

Textured Composite Seals for Low Temperature SOFCs

**SECA Core Technology Program
SOFC Seal Meeting**

July 7, 2003

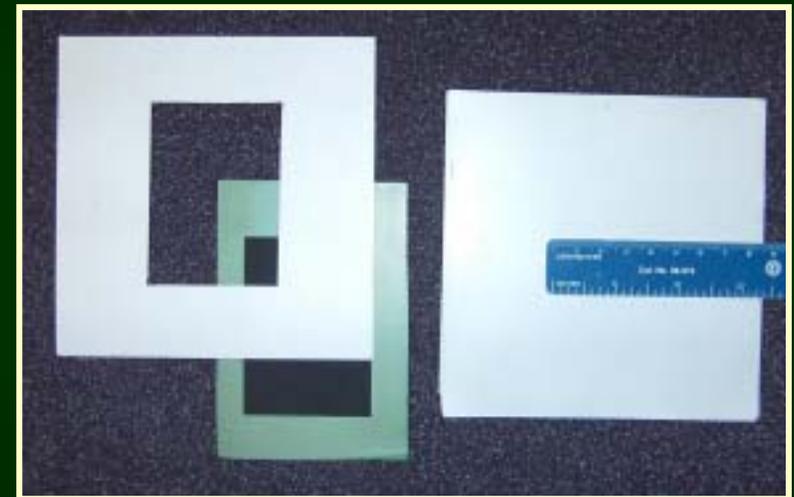


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NexTech Materials, Ltd.**

Seal Materials for Intermediate Temperature SOFCs

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- DOE Contract Number: DE-FG02-02ER83528
- Project Monitor: Lane C. Wilson
- Objectives:
 - Low Operating Temperature
 - $600^{\circ}\text{C} < T < 800^{\circ}\text{C}$
 - Flexible Configuration
 - Compatible with SECA Stack Designs and Materials
 - Thermally and Mechanically Robust
 - Able to Withstand Thermal Cycling
 - Composite Approach
 - Tailoring Thermo-mechanical Properties Chemical Compatibility



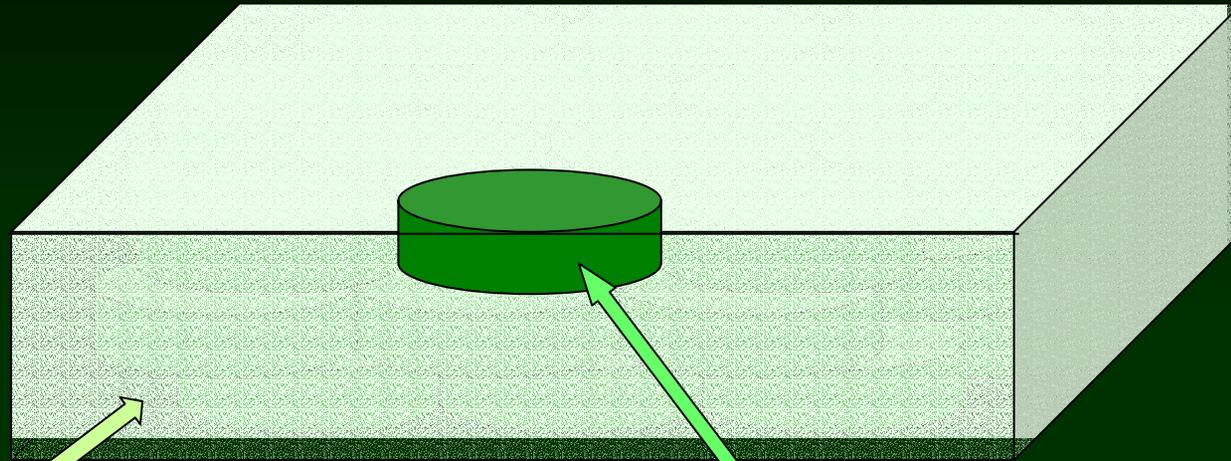
As-Produced Seal Materials
Partially Assembled
7 X 10 cm Cell Package

SOFC Seal Design Characteristics and Approaches

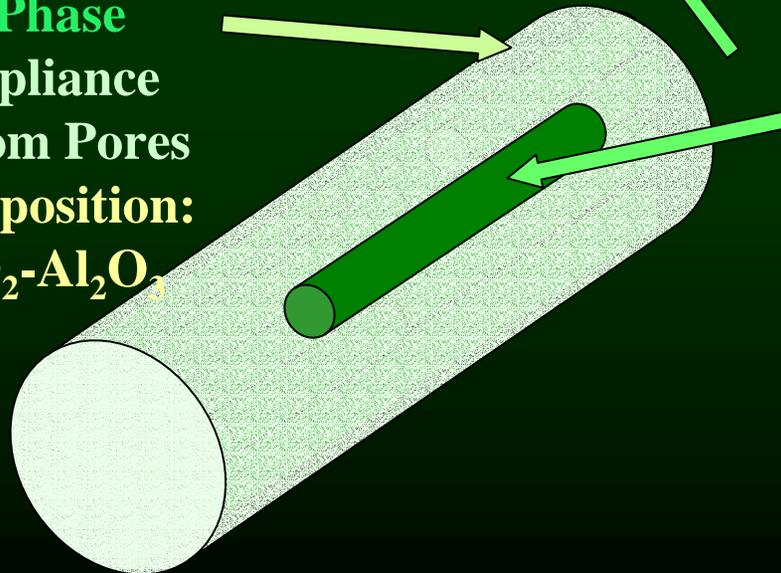
- **Gas Tight—Prevent Fuel/Air Mixing**
 - **Insulating—Prevent Current Flow**
 - **Compliant—Mechanical Stress Relief**
 - **Chemically Inert—Long Stack Life**
-
- **Glass Seals—Long Term Stability**
 - **Glass-Ceramic Seals—Thermal Cycling**
 - **Compressive Crystalline Seals—Leaky**

Textured Composite Approach

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Amorphous Phase
Provides Compliance
Seals Leaks From Pores
Candidate Composition:
 $\text{CaO-BaO-SiO}_2\text{-Al}_2\text{O}_3$



Crystalline Phase
(75-90% by volume)
Provides Rigidity,
Mechanical Strength
Retains Glass in Pores
Candidate Materials:
Mica, Talc, Alumina,
Zirconia Fibers

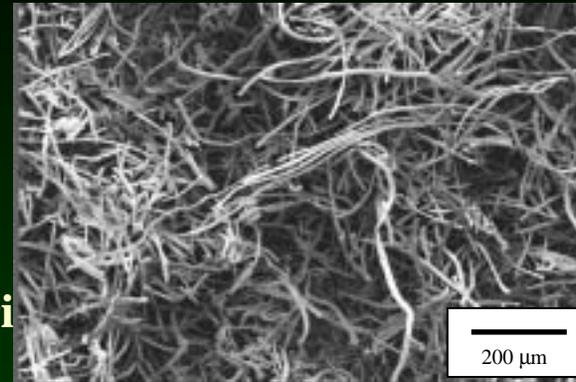
Forming Process

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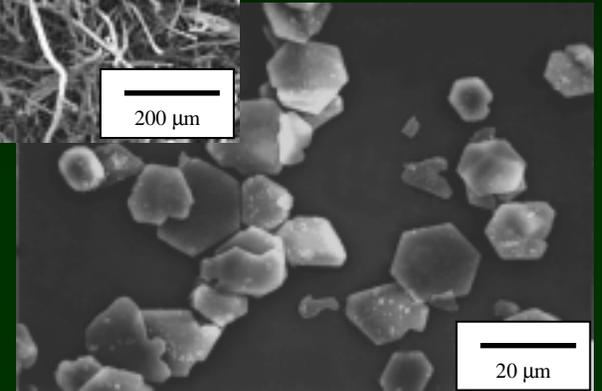
Approach

Tape Cast Seal Manufacturing

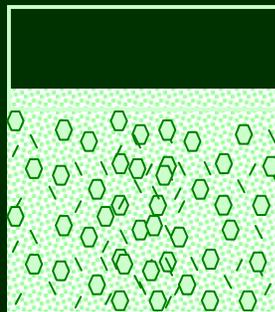
- Tailored Thickness, Size and Shape
- Easily Shaped
- Conformable Precursor Seal Material



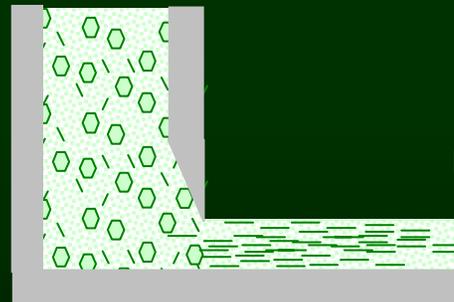
ZrO₂ Fibers



Al₂O₃ Platelets



Anisotropic Particles
Glass Formers and Binders
are Mixed to Form
Tape Casting Slurry



Tape Casting
Orients Particles
to Form
“Brick-Wall”
Microstructure



Dried Tape
Punched to Form
Gaskets

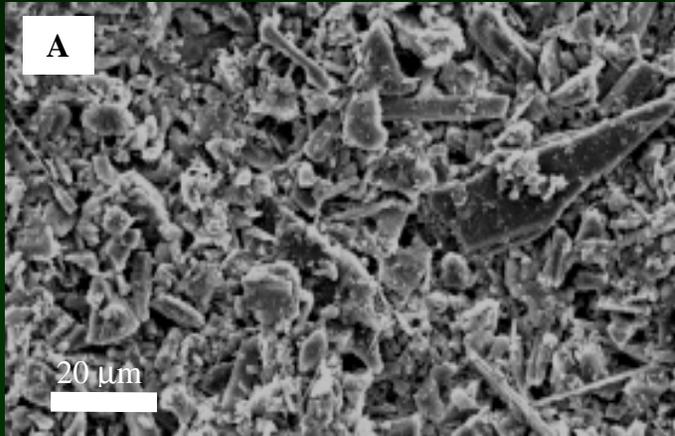
Phase I Goals

- **Identify Suitable Glass and Crystalline Phases for Preliminary Evaluation**
- **Demonstration of Forming Process**
- **Demonstration of *in-situ* Burnout**
- **Demonstration of Sealing Properties**

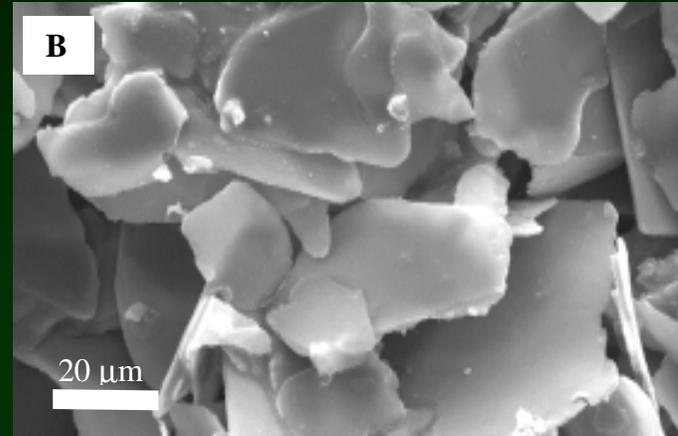
Template Particles

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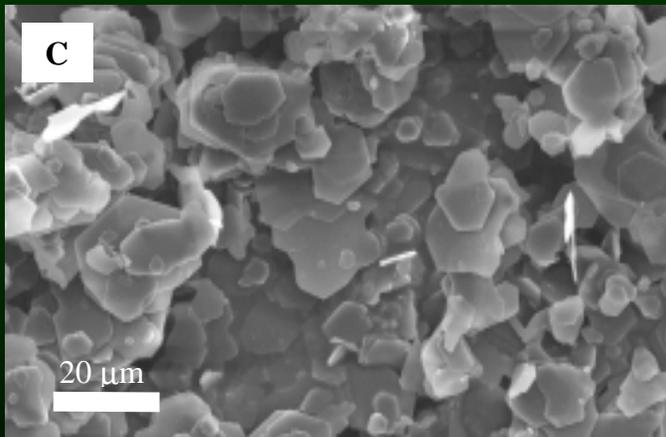
Talc



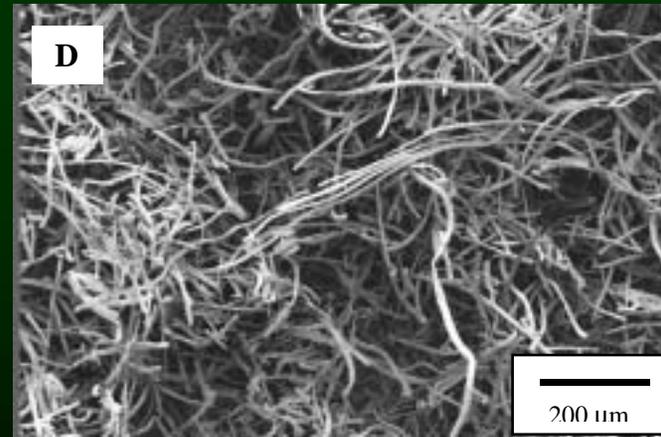
Mica



Alumina



Zirconia



Glass Phase Selection

Glass Selected for Phase I Evaluations:

15 mol% BaO

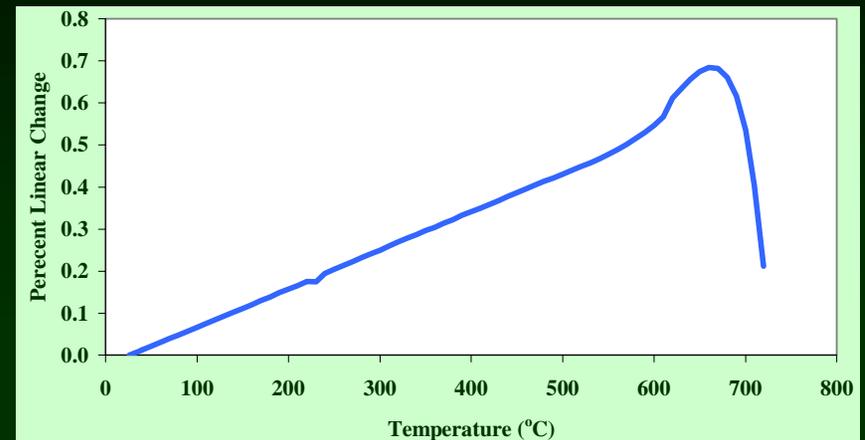
25 mol% CaO

7.5 mol% Al₂O₃

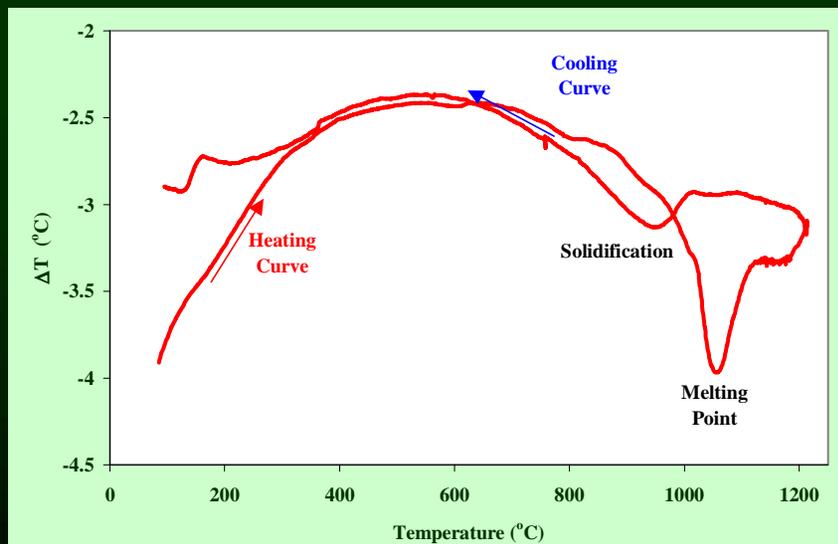
45 mol% SiO₂

CTE: 9.1 ppm (0-600°C)

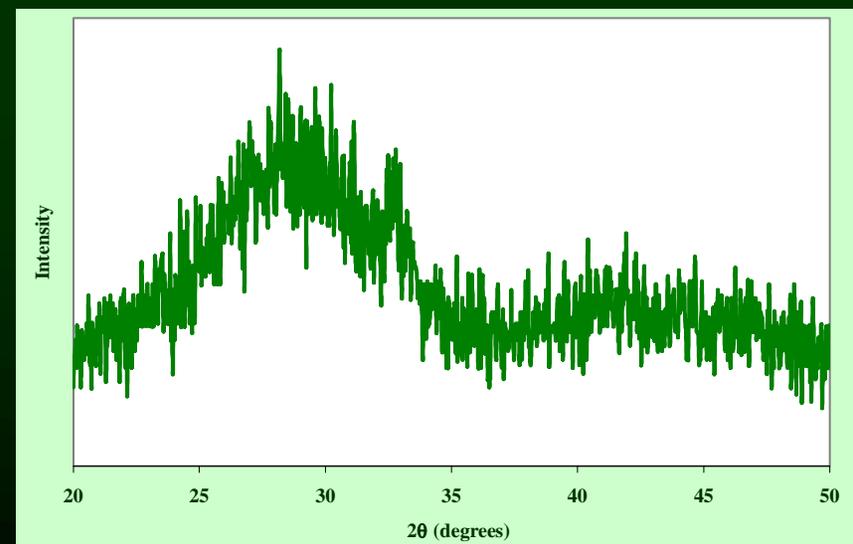
Softening Point ~650°C



Dilatometry for Sintered Glass Bar 5°C/minute

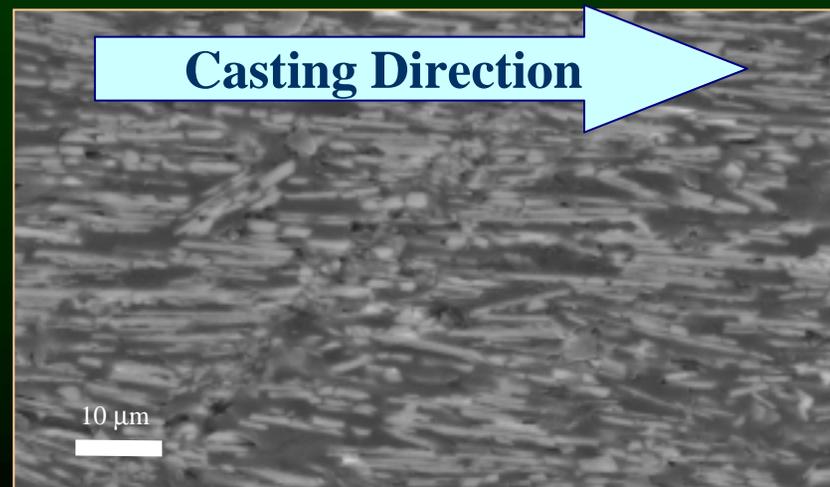


DTA of Sol-Gel Derived Glass Precursor



XRD Pattern of Glass, after 10X Cycles to 750°C

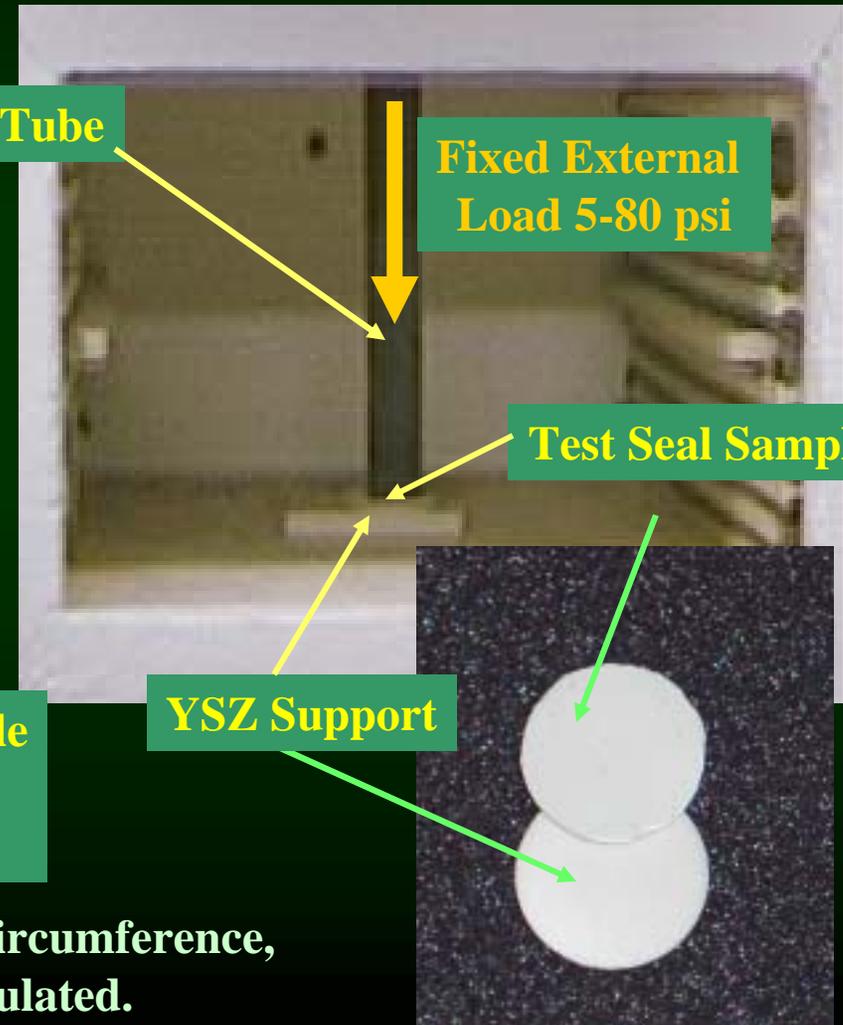
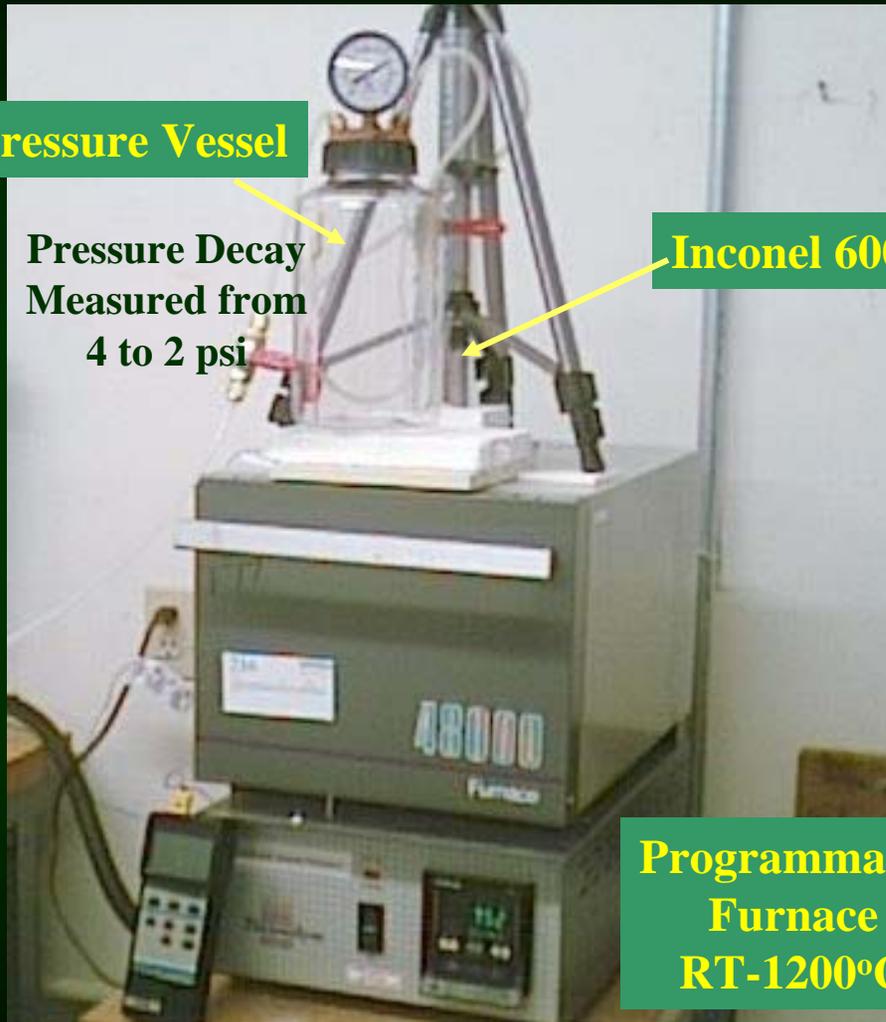
Seal Development—Orientation



Alumina-15% Glass
Composite Material

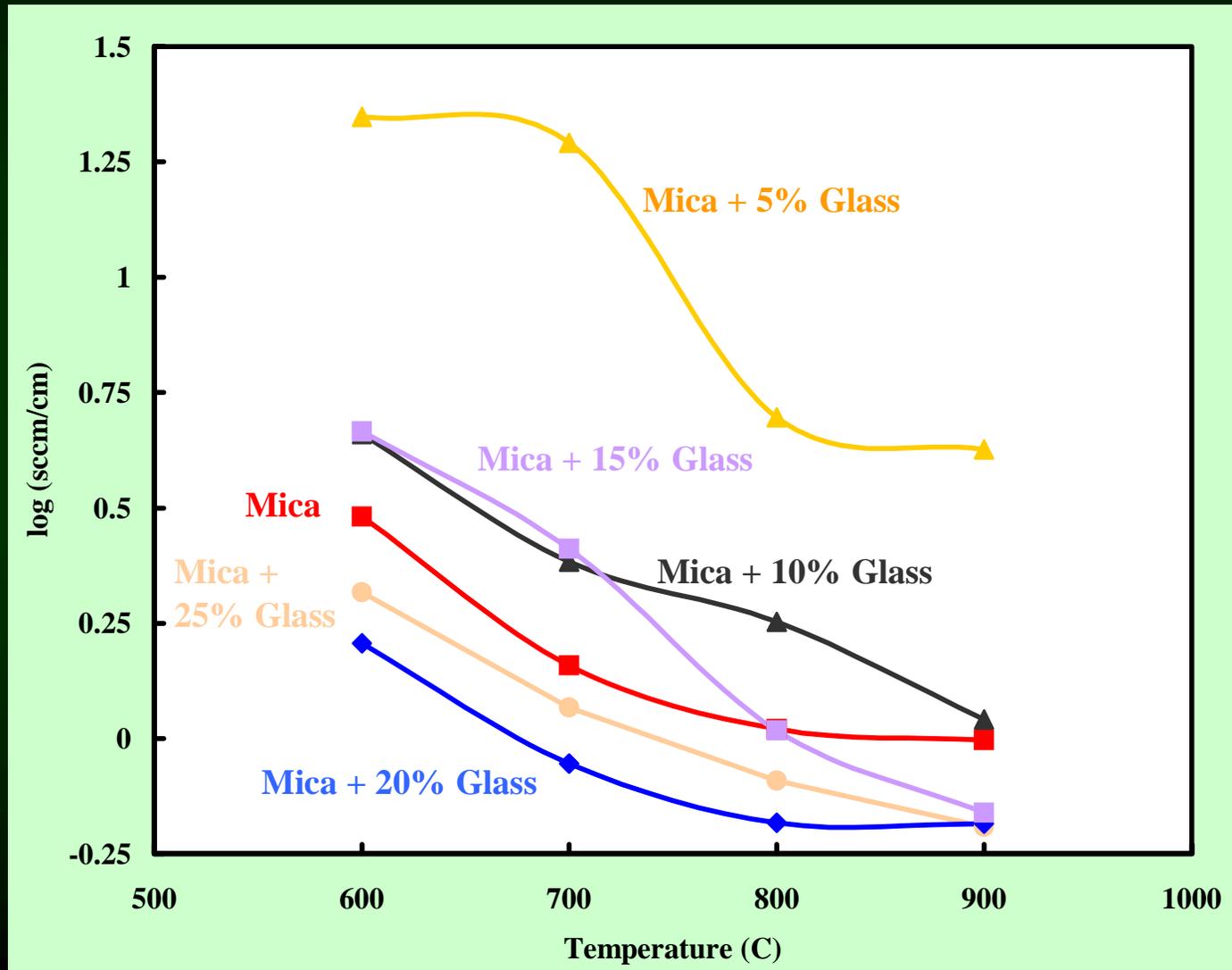
Seal Tester Apparatus

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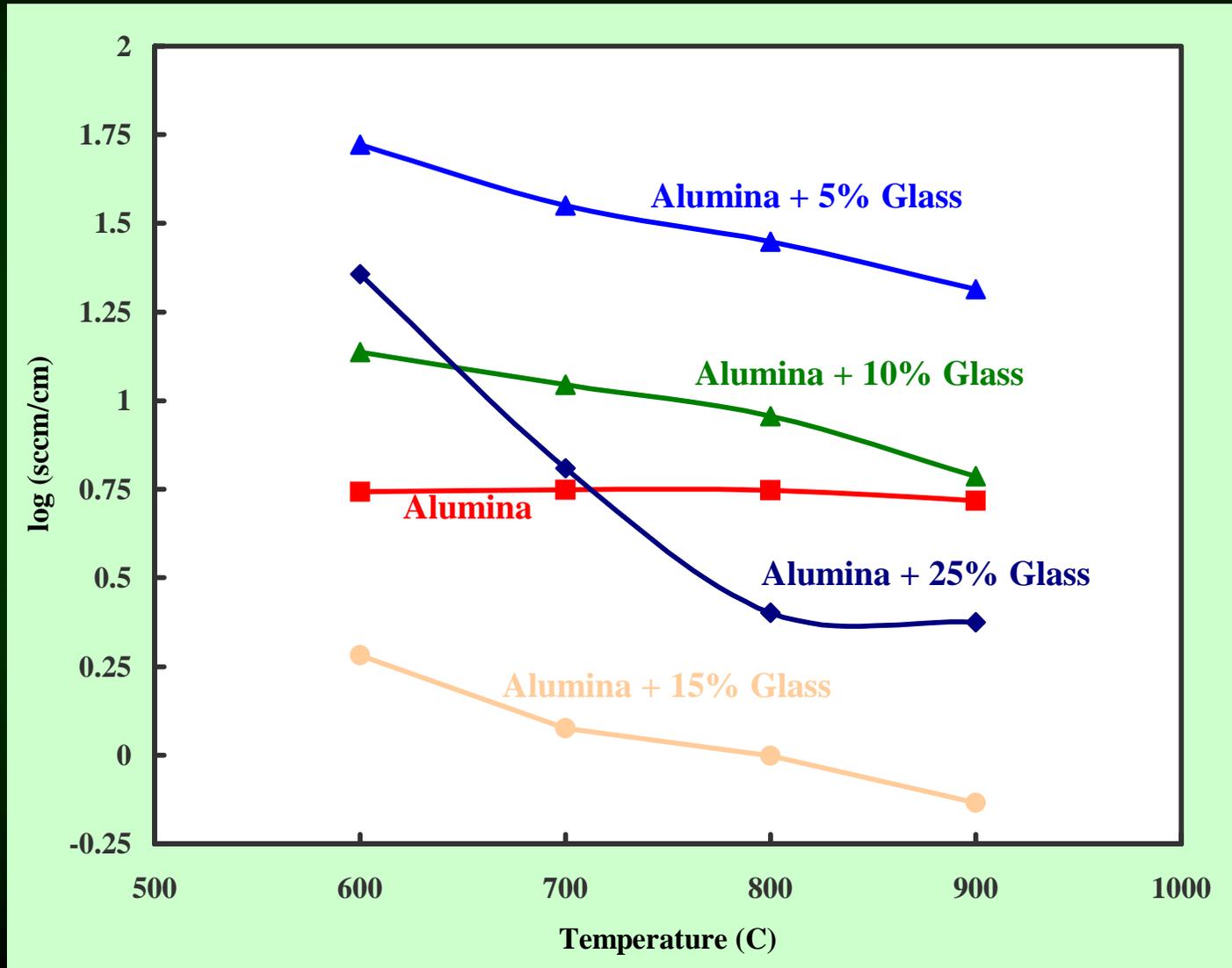
Based on Decay Time and Tube Circumference,
the Leak Rate/cm was calculated.

Seal Development—Mica Based

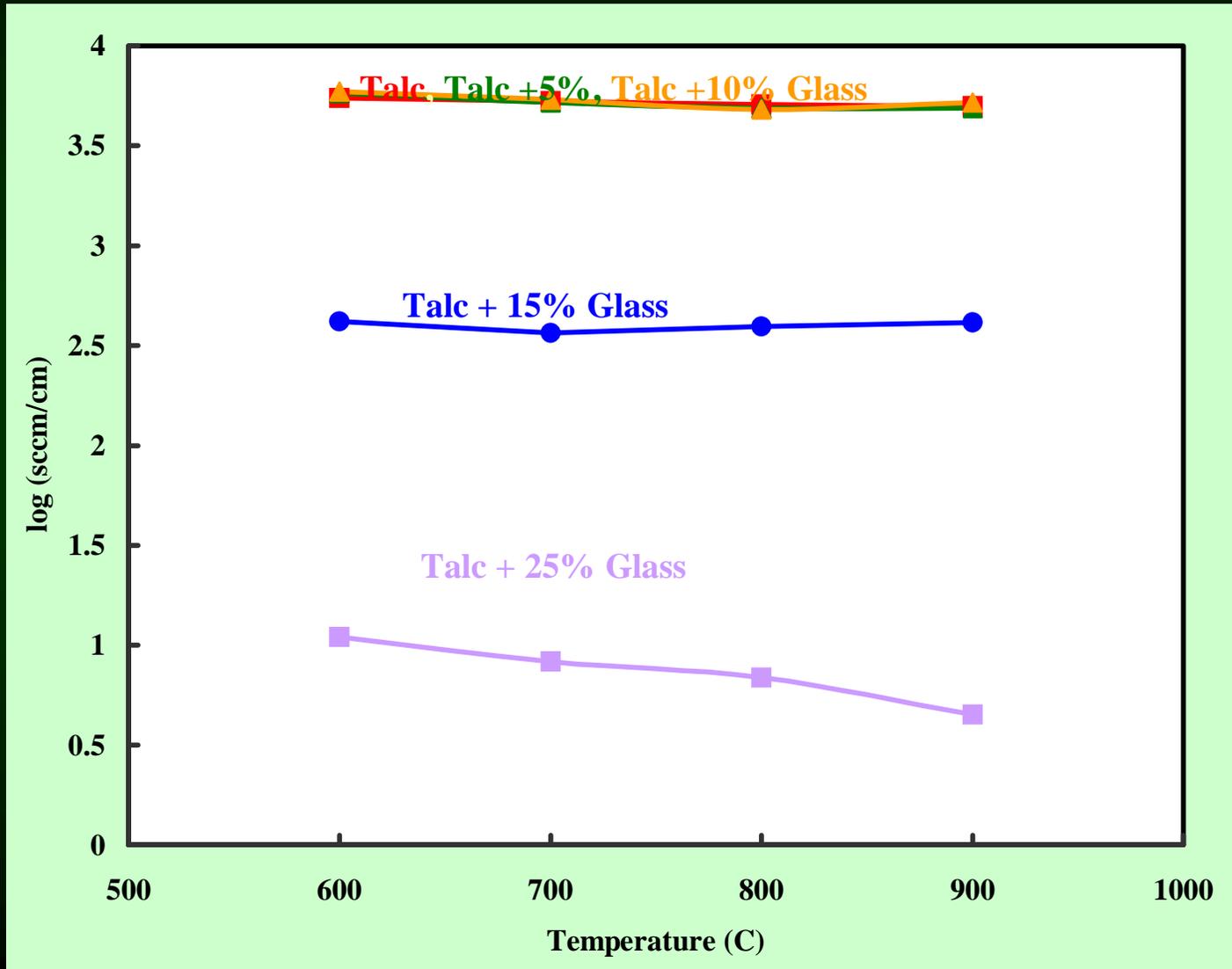


Seal Development—Al₂O₃ Based

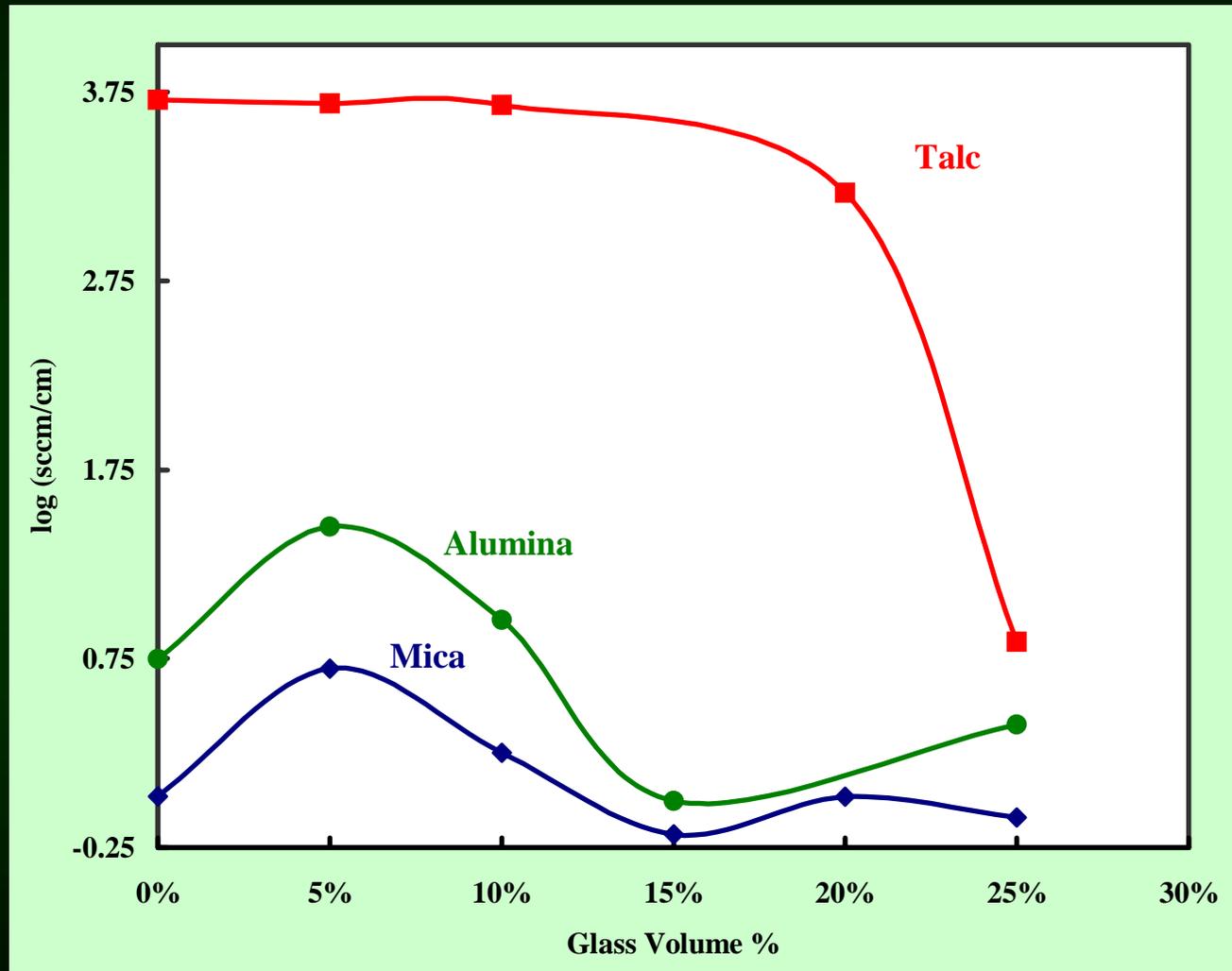
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Seal Development—Talc Based

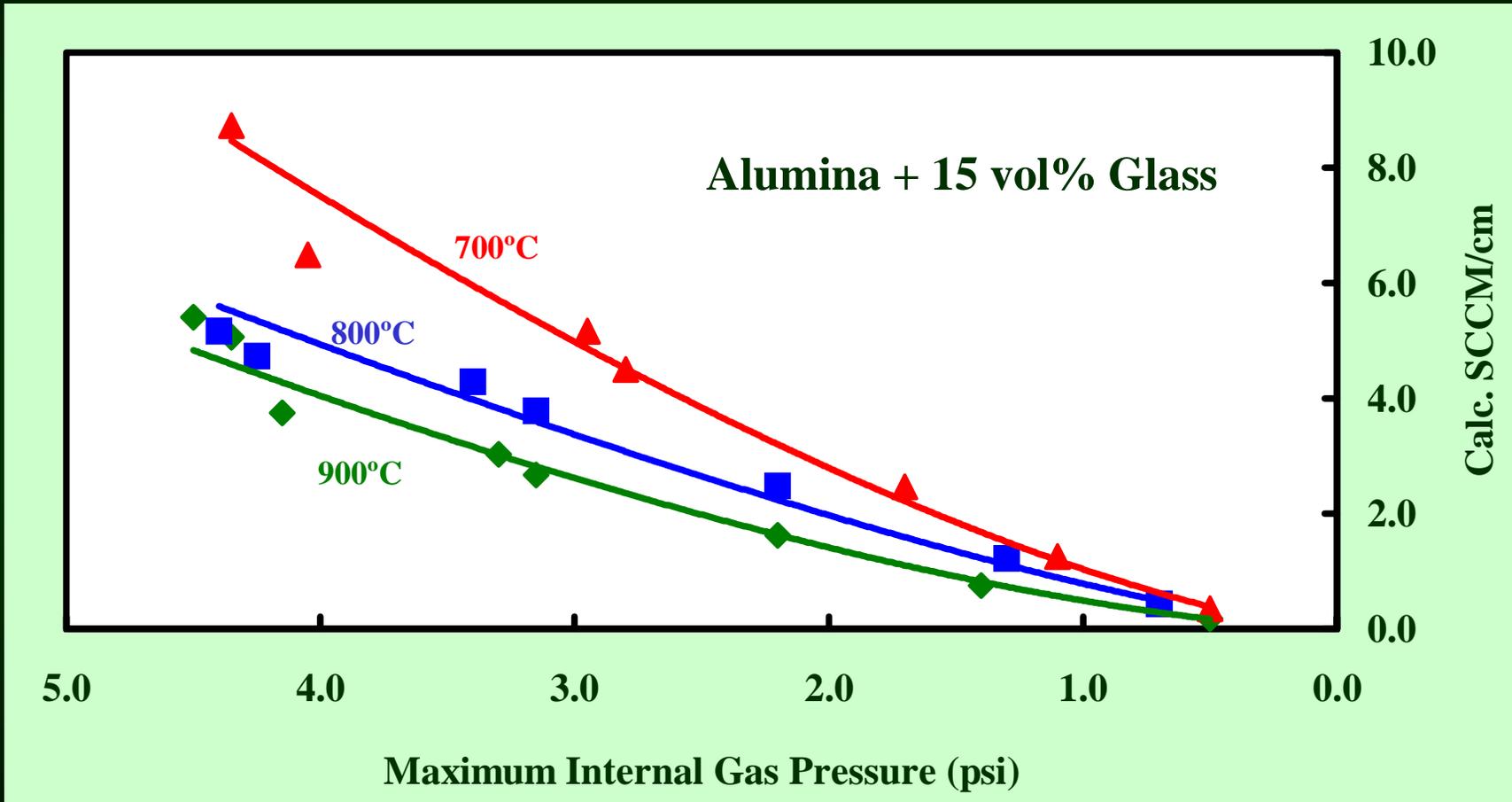


Seal Development—Glass Content



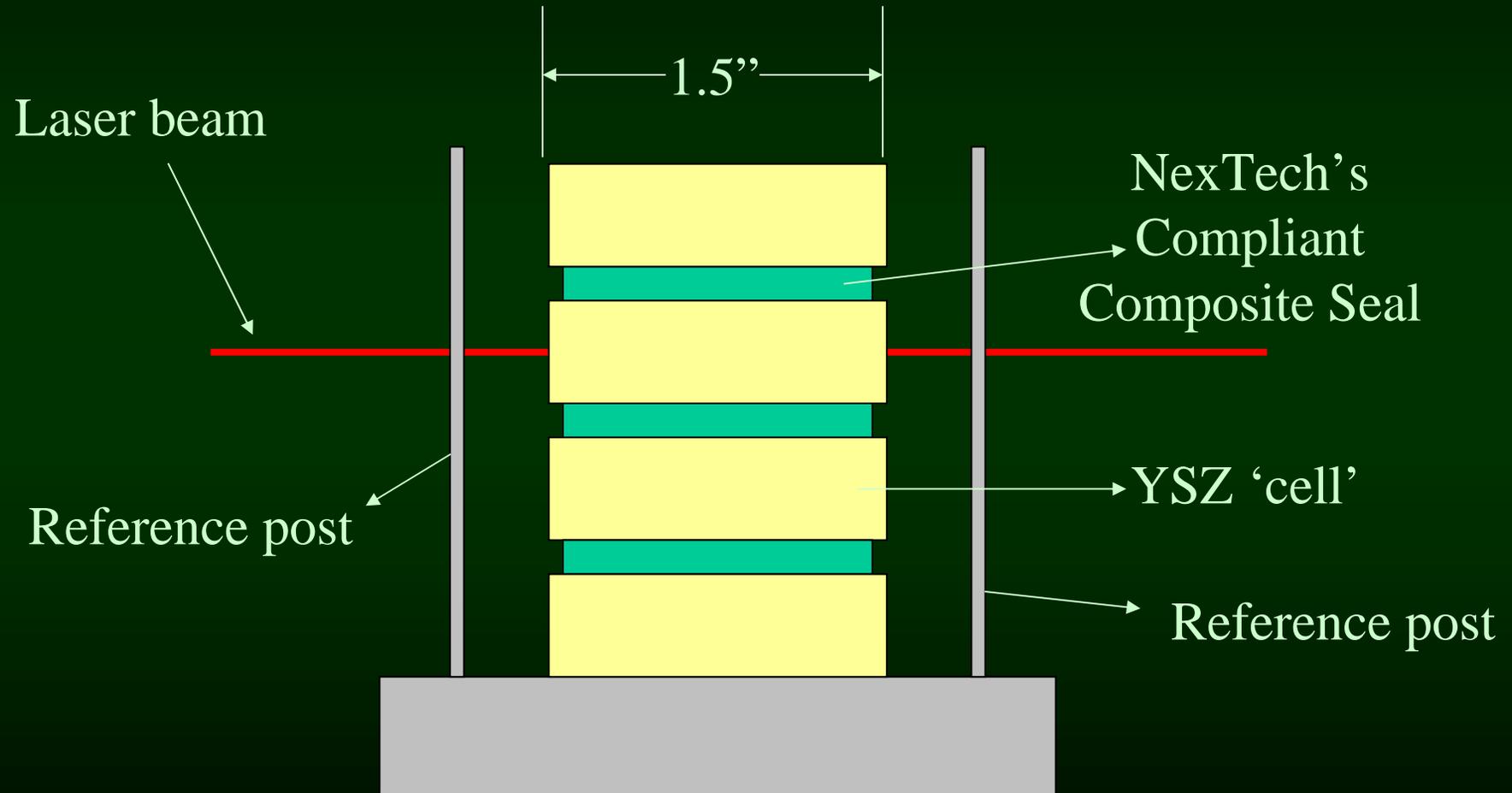
Seal Development:

Temperature and Pressure Effects

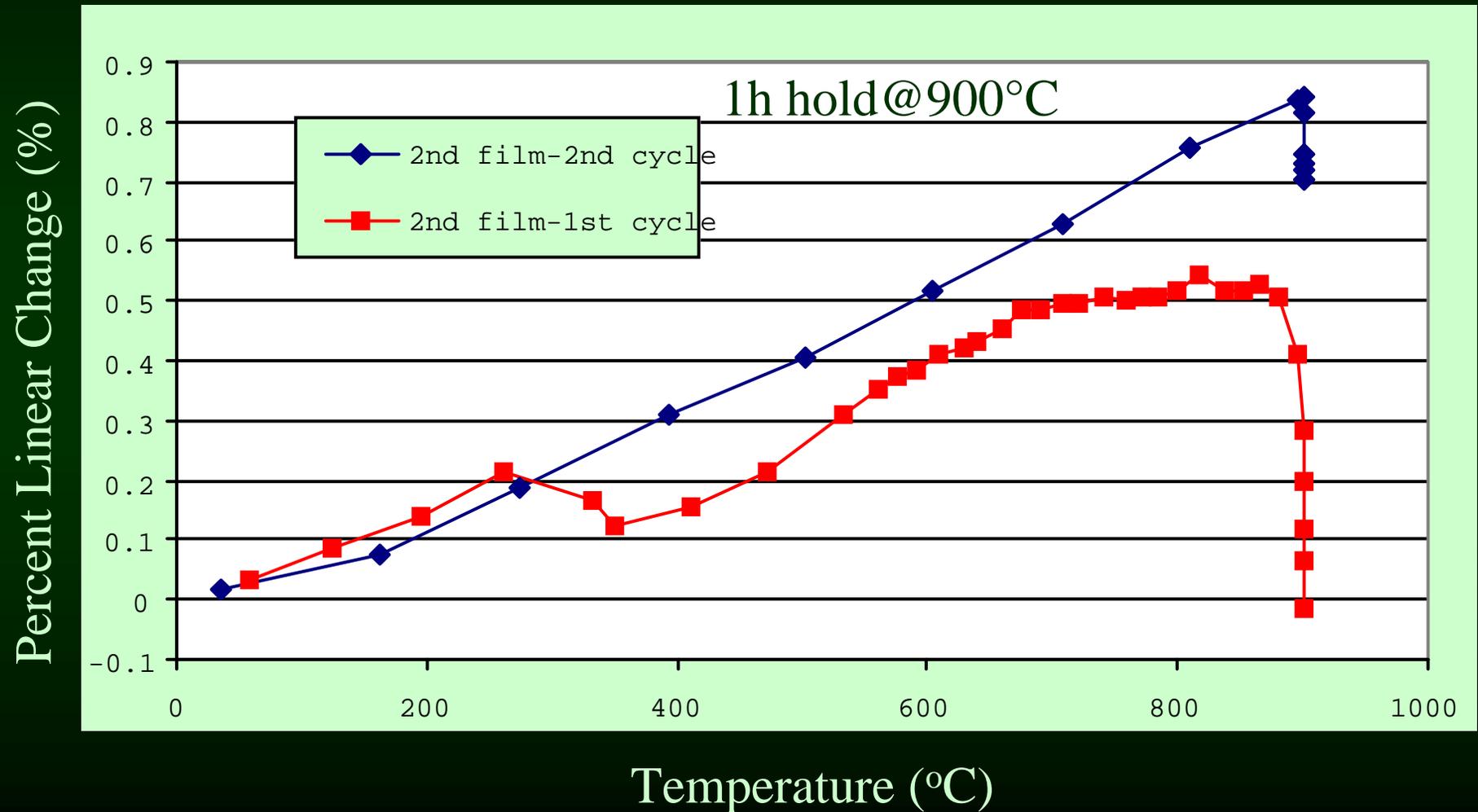


Scanning Laser Dilatometry

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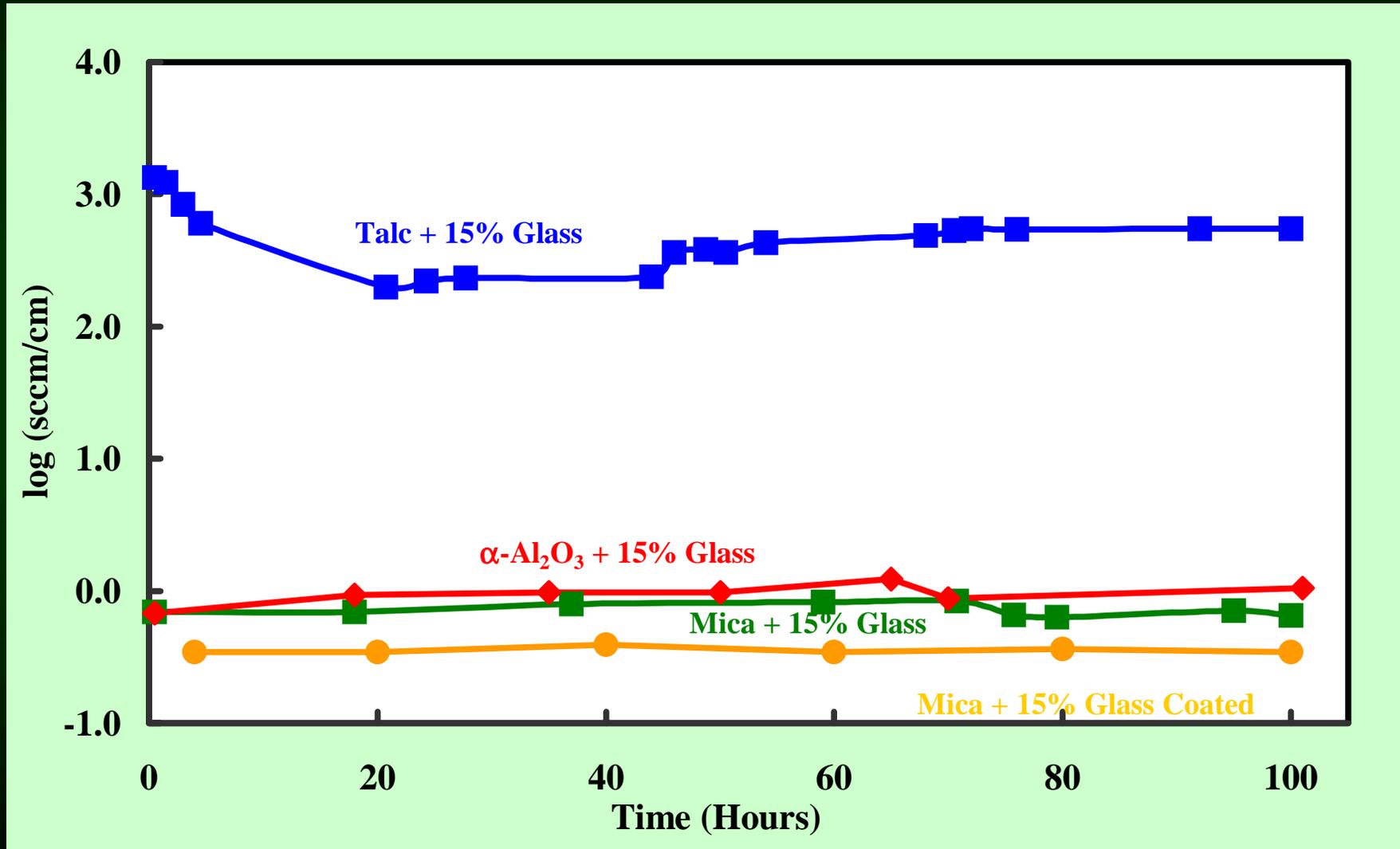


Dimensional Behavior of Seal vs. T and Cycle

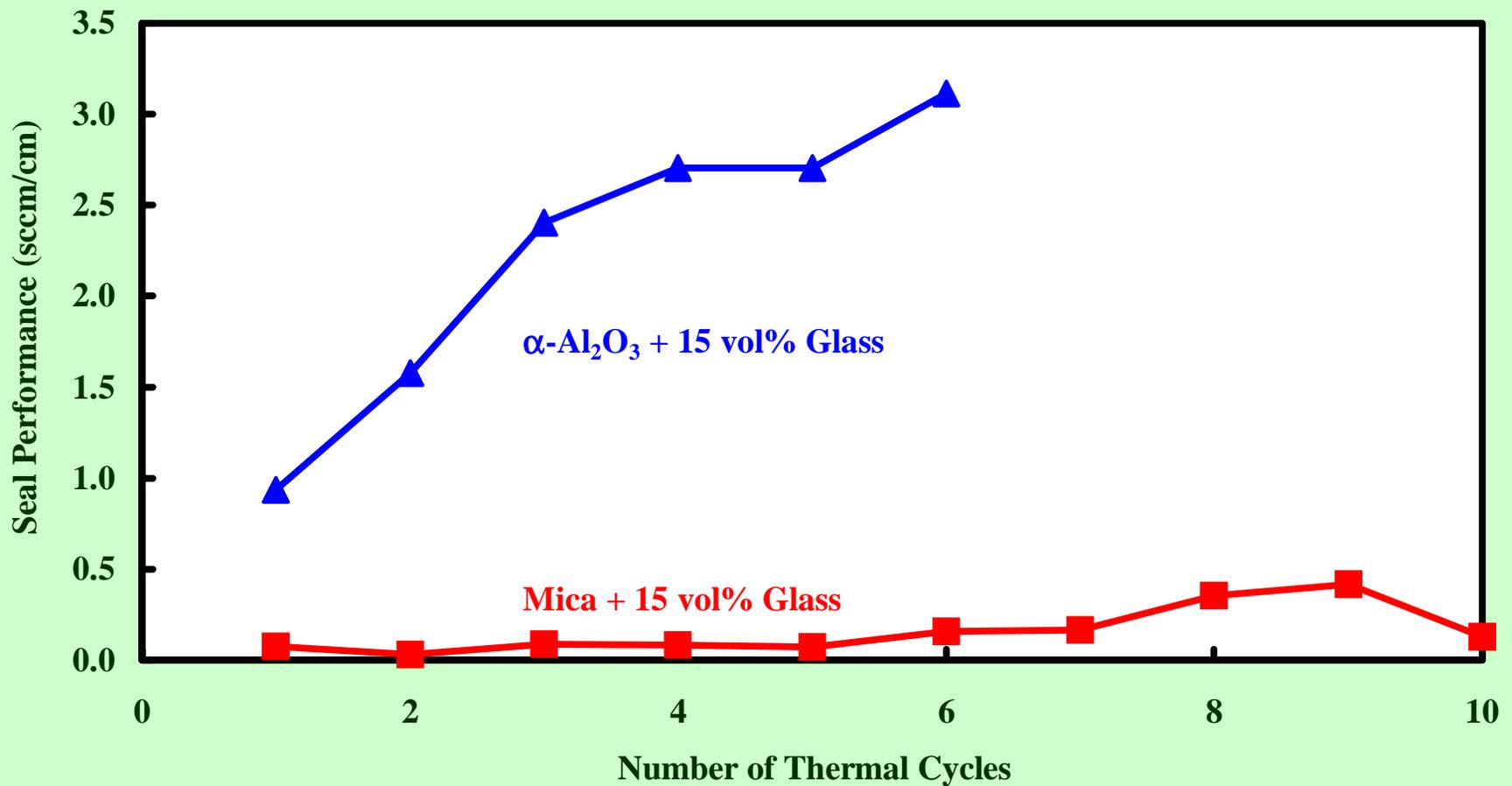


Seal Performance-100 h Tests

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Seal Performance-Thermal Cycling



Phase I Conclusions

- **Highly Textured Seal Materials Result from Tape Casting Process**
- **Green Tapes Amenable to Many Geometries**
 - **Gasket Approach Can Be Tailored**
 - **Thickness Controlled Through Lamination**
- **Seal Performance Controlled by Composition**
 - **Crystalline Phase: TCE, Mechanical Strength**
 - **Glass Content, Properties: Wetting, Stability**
 - **Binder Content**

Phase II Program Goals

- **Tailor Seals to SECA Vertical Team Targets**
 - Survey of Specifications from Vertical Teams
 - Identify Performance Target Specifications
 - Collaborative Evaluation as Target Specs Met
- **Identify Improved Glass Compositions:**
 - **Chemical Stability with Component Phases**
 - Tailored Compositions for Each Contact Surface?
 - **Good Wetting of Component Phases**
 - **Resistant to Devitrification**
- **Improve Shrinkage Match/Mechanical Compatibility**
 - **Reduce/Eliminate Need for Compression**
 - **Reduce Organic Content**

Phase II Plan and Timeline

- **Year 1**
 - **Survey SECA Team Members (Q1)**
 - **Experimental Matrix of Glasses (Q1-Q3)**
 - **Contact Specific Compositions**
 - **Evaluate Chemical Stability, CTE, Crystallization Resistance**
 - **Refine Green Forming, Glass Loading**
 - **Internal Evaluation of Seals**
 - **Button Cell Evaluations**
 - **10 x 10 cm Electrolyte Supported Cells**

Phase II Plan and Timeline

- **Year 2**
 - **Continued Evaluation/Refinement of Seal Materials—SOFC-based Testing**
 - **Quarterly Delivery of SECA Team Specific Seal Products**

Acknowledgements

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