

# Composite, High Temperature Seals for Gas Separation Membrane Devices

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SBIR Phase I

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# Approach

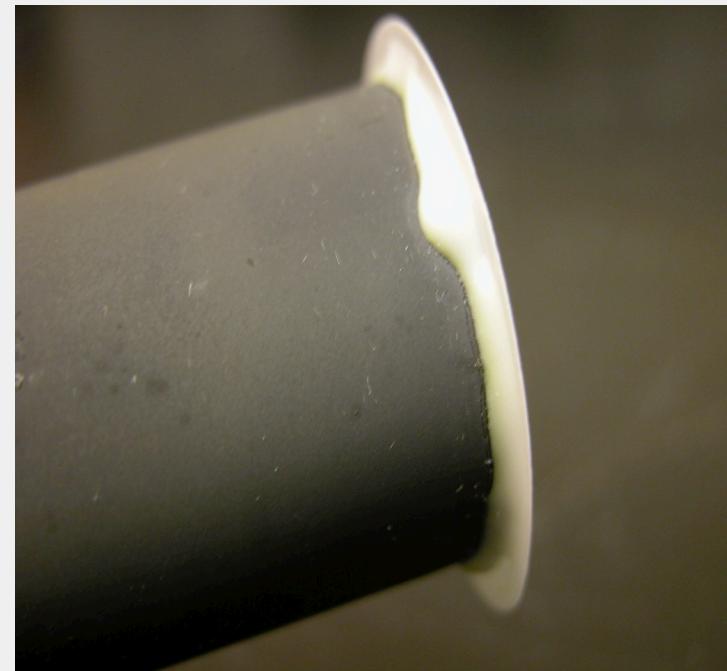
- **Composite Mixture**

- Vitreous Phase -
  - Control softening point,
  - Sealing temperature,
  - Adhesion
  - Compliancy
- Crystalline Phase -
  - CTE control
  - Stability
  - Flatter viscosity (T) curve
- Mixtures of different ratios
  - Overall CTE
  - Flow control
- Potential for graded seal

# Materials Evaluation Planned

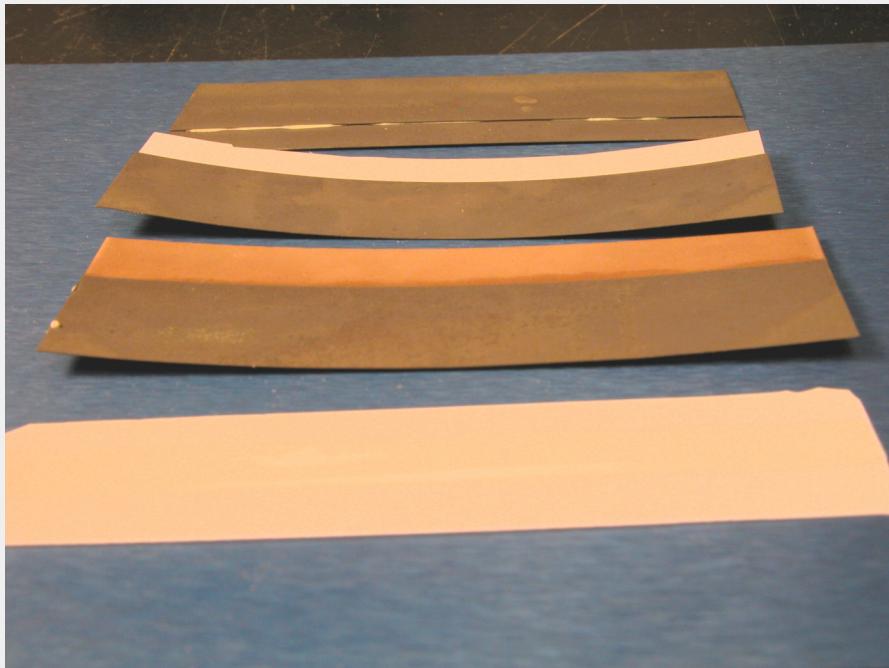
- Ceramics
  - Oxygen ion conductors
    - Zirconia
    - Lanthanum Gallate
  - Mixed conductors
    - Strontium Ferrite
    - Lanthanum Cobaltite
- Metal
  - Stainless steel
  - Super alloys

# Ceramic-metal seal



- **Zirconia electrolyte on Stainless steel tube  
(B20008 seal)**

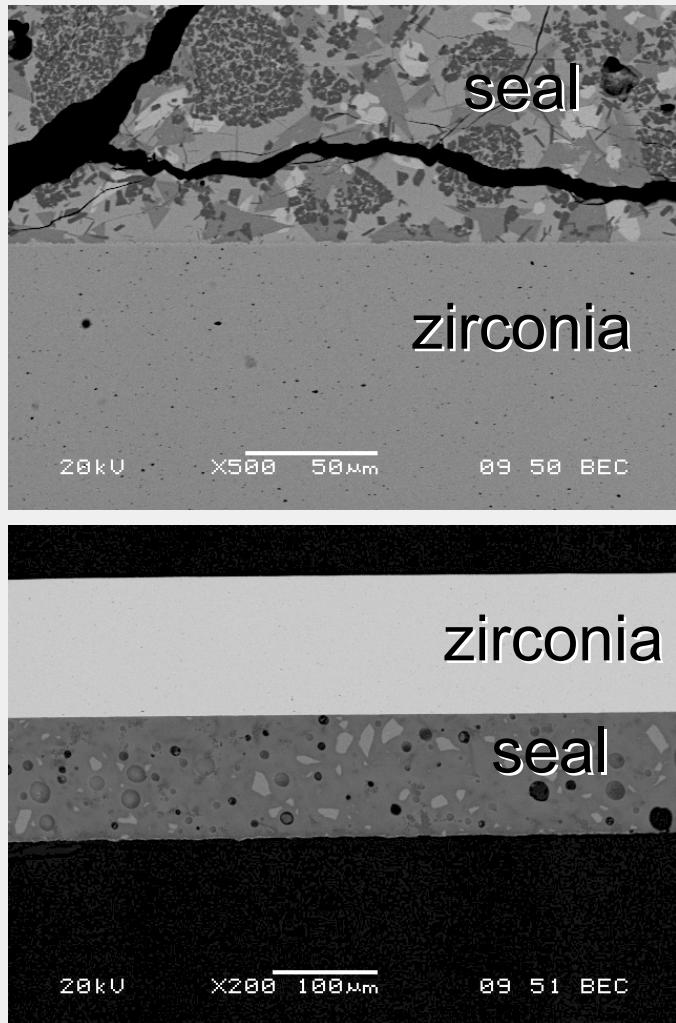
# Sealing of Materials Combinations



- SS - SS
- Zirconia - SS
- Gallate - SS
- Zirconia - Zirconia

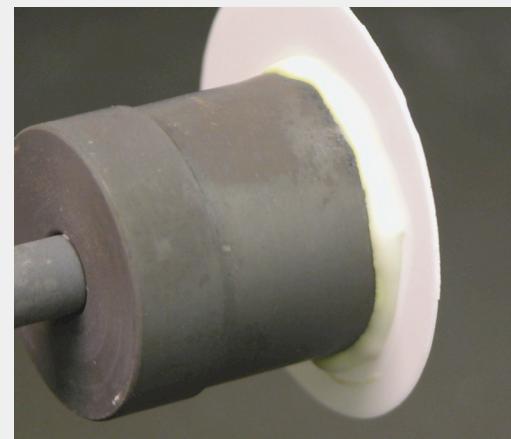
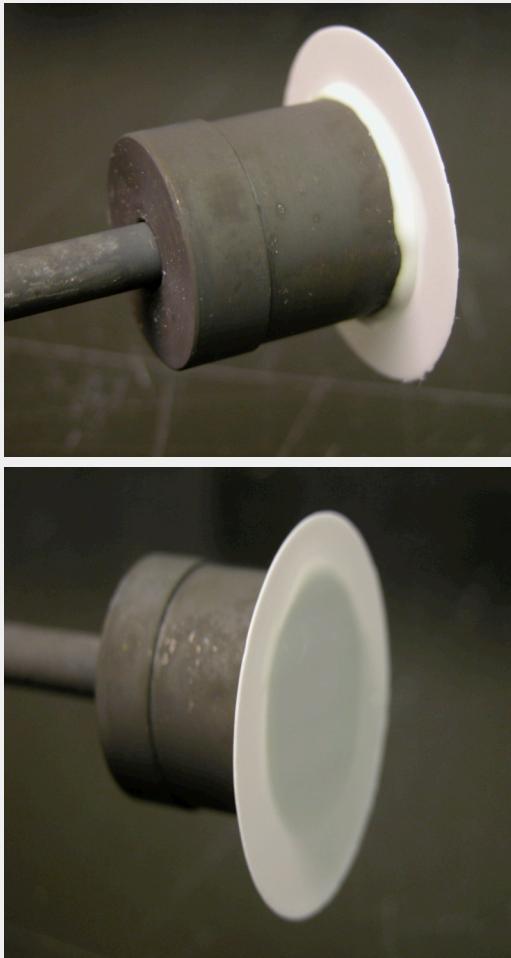
- Flat specimens when components have matching CTE (SS-SS & Zirconia-Zirconia)
- With CTE mismatch graded seal may be suitable

# Zirconia - Seal Interface



- **Initial trial (Batch B20004)**
  - Large CTE mismatch
  - Poor distribution of phases
  - Interfacial cracks
- **Batch 20008**
  - Closer CTE
  - Better distribution of phases
  - Well-bonded interface

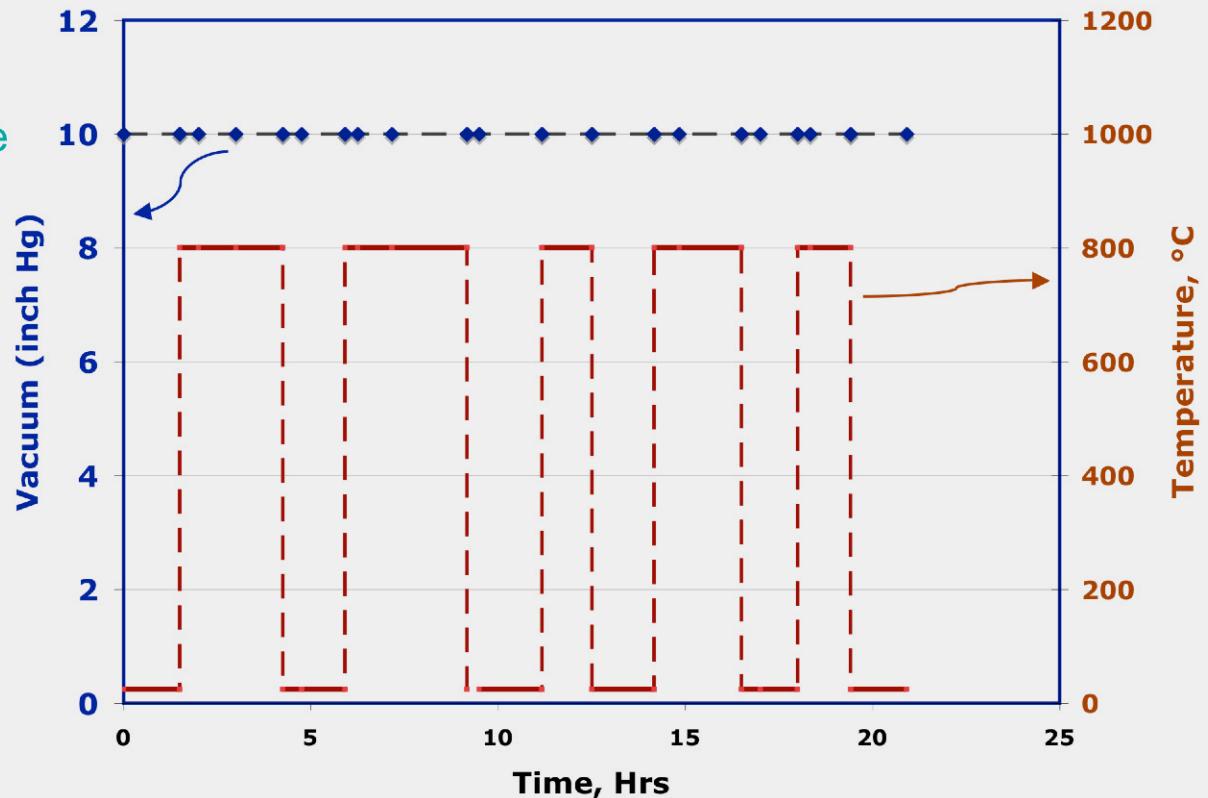
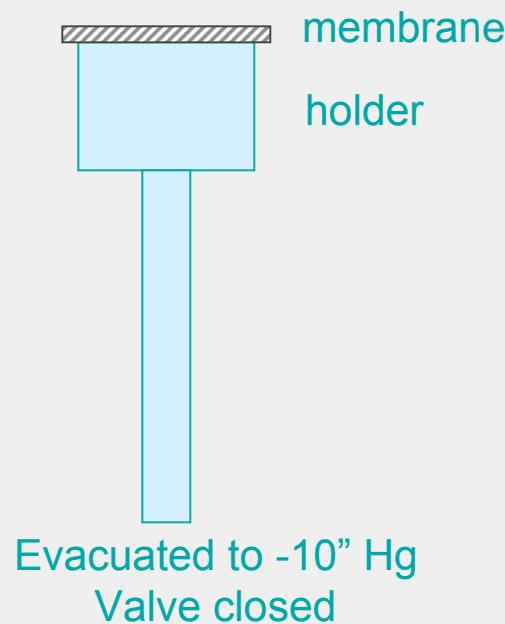
# Hot Test Rig (Zirconia on SS Holder)



- As Fabricated

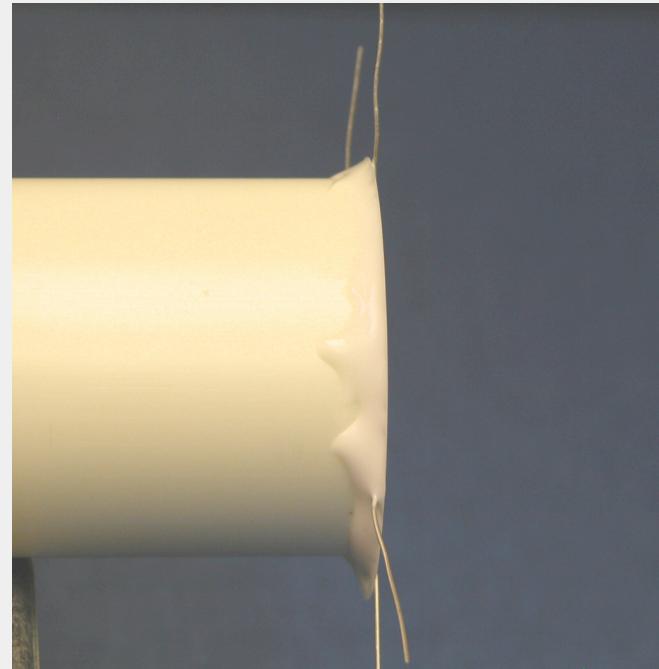
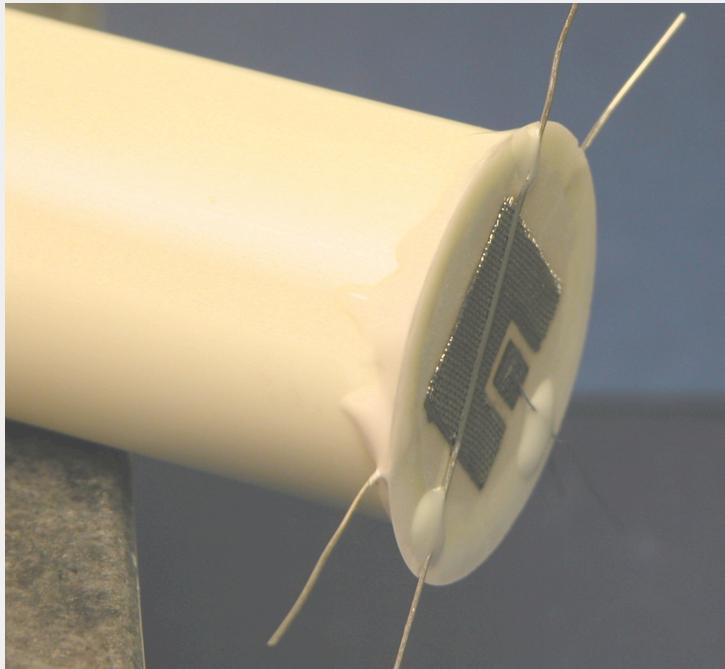
- After Five Thermal cycles to 800°C  
(Pressure test at RT and 800°C)

# Seal Test Results



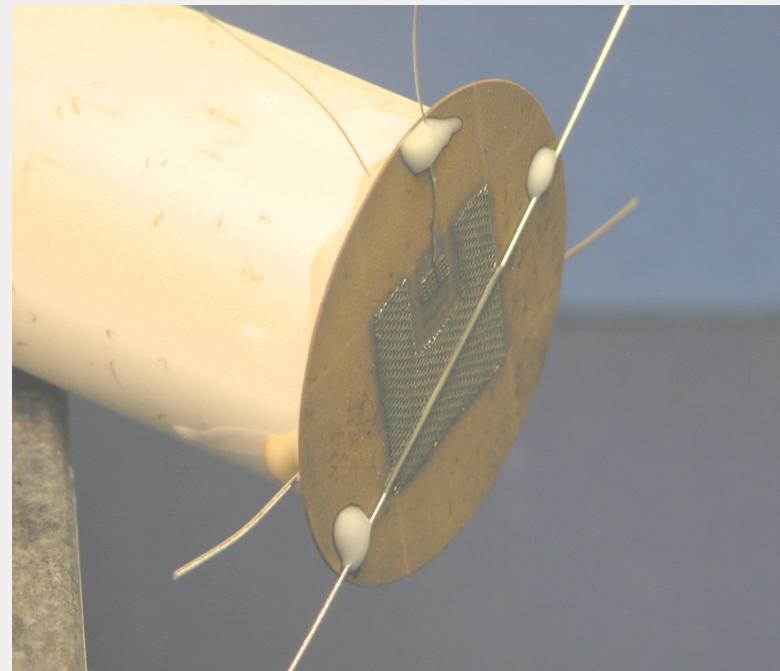
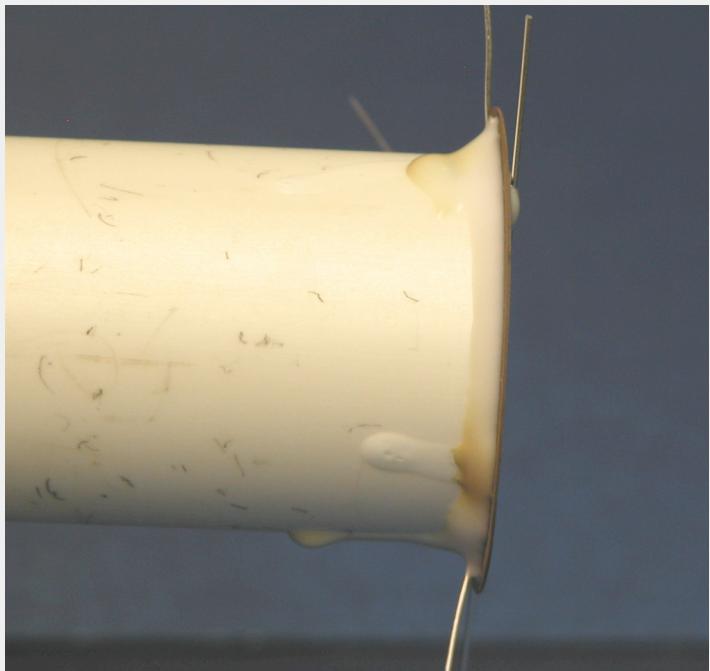
- Pressure decay test at pressure differential (~ 5 psi)
  - Monitored for 1 to 2 hours at RT and 800°C; five thermal cycles
  - Stable both at room temperature and 800°C

# Button Cell Test



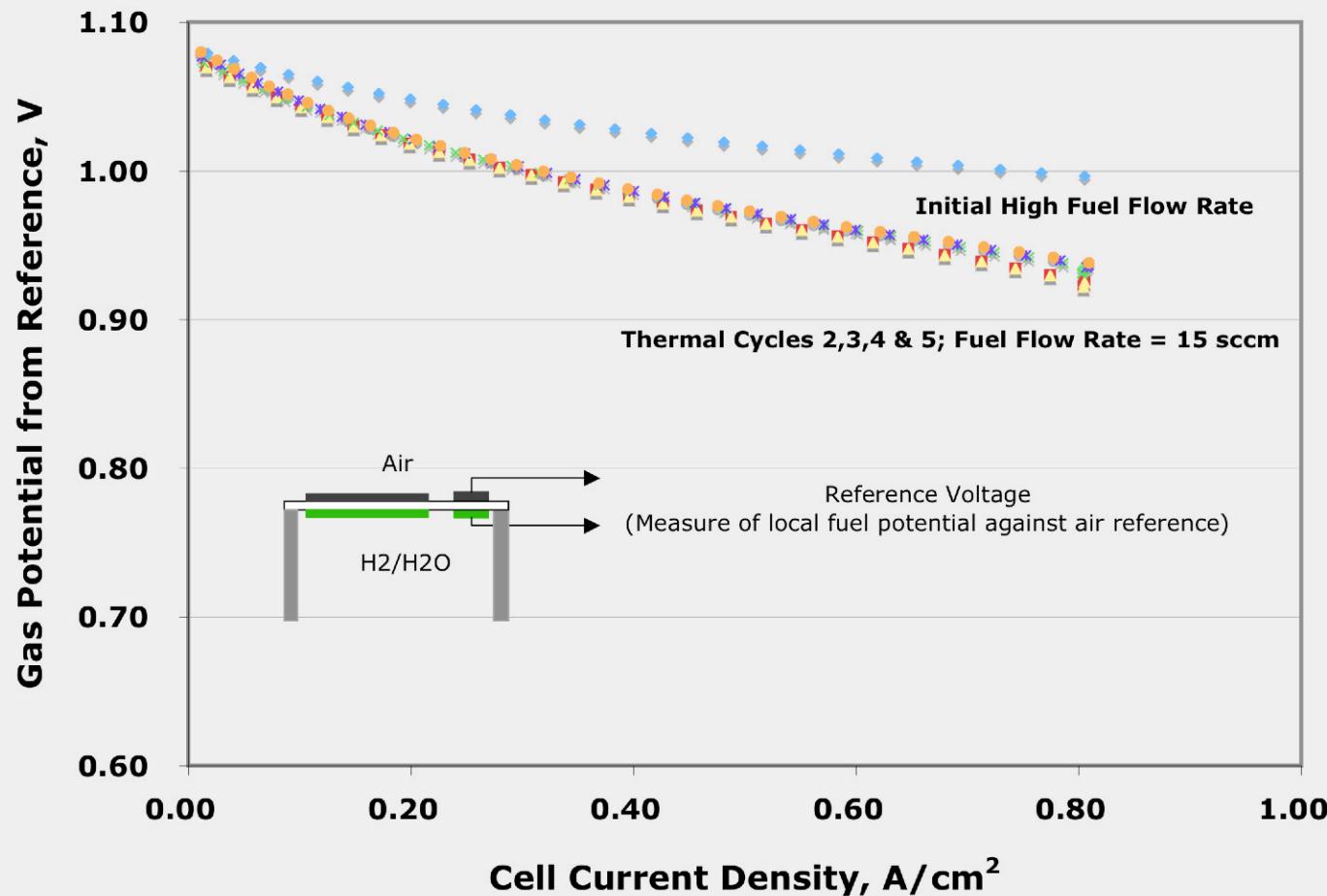
- Zirconia electrolyte cell on Zirconia tube  
(B20008 seal)

# Button Cell Test



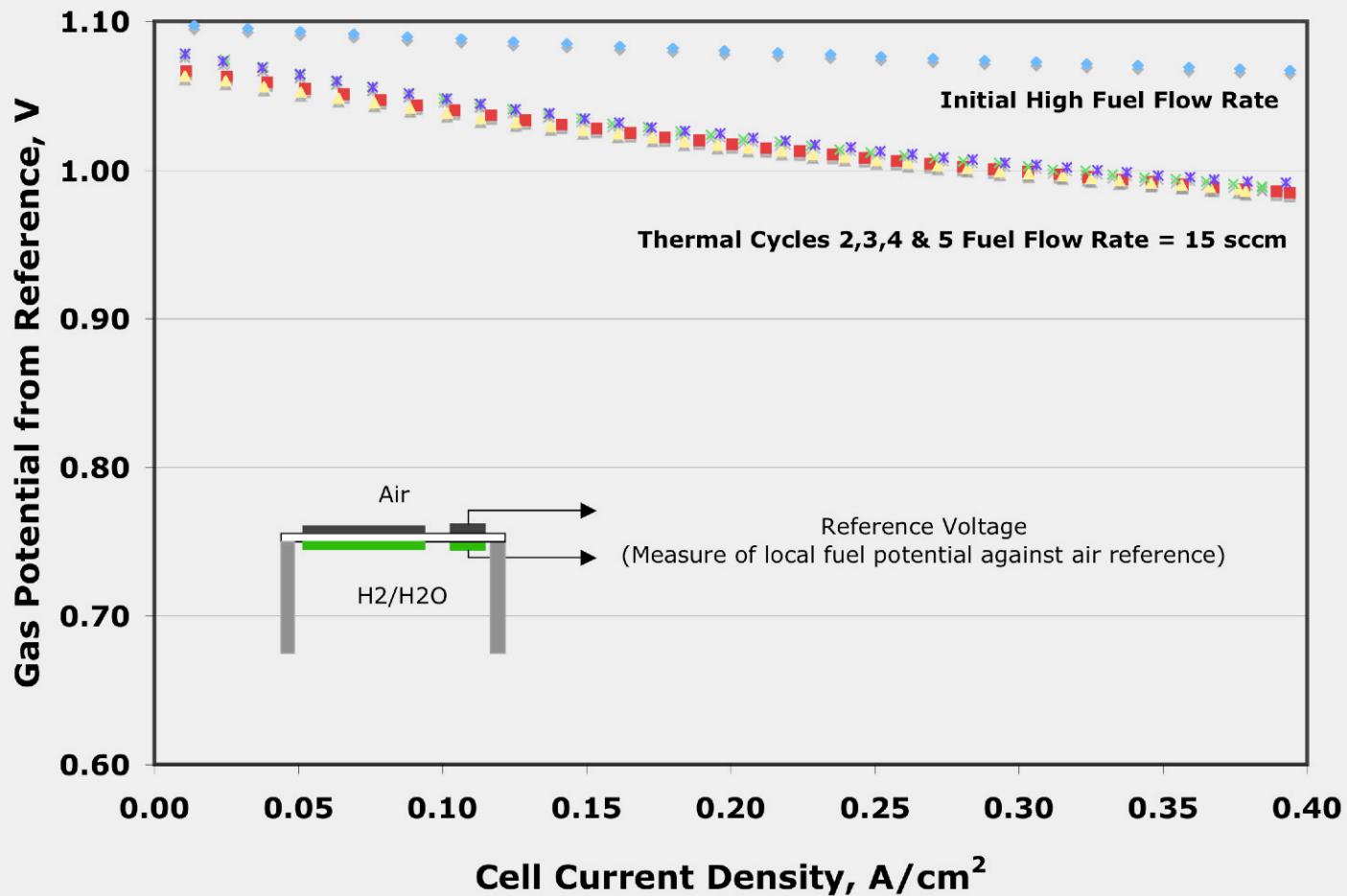
- **Gallate electrolyte cell on Zirconia tube**  
(B20008 seal)

# Zirconia Button Cell: Reference Voltage with Thermal Cycles



- **Zirconia:** Low fuel flow rate, unchanged gas potential with thermal cycles

# Gallate Button Cell: Reference Voltage with thermal cycles



- **Gallate:** Low fuel flow rate, unchanged gas potential with thermal cycles

# Summary

- New composite glass seal material developed
  - Flexibility in CTE, flow
    - Through modification to crystalline phase
    - Ratio of crystalline to vitreous phase
  - Promising results from seal tests
    - Pressure test (RT and 800°C) of zirconia to stainless
    - Button cell tests of zirconia - zirconia and gallate - zirconia
    - Five thermal cycles
  - Reactivity & Seal tests with perovskite materials (hydrogen and mixed oxygen-electron conductors) planned