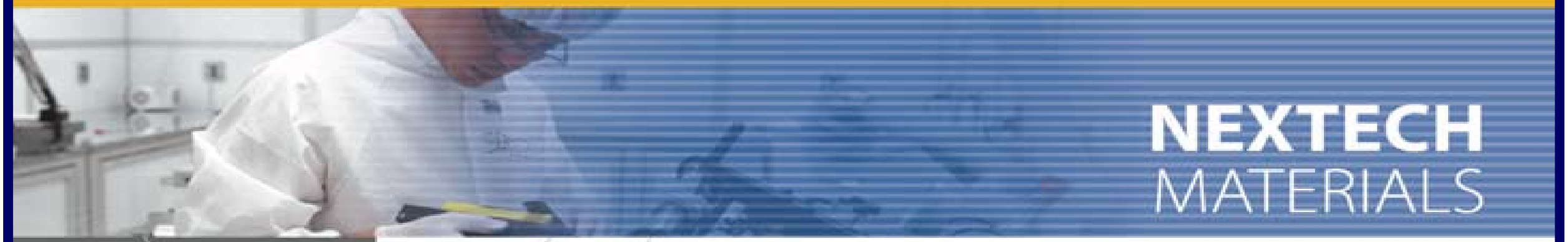

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# *Continuous Process for Low-Cost, High-Quality YSZ Powder*


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**Lewis Center, OH**


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**Project Details**

***Contract No.: DE-FC26-02NT41575***
***Project Monitor: Shawna Toth***
***Timeline: 10/1/2002 to 9/30/2005***



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## R&D Objectives

***Development of a low-cost synthesis process for YSZ electrolyte powder tailored or SOFC fabrication processes***

## Process Development

- Homogeneous precipitation
- Low-cost precursors
- Continuous – where possible
- Aqueous
- Agile

## Powder Quality Metrics

- Surface area: 10-15 $m^2$/gram
- Average particle size: <0.5 microns
- Sinterability: $\rho$ > 98% at $T_S$ < 1300°C
- Conductivity: $\sigma$ > 0.05 S/cm at 800°C

## Issues being Addressed

- Low-cost scalable powder synthesis and production processes.
- Lower sintering temperatures.
- Effects of dopants and processing on conductivity and mechanical properties.
- Long-term degradation of conductivity of zirconia-based electrolytes.
- Tailoring of YSZ electrolyte powder for different SOFC fabrication processes.
- Batch-to-batch reproducibility.

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# Applicability to SOFC Commercialization

***Different manufacturing processes are used for anode and electrolyte layers in SOFCs.***

| SECA Industry Team | Electrolyte Fabrication | Anode Fabrication |
|---|---|---|
| Delphi/Battelle | Tape Casting | Tape Casting |
| GE | Tape Calendaring | Tape Calendaring |
| Cummins/SOFCo | Tape Casting | Screen Printing |
| SWPC | Plasma-Spray | Plasma Spray |
| Fuel Cell Energy | Screen Printing | Tape Casting |
| Accumentrics | Dip Coating | Extrusion |

***Agile processing allows tailoring of YSZ powder production to the requirements of different SOFC fabrication methods***

- **Tape Casting Methods:** Tight control of particle size distribution is important; relatively low surface areas needed for high green density.
- **Co-Sintering Processes:** Lower sintering temperatures are desired; control of sintering shrinkage rates is essential.
- **Colloidal Deposition:** Dispersion chemistry is critical; higher surface areas can be tolerated; tailored particle size distributions are beneficial.
- **Plasma-Spray Methods:** Large particle size and spherical morphology are required for optimum flow characteristics.
- **Extrusion:** Lower surface areas needed for dimensional control; particle size requirements vary by developer.
- ***Batch-to-batch reproducibility is essential for all processes!***

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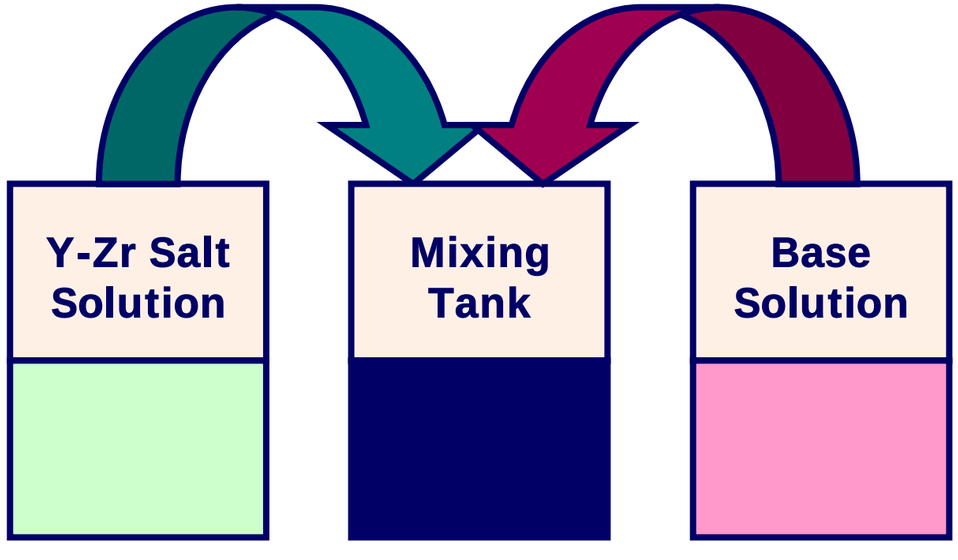
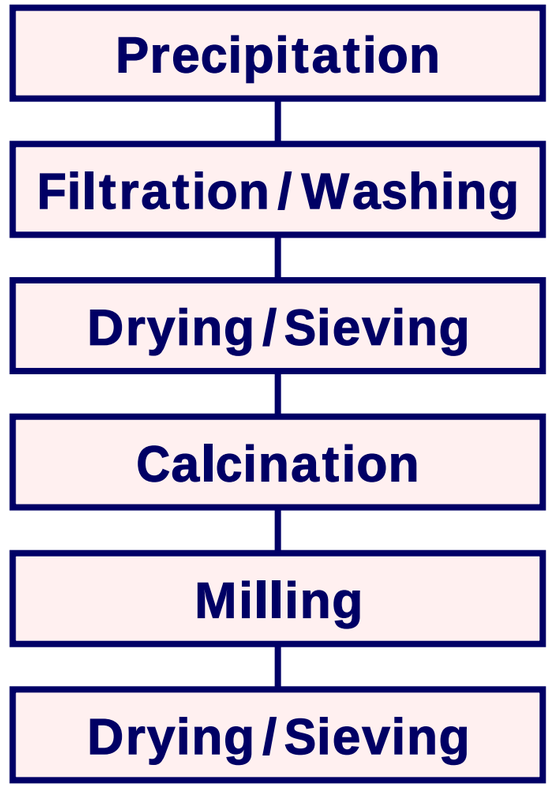
# Powder Synthesis and Processing

## Homogeneous Precipitation

***pH remains constant throughout process***




## Synthesis Process Variables

- Batch Size (typically 3-5 kg)
- Precipitation Conditions
- Chemical Purity (e.g., silica content)
- Dopants – sintering aids
- Solvent System (water or alcohol)
- Drying Methods
- Calcination – control of surface area
- Milling Methods – particle size control

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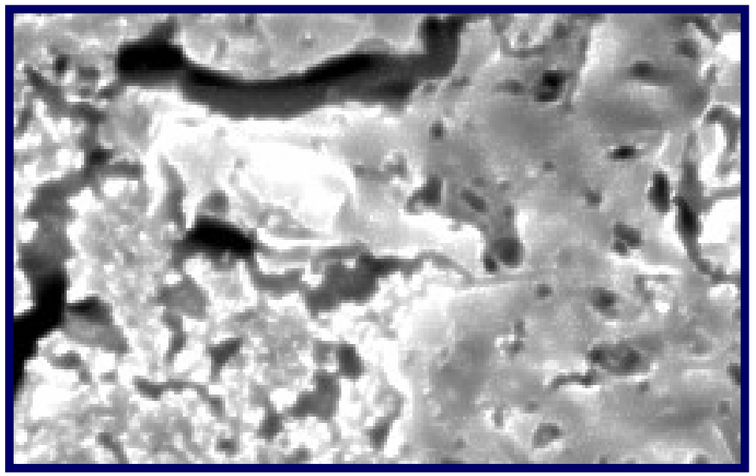
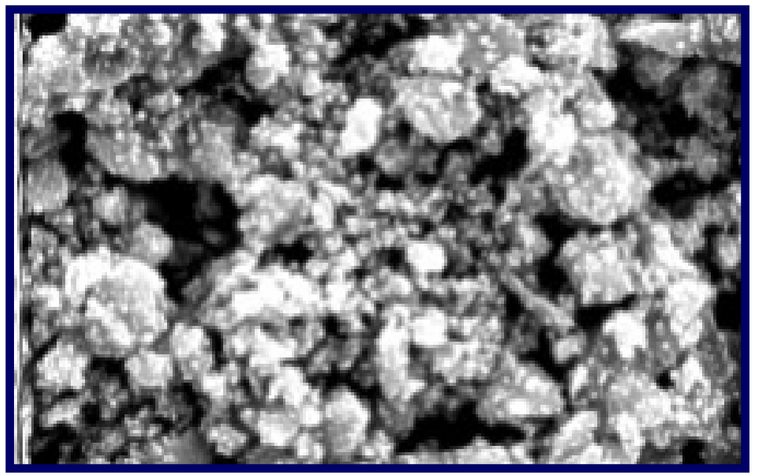
# Non-Optimized Process



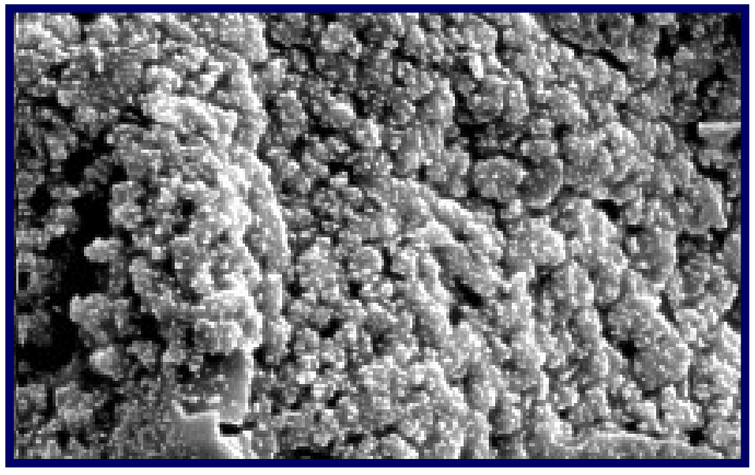
As-Precipitated

1 μm

Calcined and Milled



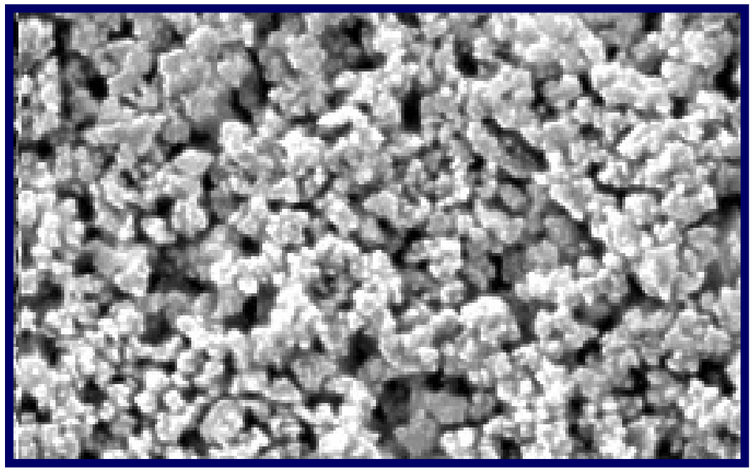
# Optimized Process



As-Precipitated

1 μm

Calcined and Milled



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# Powder Evaluation Protocol

## Powder Characterization

- Particle Size Distribution (centrifugal analysis)
- Surface Area (multi-point BET)
- Chemical Analysis (ICP)

## Sintering Performance Studies

- Samples: pressed pellets or tape-cast substrates
- Temperature range: 1100 to 1400°C
- Density measurements by Archimedes method

## Characterization of Sintered Ceramics

- Ionic conductivity (four-point method)
- Long-term conductivity testing
- Mechanical properties
- Microstructural analyses

## Process Development Challenges

- Assuring “apples to apples” comparisons.
- Drying and milling processes are more efficient at larger production scales.
- Labor-intensive process at current scale of production.
- Complex relationships between precipitation variables on downstream processes.
- Difficult to achieve simultaneous control of surface area and particle size during milling.
- Lack of accelerated tests for long-term stability.

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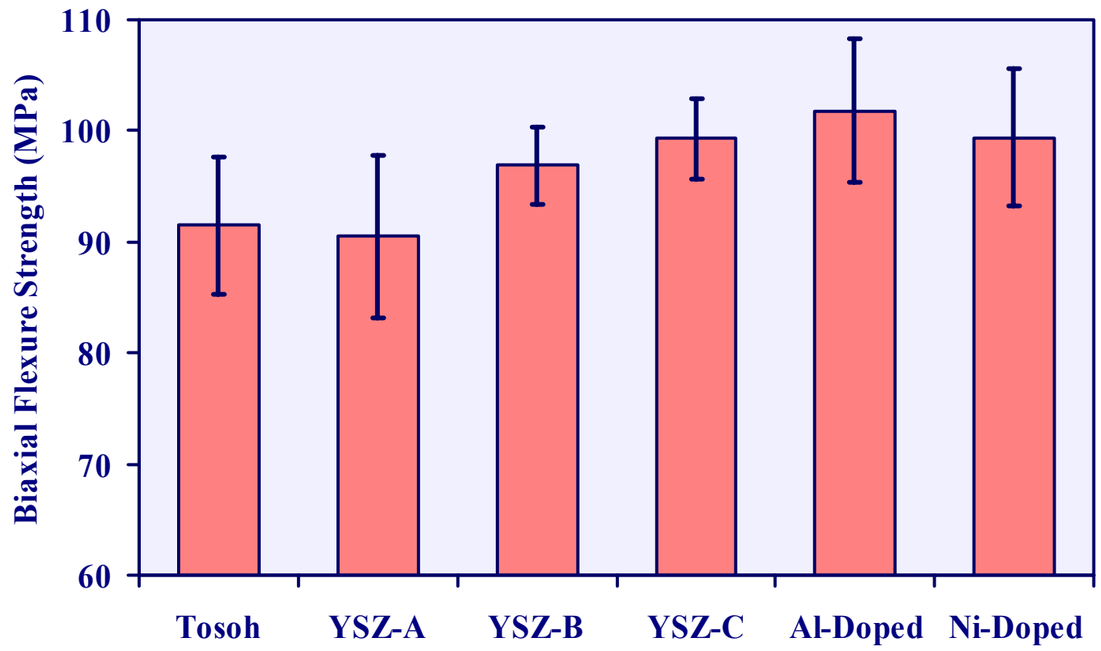
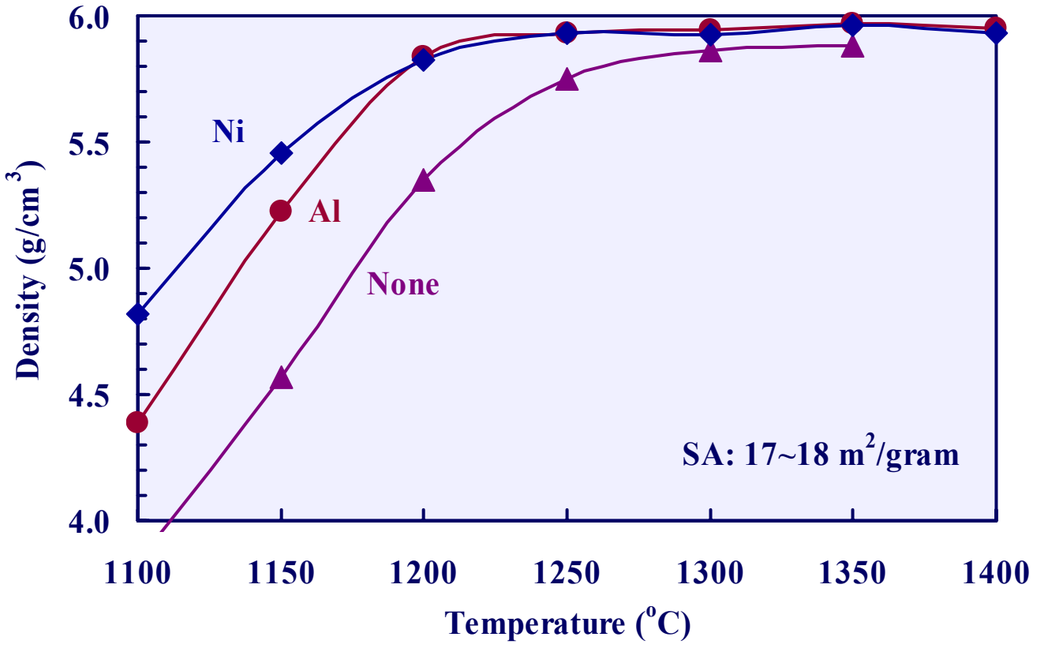
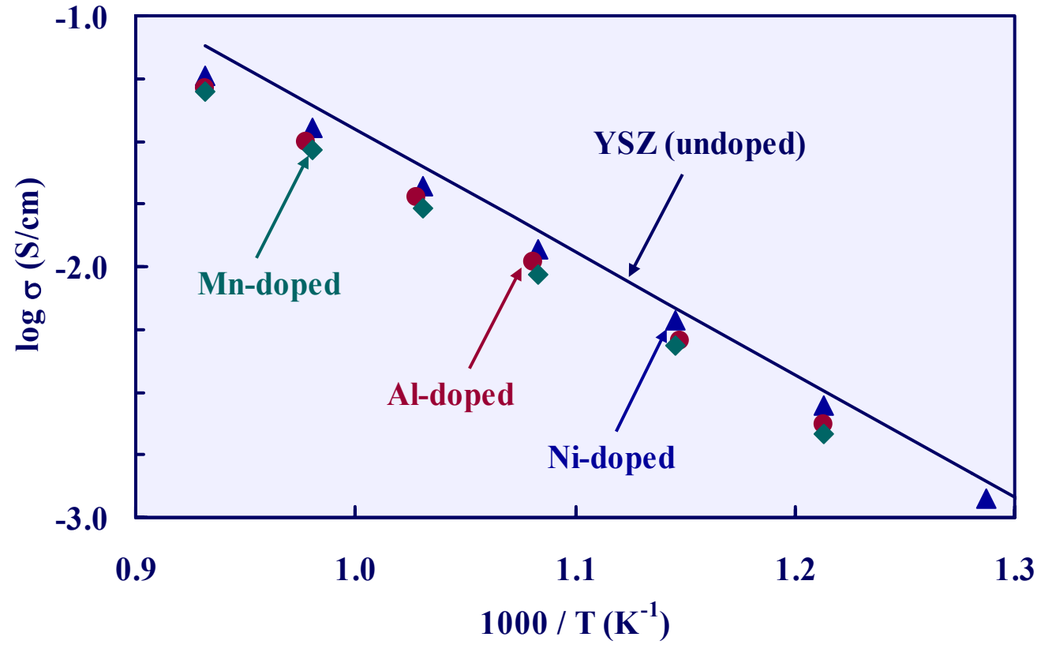
# Ceramic Performance Studies



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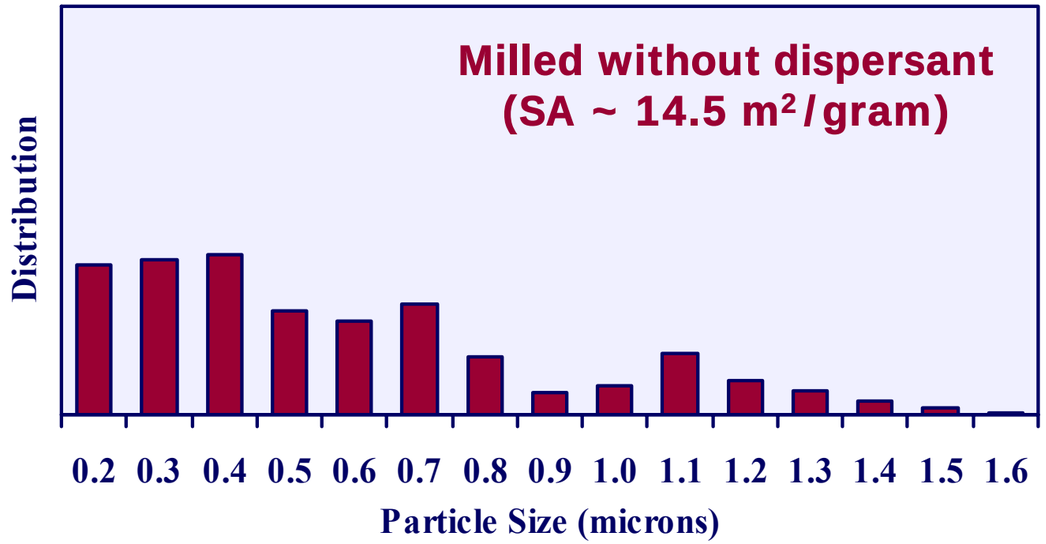
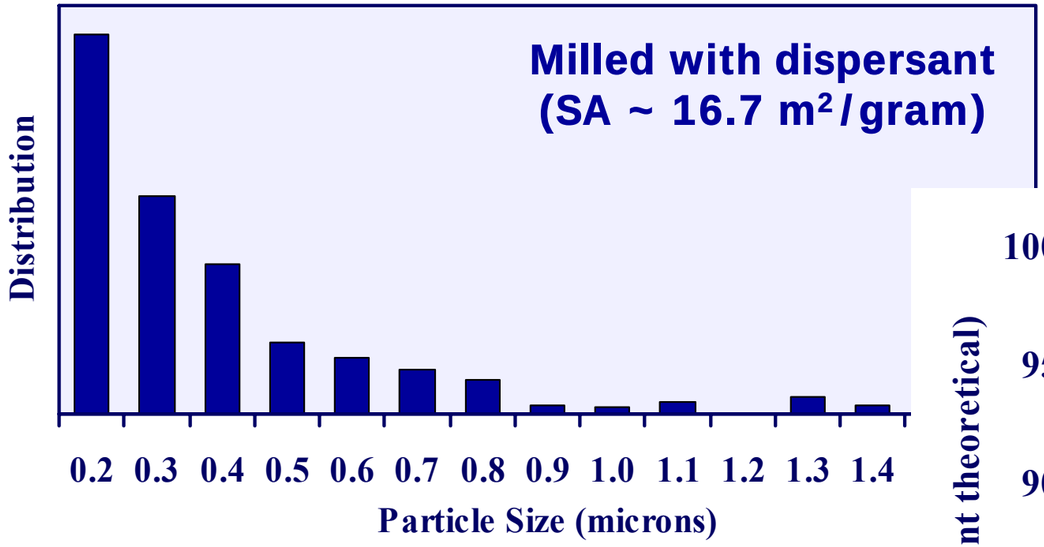
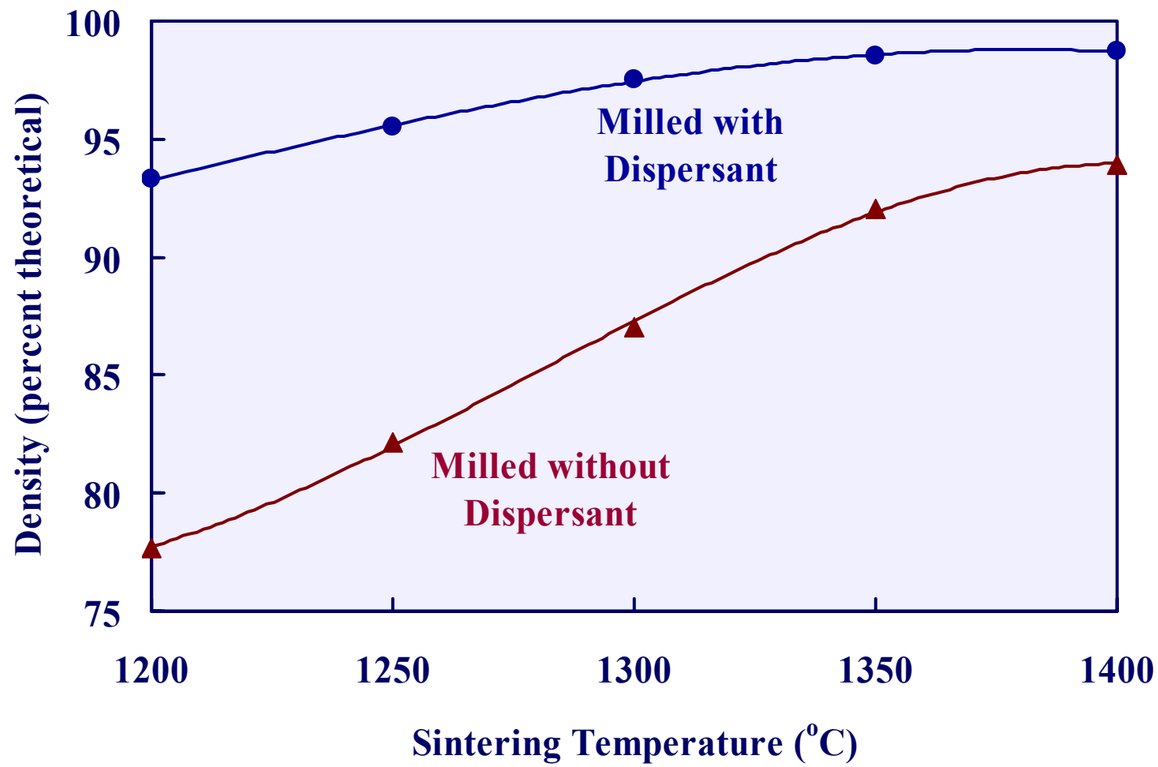
# Process Development Studies



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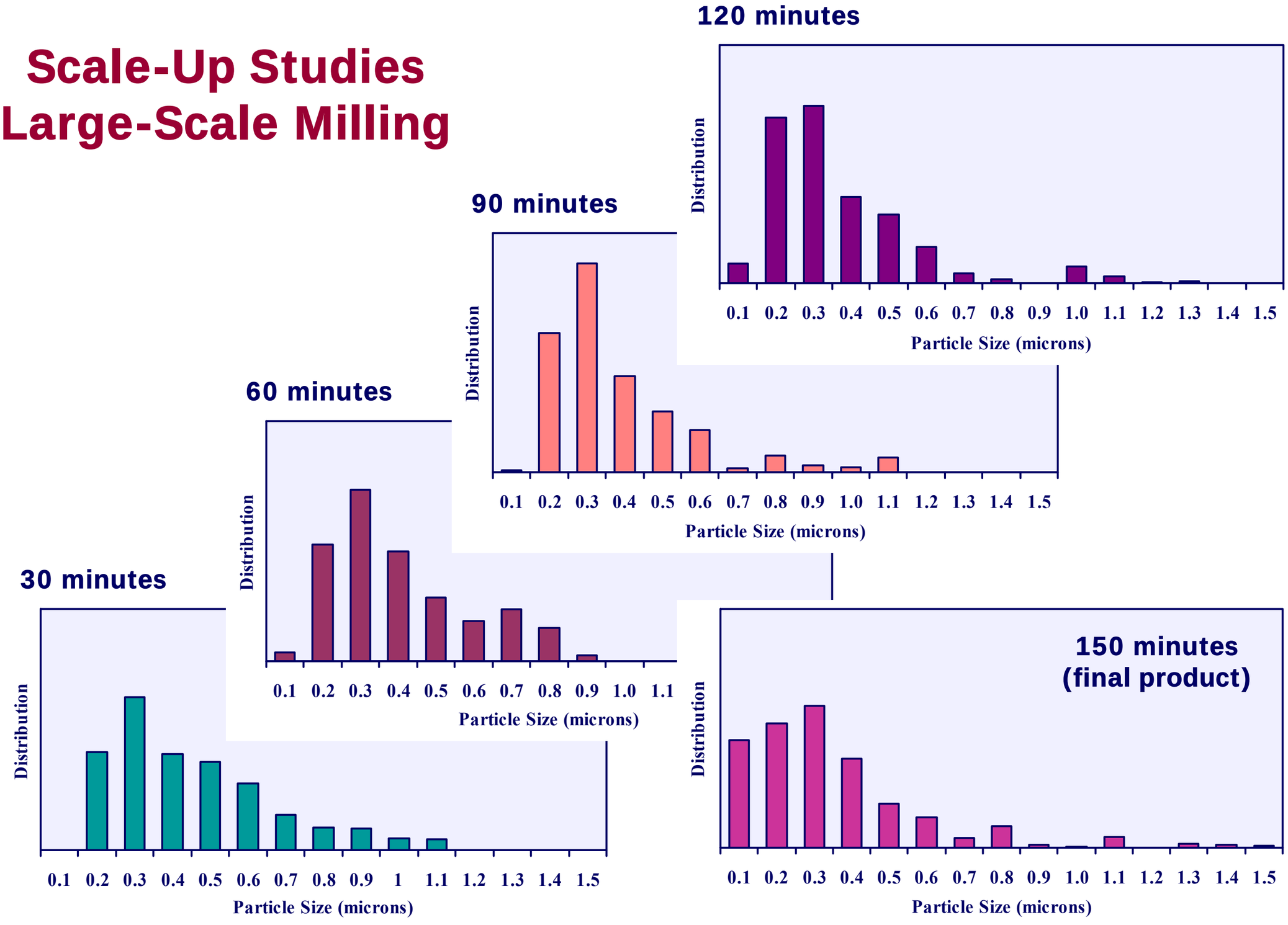
# Scale-Up Studies
# Large-Scale Milling



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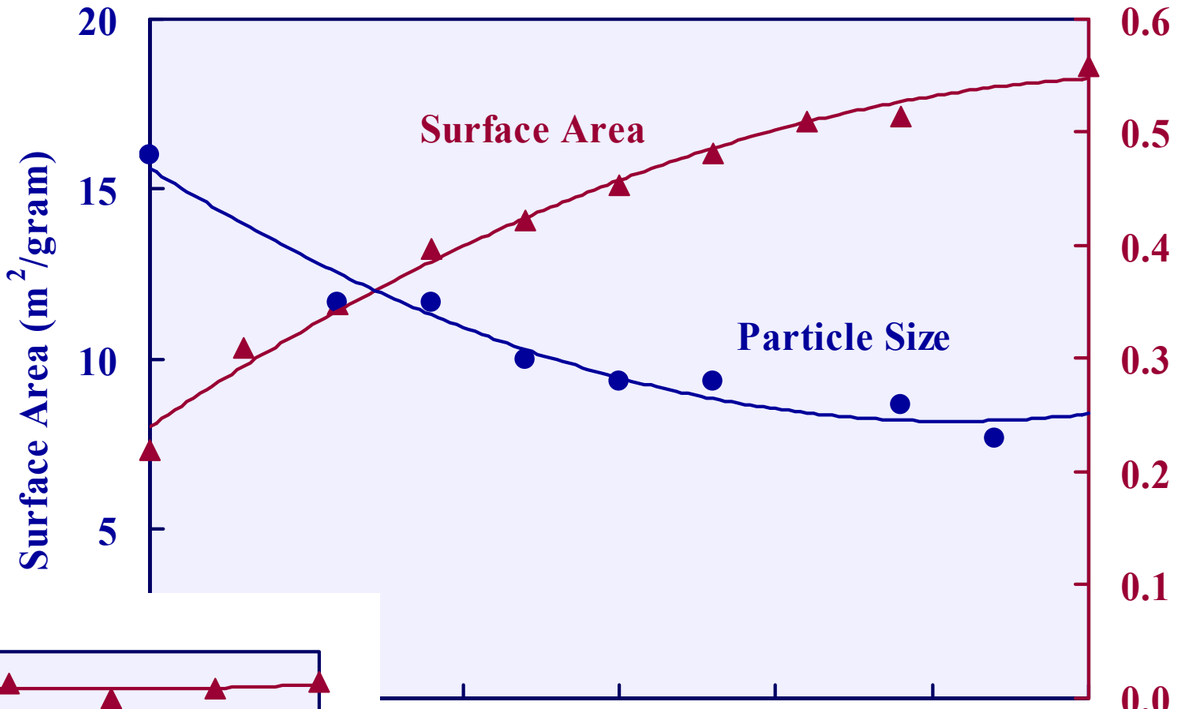


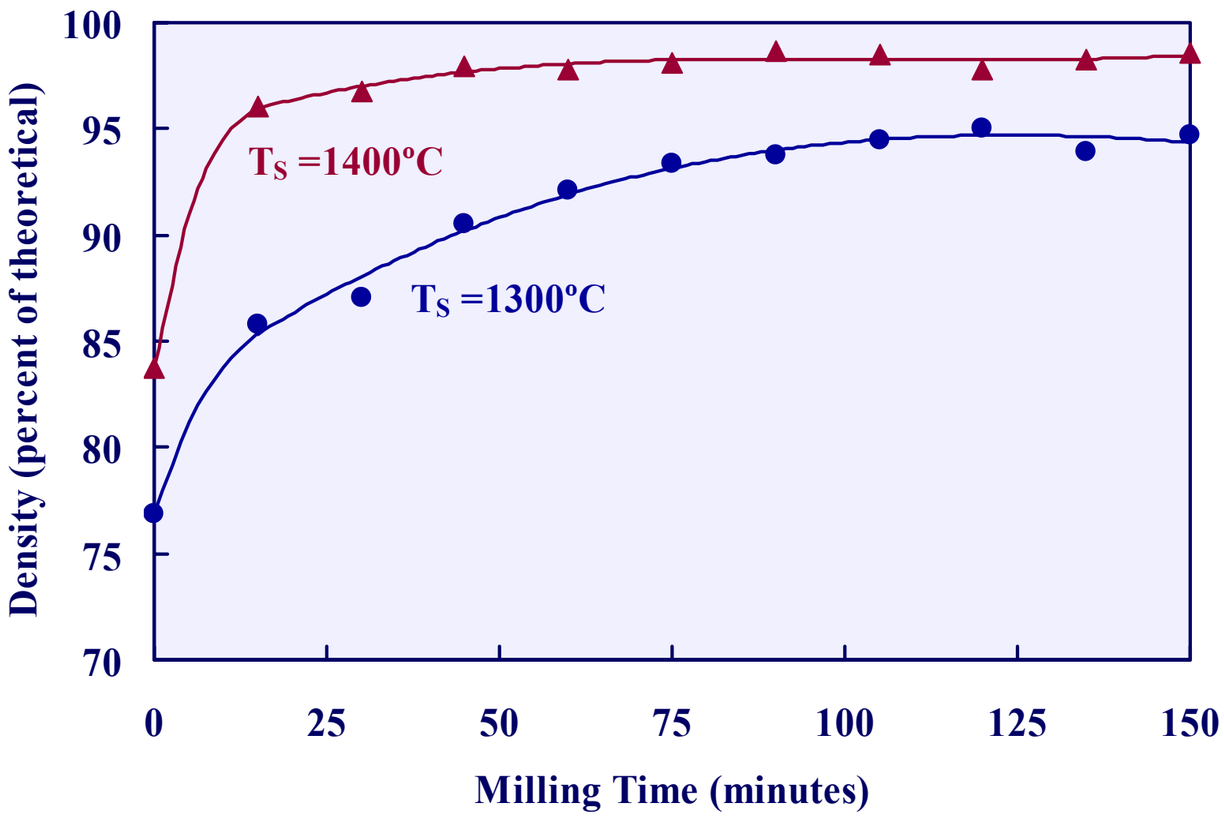

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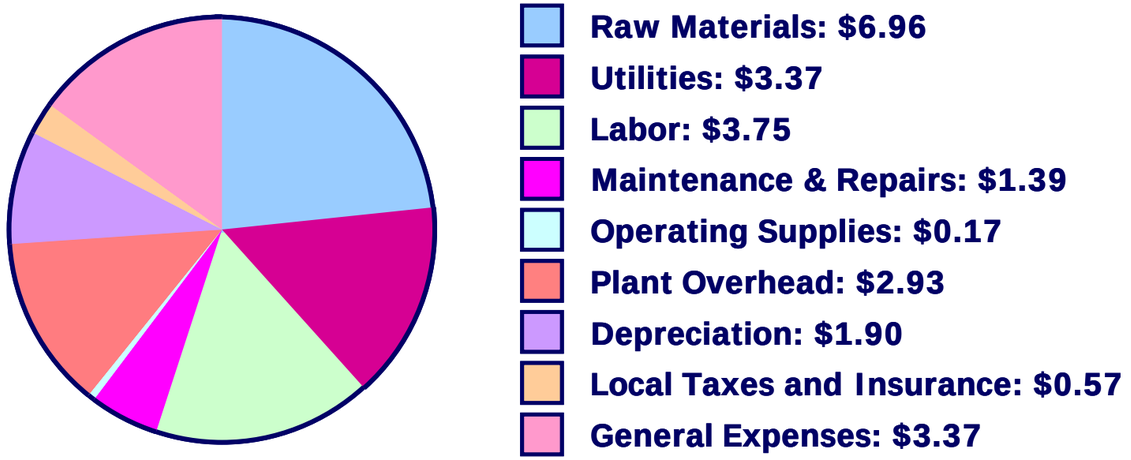
# Summary of Results (to date)

- **Established homogeneous precipitation process for synthesis of YSZ powders.**
- **Established calcination and milling methods to meet surface area and particle size targets.**
- **Achieved state-of-the-art performance levels, relative to commercially available YSZ powders:**
  - **Improved low-temperature sinterability**
  - **Achieved identical ionic conductivity values**
- **Demonstrated reproducibility of baseline process.**
- **Demonstrated potential for achieving manufacturing cost of less than $25/kg target.**
  - **Identified cost drivers for process.**

## Manufacturing Cost Analysis

- **Plant Size: 500 MT/year**
- **Fixed Capital Investment: $11.2 M**
- **Cost per kilogram: $24.41**



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# Future Plans

- Adapt process to scandium doped zirconia electrolyte compositions
- Demonstrations in SOFC fabrication processes
  - Tape casting
  - Ultrasonic spray deposition
- Scale-up to 5-kg batch sizes
- Production of evaluation samples for SECA industry teams
- Conduct manufacturing cost analysis on final process

## Look who's interested in fuel cells!


## Acknowledgments


- DOE / SECA
- Shawna Toth & Don Collins
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- NexTech's Team
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