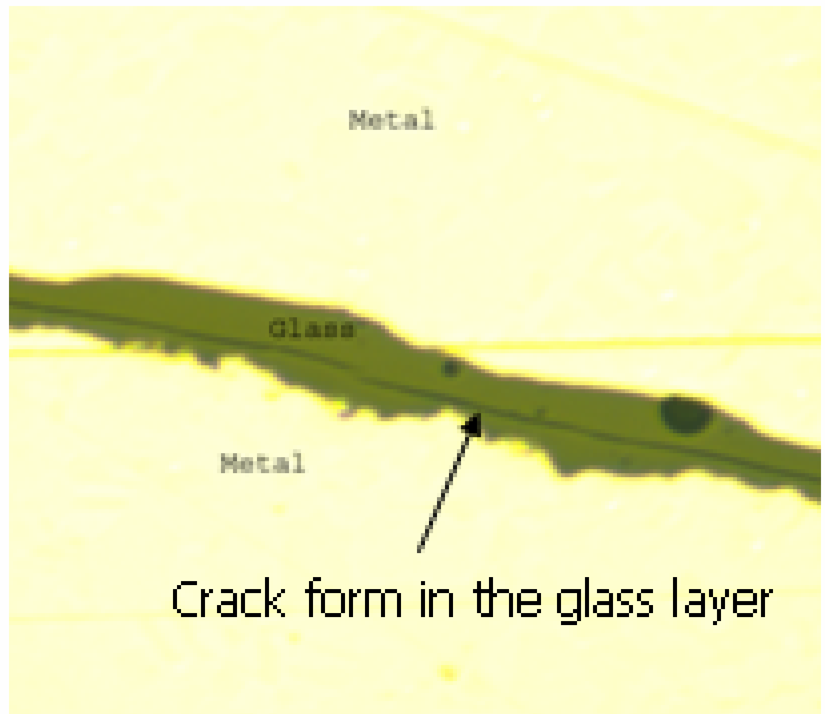
Coated button sample failed as a Result of thermal cycling

Optical Microscopy

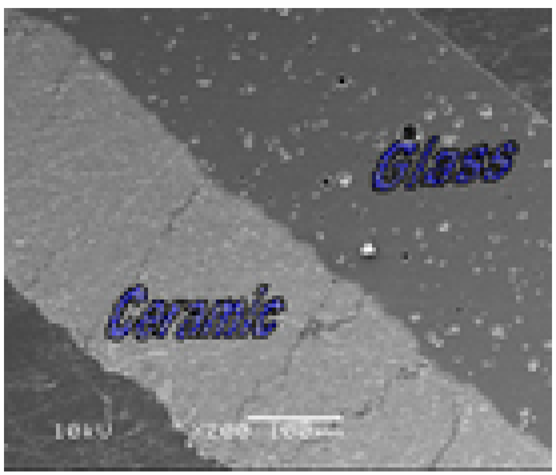




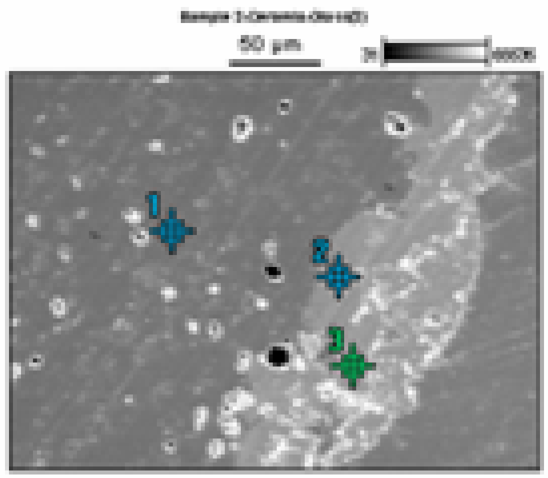
Picture of glass/ceramic interface shows good wetting behavior

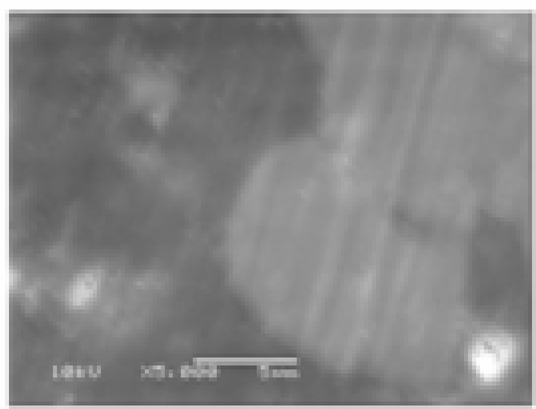


Scanning Electron Microscopy

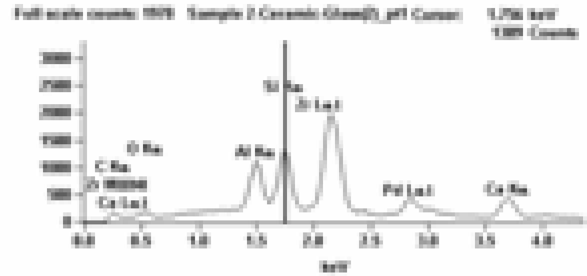


Sample 2

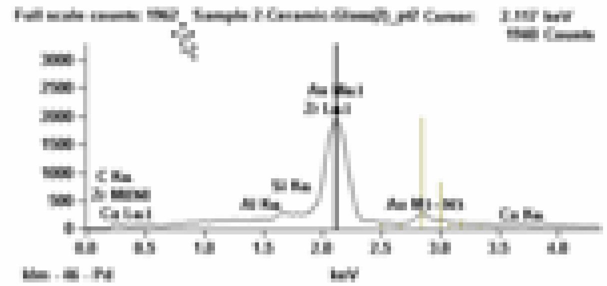




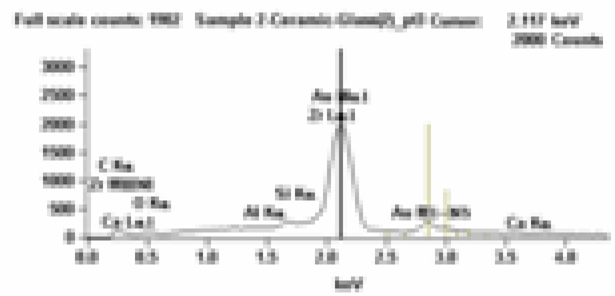
Glass and ceramic interface at high magnification



Point 1, on Glass



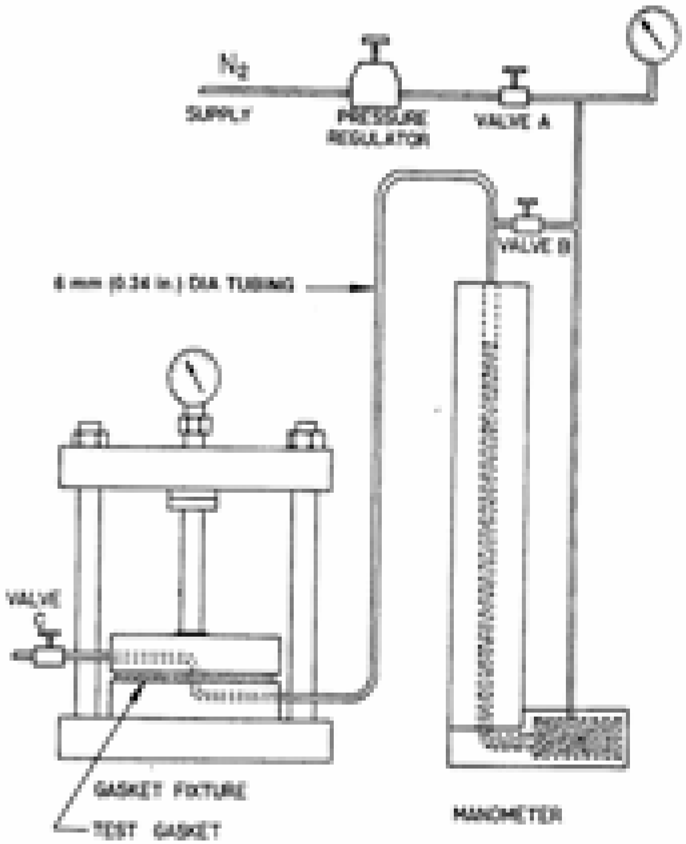
Point 2, on grey area



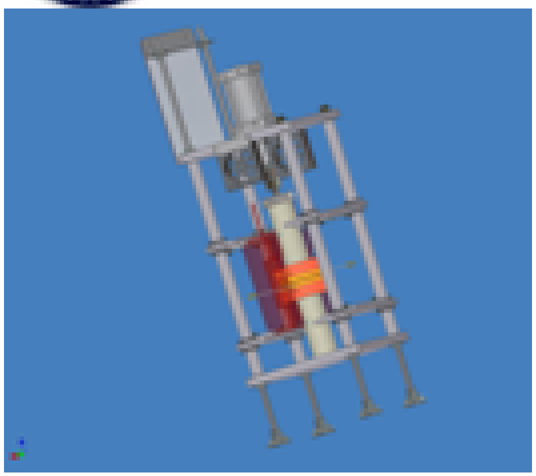
Point 3, on Ceramic

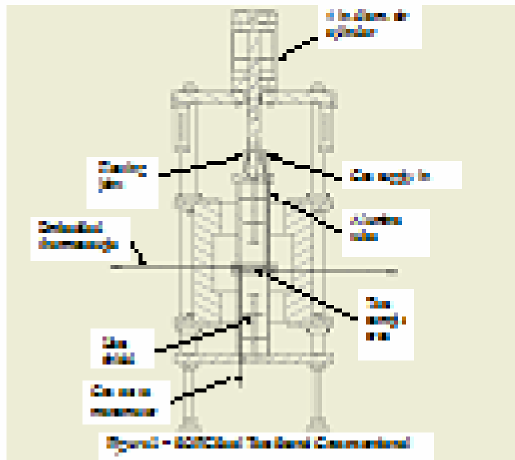
Sealing Performance: Leak Rate Testing

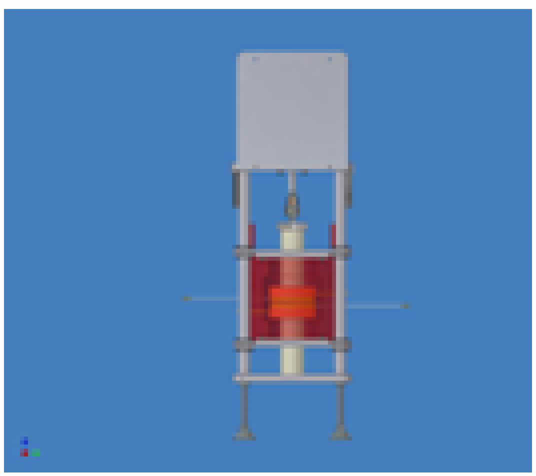
- Objective: measure fuel leak rate (sccm) per bond line length (cm) per pressure difference (psig)
- Reference: ASTM F 37-00 with controlled temperature and gas environment (air + wet fuel)
- Effect of aging and thermal cycle on seal performance:
  - Leak rates v.s. # of thermal cycles
  - Leak rates v.s. hrs of aging time

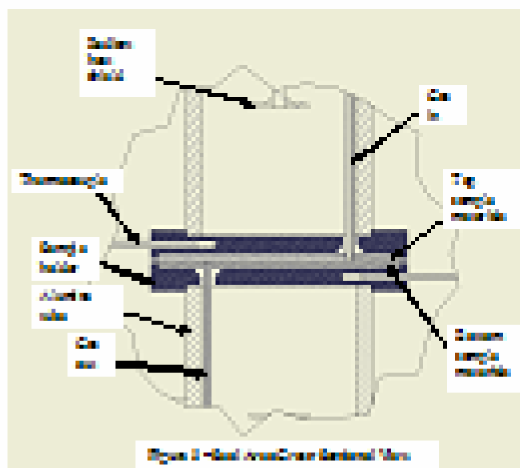


SOFC Seal Test Stand



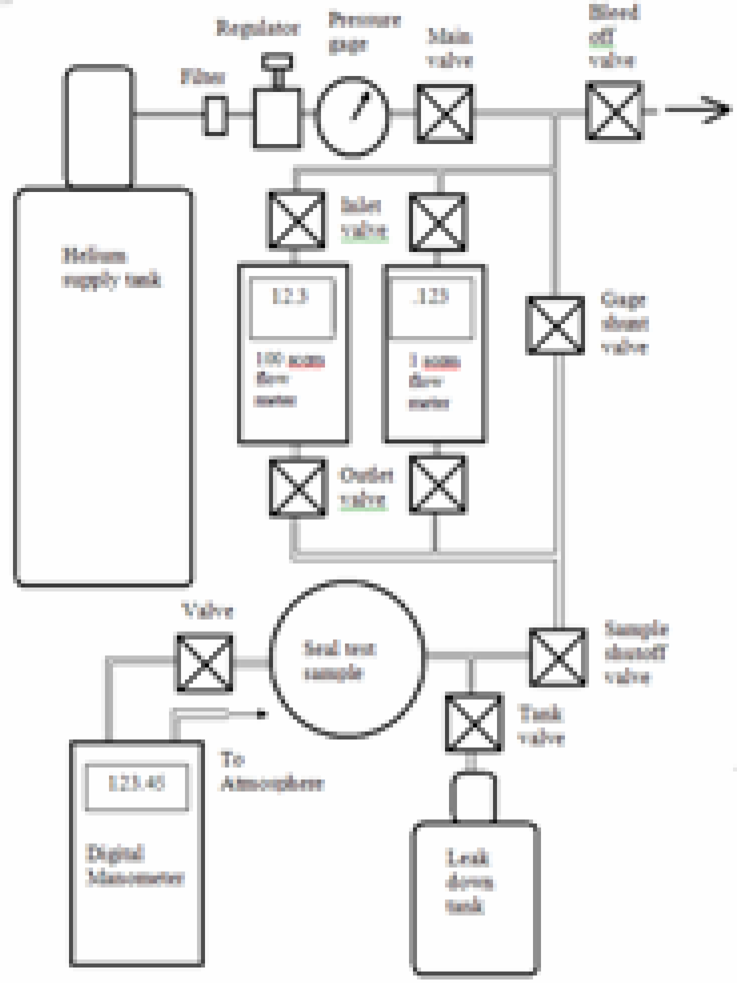






Leak Rate Testing Method

- Direct leak flow rate measurement
  - Measure flow rate of gas supply into sealed chamber
  - Allow continuous monitoring of leak rate
- Pressure leak-down test
  - Sealed chamber initially pressurized, pressure decay recorded
  - Effective in ultra-low leak rate regime

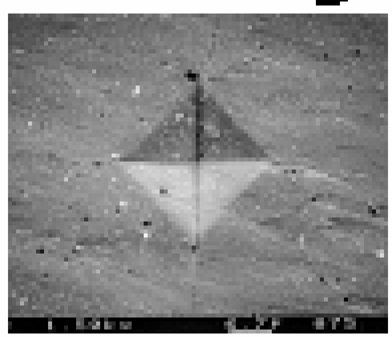


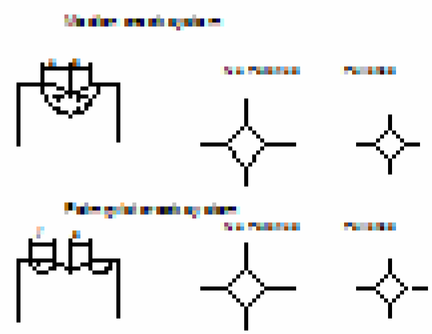
Baseline Mechanical Properties

- Baseline mechanical data for constituent materials in the composite structure
- *Modulus, Poisson's ratio, thermal expansion*, bulk strength, Weibull modulus, yield point, toughness, etc., as functions of temperature
- Source: literature data, vendor data sheet, and limited in-house testing
- Mechanical testing facility in HTML @ ORNL (Edgar Lara Curzio) available

Interfacial Mechanical Strength & Toughness

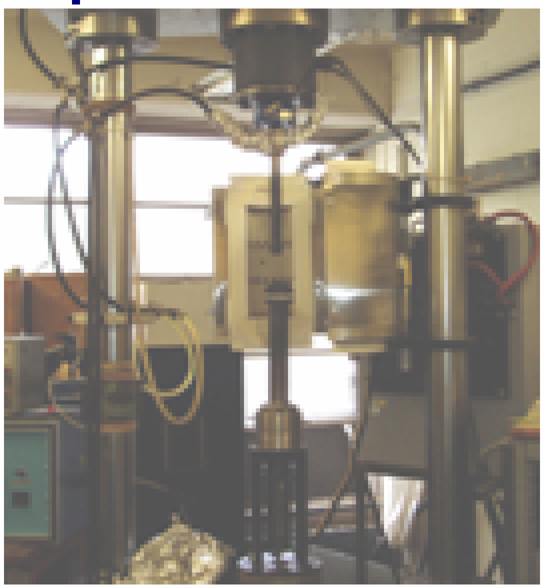
- Crack initiation resistance: strength
  - Pull-out test @ RT and high temperature
- Crack propagation resistance: toughness
  - Three/Four point bend test on a composite beam @ RT and high temperature
- Localized material strength/toughness
  - Vicker's indentation test (RT only)



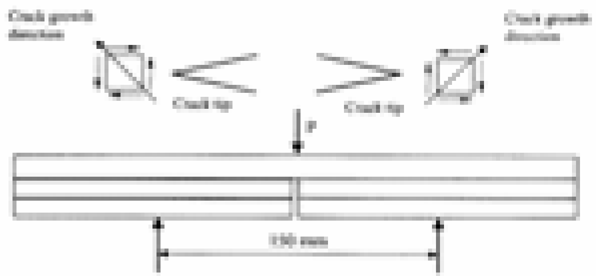


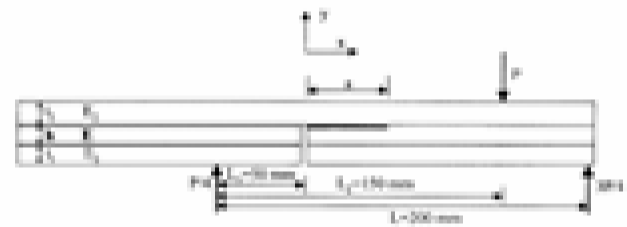
Ke An, PhD Dissertation, ESM Dept, Va Tech 2002

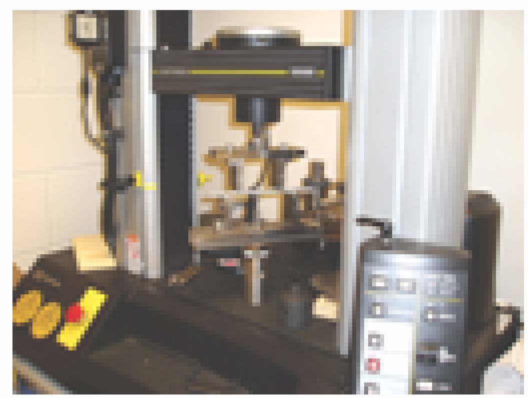
Mixed-mode Interfacial Toughness using Composite Beam Samples



$$G = -\frac{1}{b}\frac{\partial U}{\partial a} = \frac{P^2 (L_1 + a)^2}{32 b E_2}\left(\frac{1}{I_2} - \frac{1}{I}\right)$$







Temperature Programmed Acoustic Emission

- Stimulus, cooling rate and ΔT
- Capturing critical T when crack initiates propagates
- Estimate locations of the strain energy release events
- Help establish a timeline of "failure" events

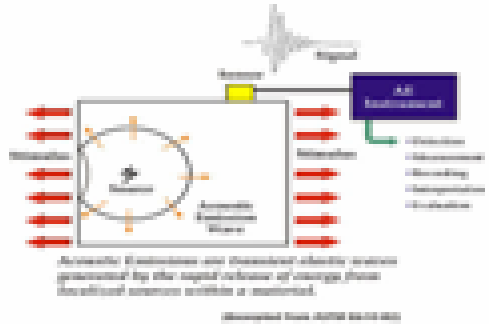


Figure 1. Acoustic emission and its detection.

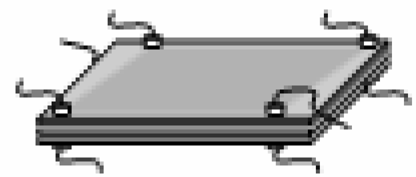
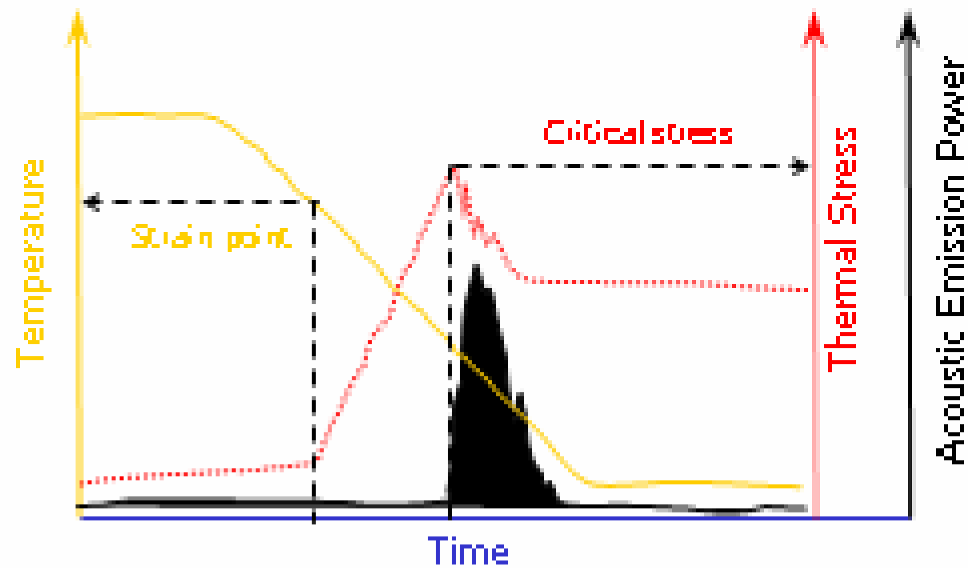
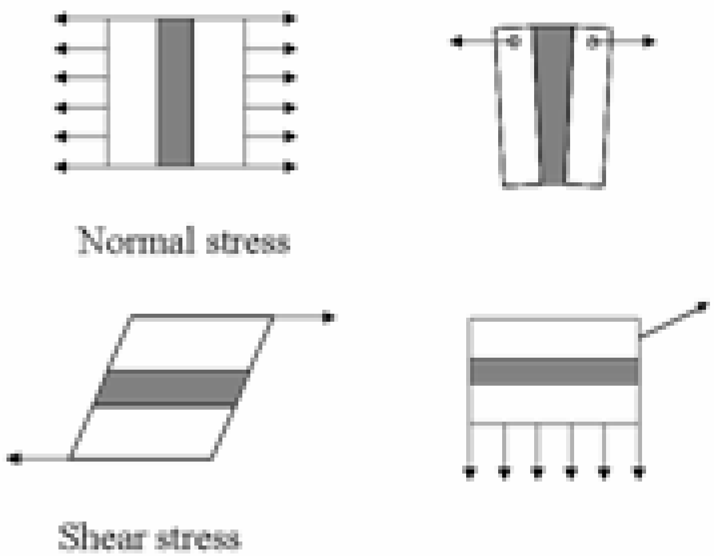


Figure 2. Sensor layout in monitoring and detection of crack initiation and growth in SOFC seals.



Numerical Modeling of Stress/Strain States in the Bonded Seal

- Finite element based stress/strain model
- Mechanical failure criterion (stress controlled or energy release rate controlled processes?)
- Validate the predictive power of models:
  - Can we correlate single cell failure using small coupon test results?
- Parametric analysis



Possible local loading conditions

Towards Engineering of a Robust Composite Seal for SOFC