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

- **SECA Core Technology Project**
  - Contract No. DE-FC26-02NT41575
  - Project Monitor: Shawna Toth
  - Start Date: October 1, 2002
- **NexTech's Team**
  - Principal Investigator: Scott Swartz
  - Lead Engineer: Michael Beachy
  - Scientific Support: Matt Seabaugh
  - Technical Support: NexTech's Fuel Cell Group

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

Outline

- Technical Issues Addressed
- R&D Objectives and Approach
- Phase I Results
- Applicability to SOFC Commercialization
- Activities for the next 6-12 Months

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Technical Issues being Addressed

- **Most advanced synthesis processes for high-purity YSZ powder are expensive:** **Lower-cost scalable powder synthesis processes are needed to meet SECA's cost targets.**
- **Chemical purity of YSZ has a significant impact on long-term degradation:** **Dopant strategies are being pursued to address silica contamination (rather than expensive purification steps).**
- **Commercially available YSZ powders require high sintering temperatures:** **This results in higher energy costs, increased chemical interactions, and difficulties in co-sintering.**
- **Most YSZ powder suppliers offer a *one-size-fits-all* product:** **Tailoring of the YSZ electrolyte powder for different SOFC fabrication processes would provide significant advantages.**

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Key Technical Issue: Purity

- Silica is a known cause of long-term degradation of YSZ conductivity and electrode reactions.
- Three approaches to address silica contamination:
  - High purity raw materials (expensive)
  - Additional purification steps during synthesis (expensive)
  - Dopants (e.g., alumina)
- Potential benefits of alumina dopants:
  - Allows use of less expensive raw materials
  - Limits degradation (sequesters silica in triple points)
  - Reduces sintering temperature

Dopant strategy must be validated via long-term performance and comprehensive microstructural studies!

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R&D Objectives

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

❖ Process Development Goals

- Homogeneous precipitation
- Utilization of low-cost precursors
- Continuous – where possible
- Aqueous
- Agile

❖ Powder Quality Metrics

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

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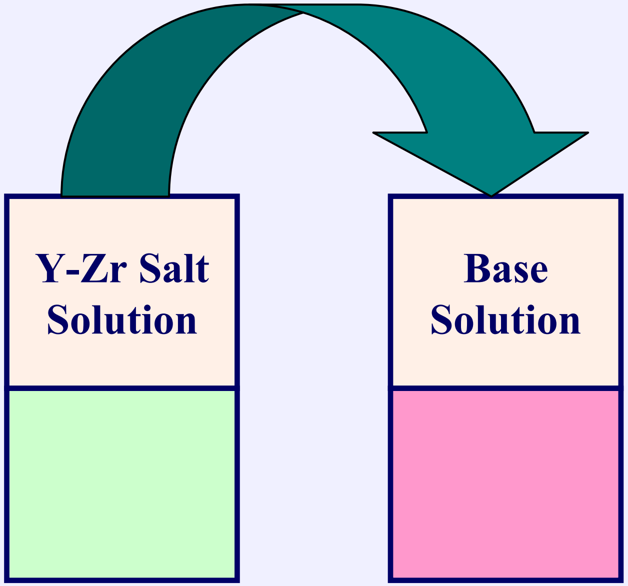
Continuous Precipitation

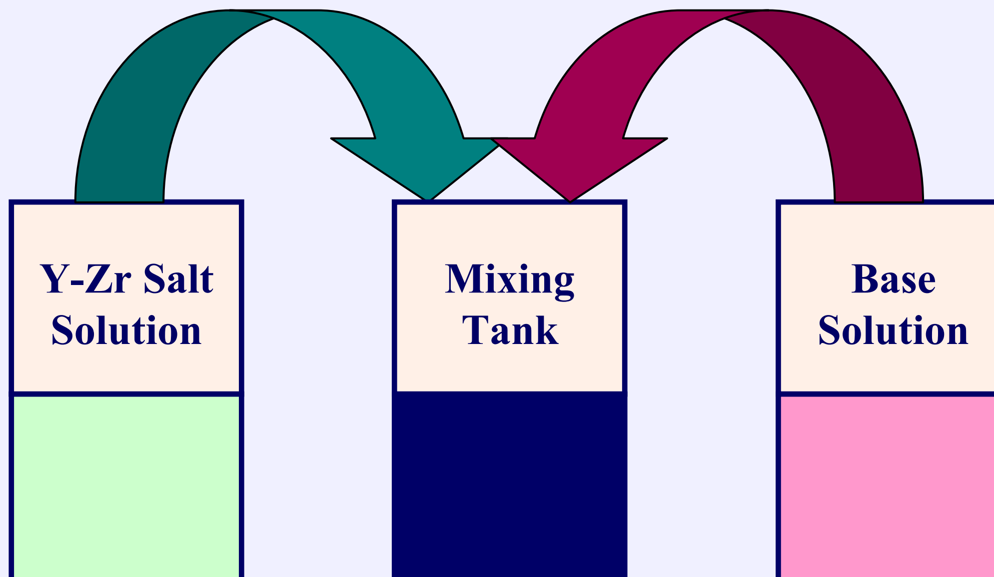


Standard Precipitation

pH varies continuously during process

Homogeneous Precipitation

pH remains constant throughout process



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Powder Processing Approach

- Precipitation
- Filtration/Washing
- Drying/Sieving
- Calcination
- Milling
- Drying/Sieving

Synthesis Process Variables

- *Batch Size (typically 1-3 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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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: 1200 to 1400°C
- Density measurements by Archimedes method

Characterization of Sintered YSZ Ceramics

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

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Phase I Results

- 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 industry standard (Tosoh) YSZ powder:
  - Improved low-temperature sinterability (at same surface area)
  - Identical ionic conductivity
- Demonstrated potential for achieving manufacturing cost of less than $25/kg target.

Achieved all proposed Phase I powder morphology and ceramic performance metrics.

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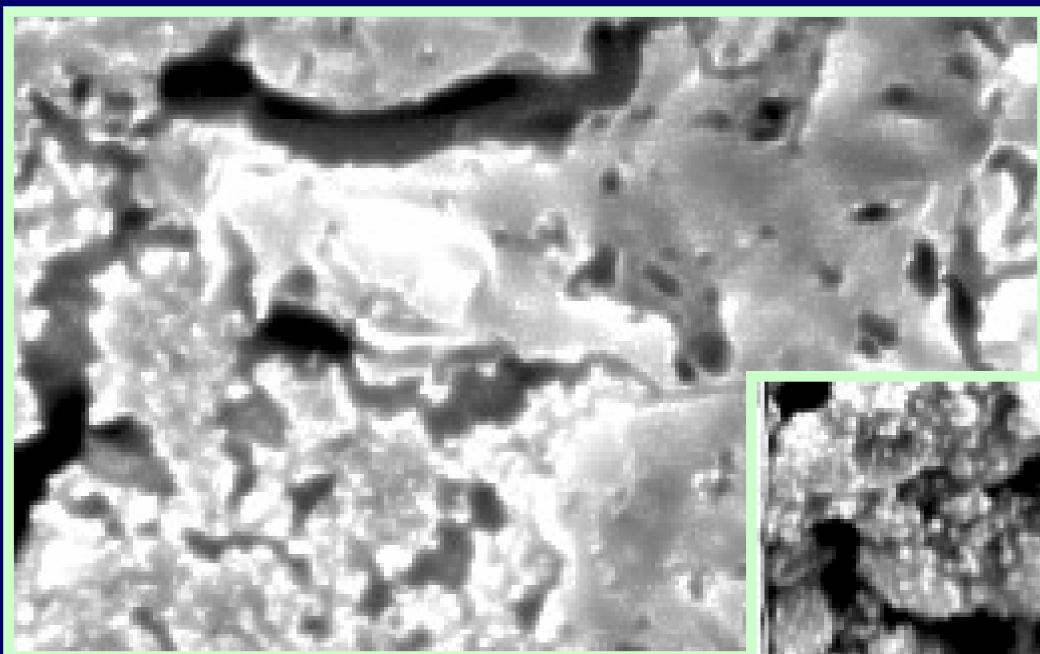
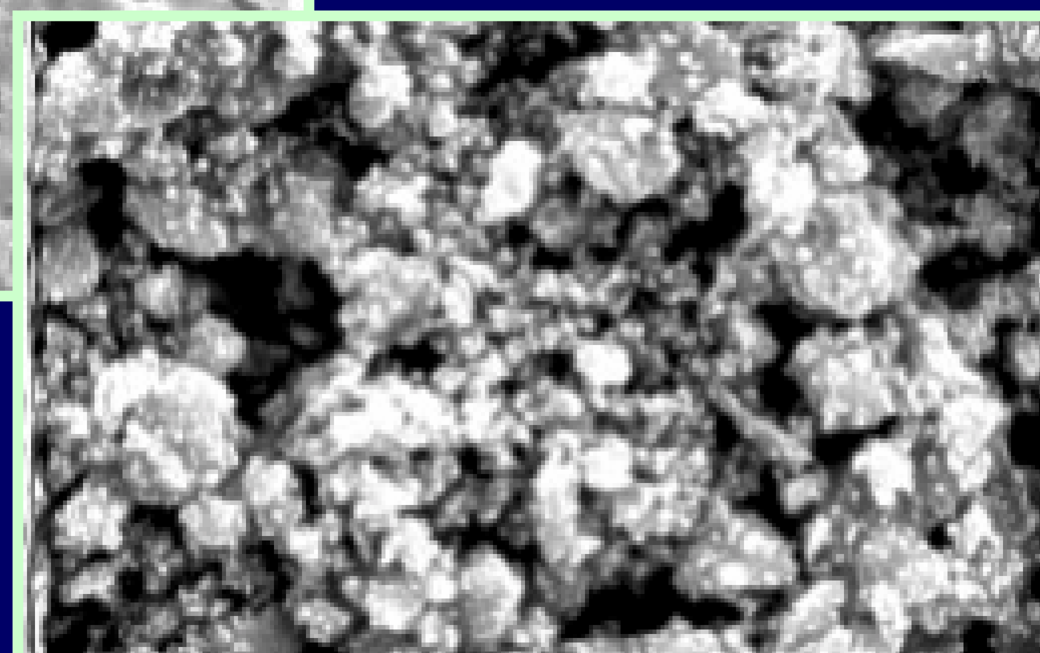
Non-Optimized Process



As-Precipitated

Calcined and Milled



1 µm

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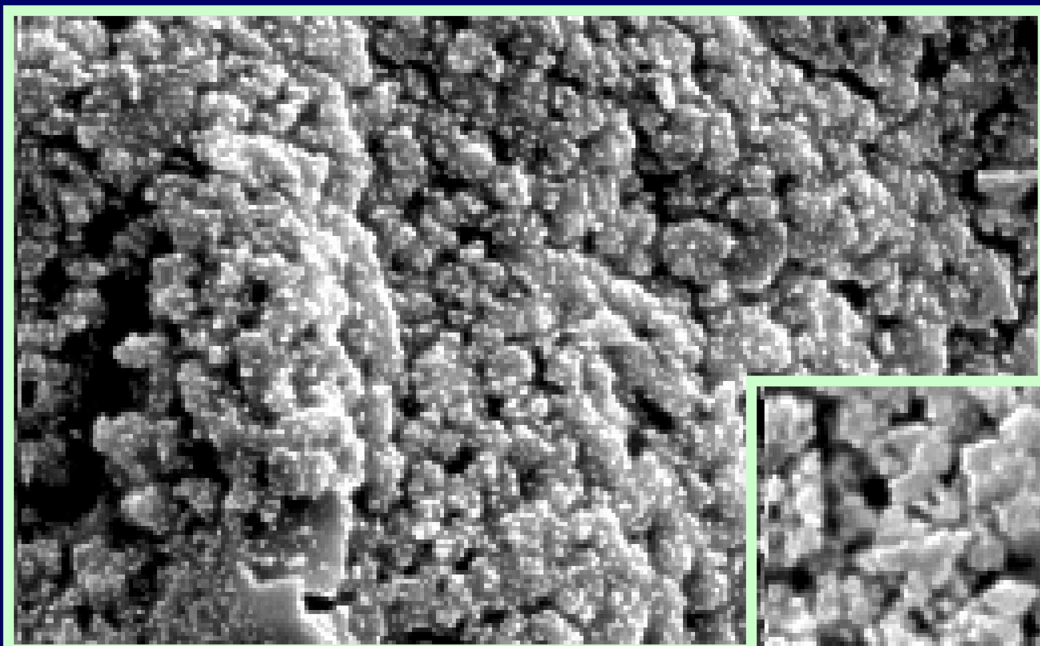
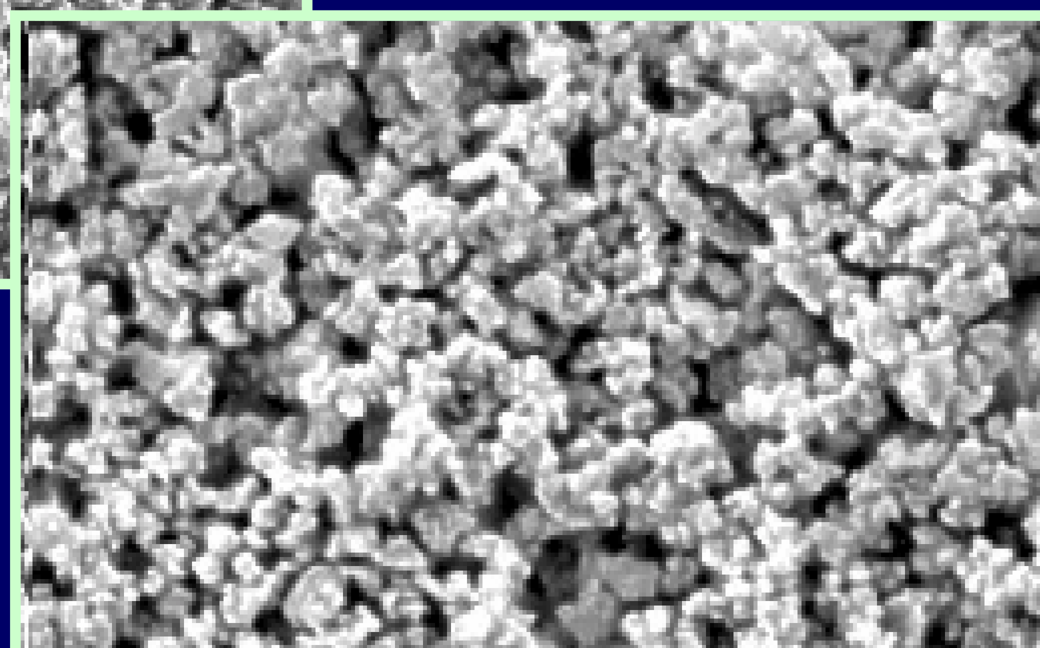
Optimized Process



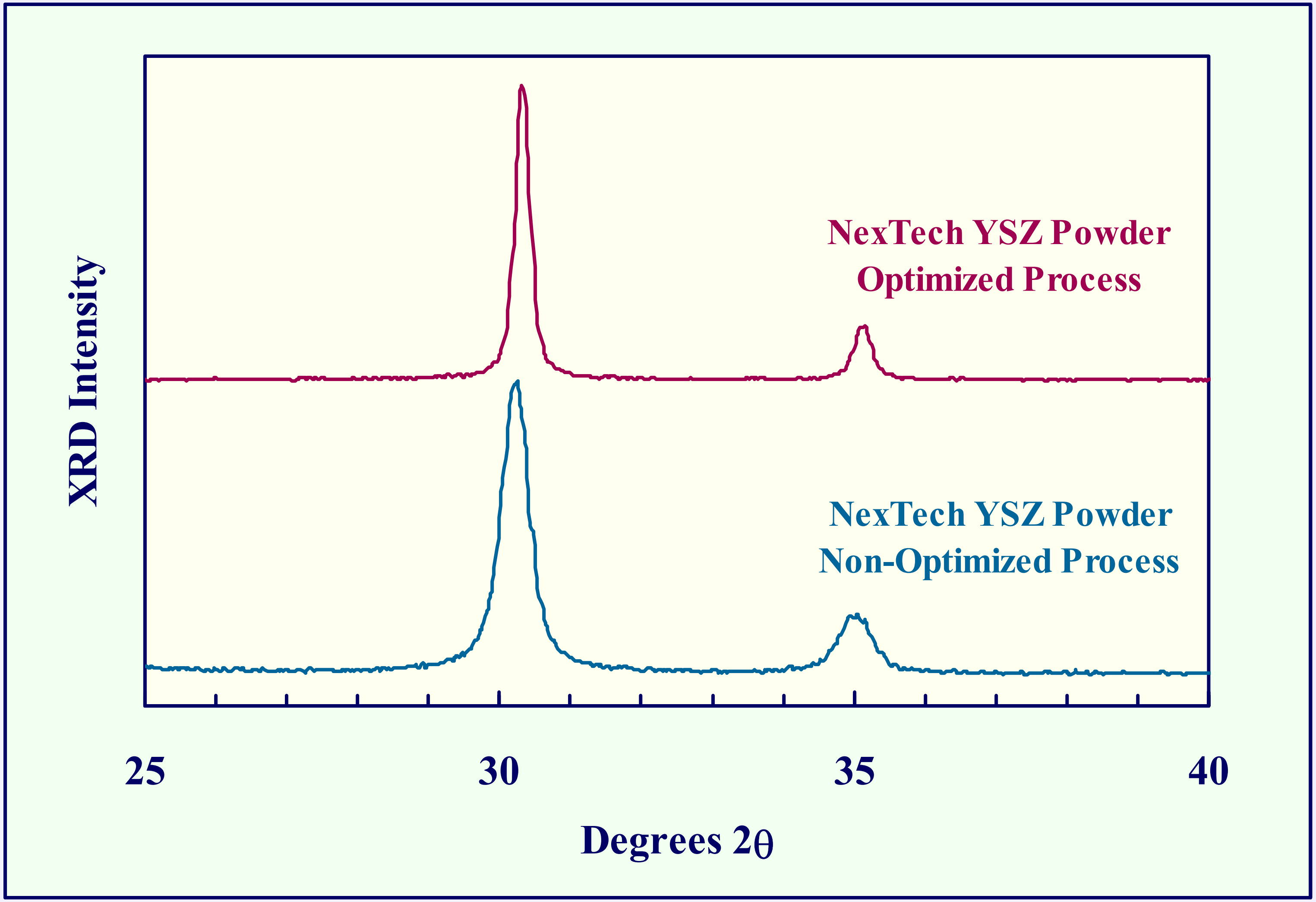
As-Precipitated

Calcined and Milled

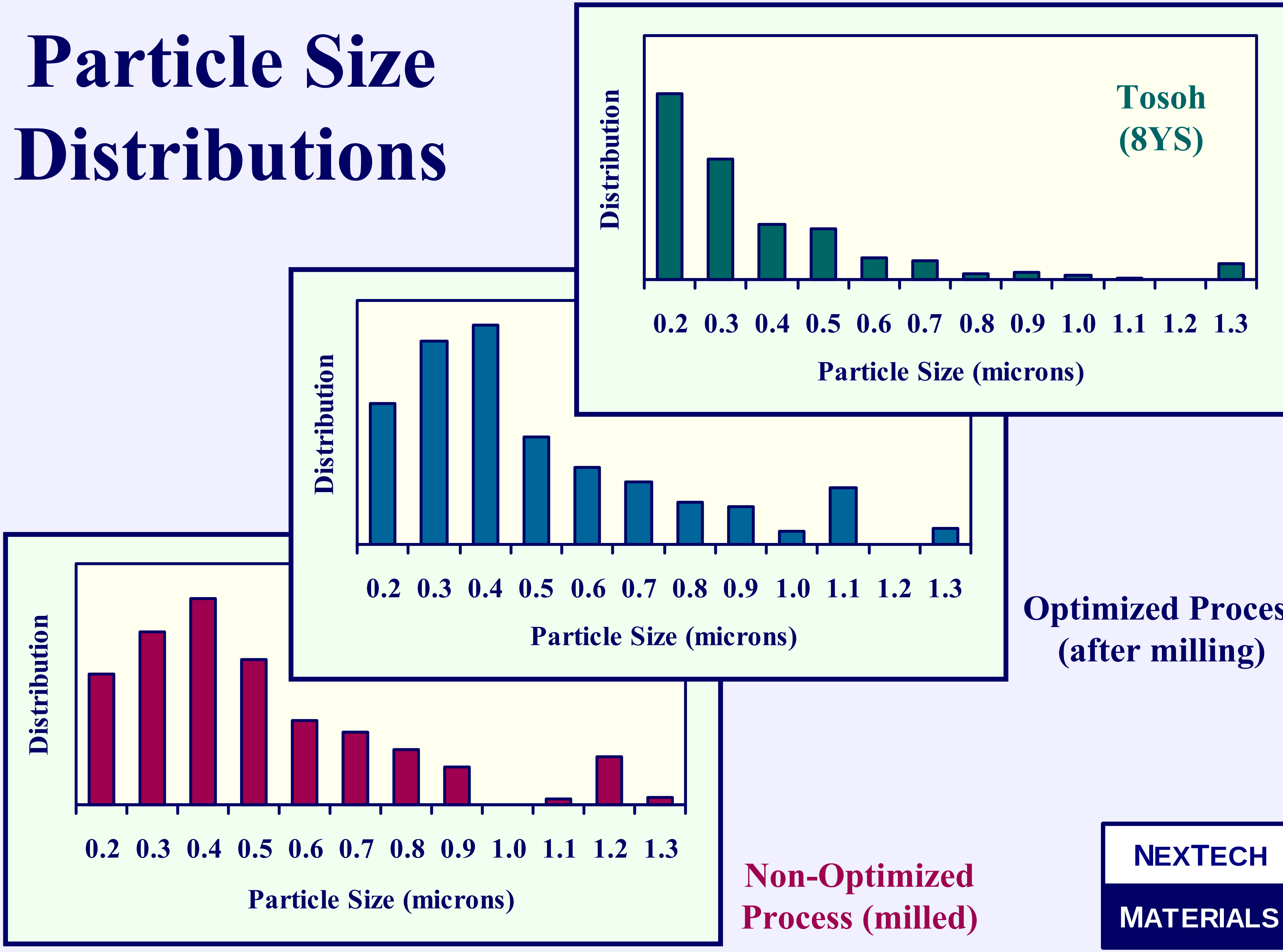


1 µm

X-Ray Diffraction



Particle Size Distributions



Optimized Process (after milling)

Non-Optimized Process (milled)

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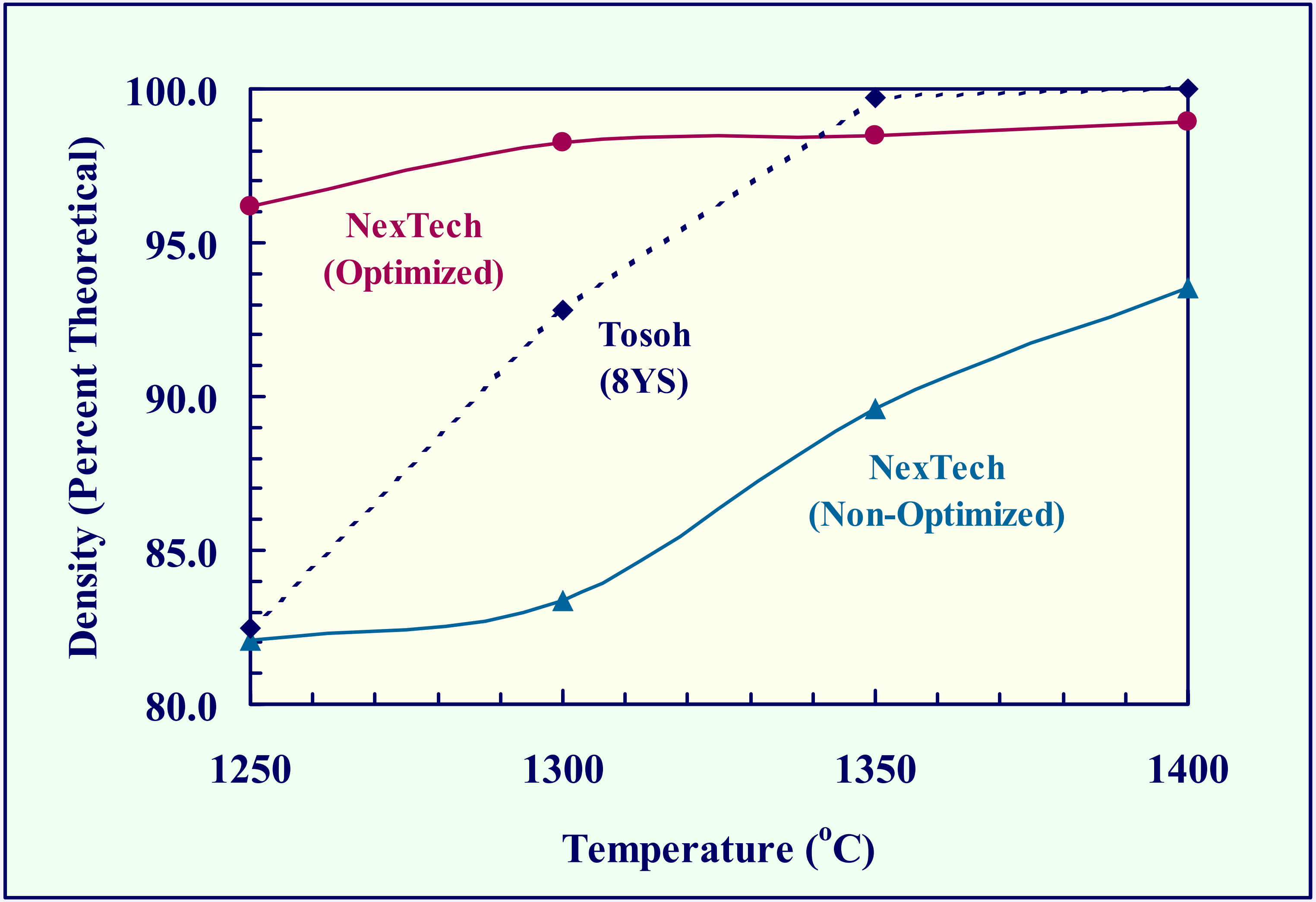
Sintering: Effect of Synthesis



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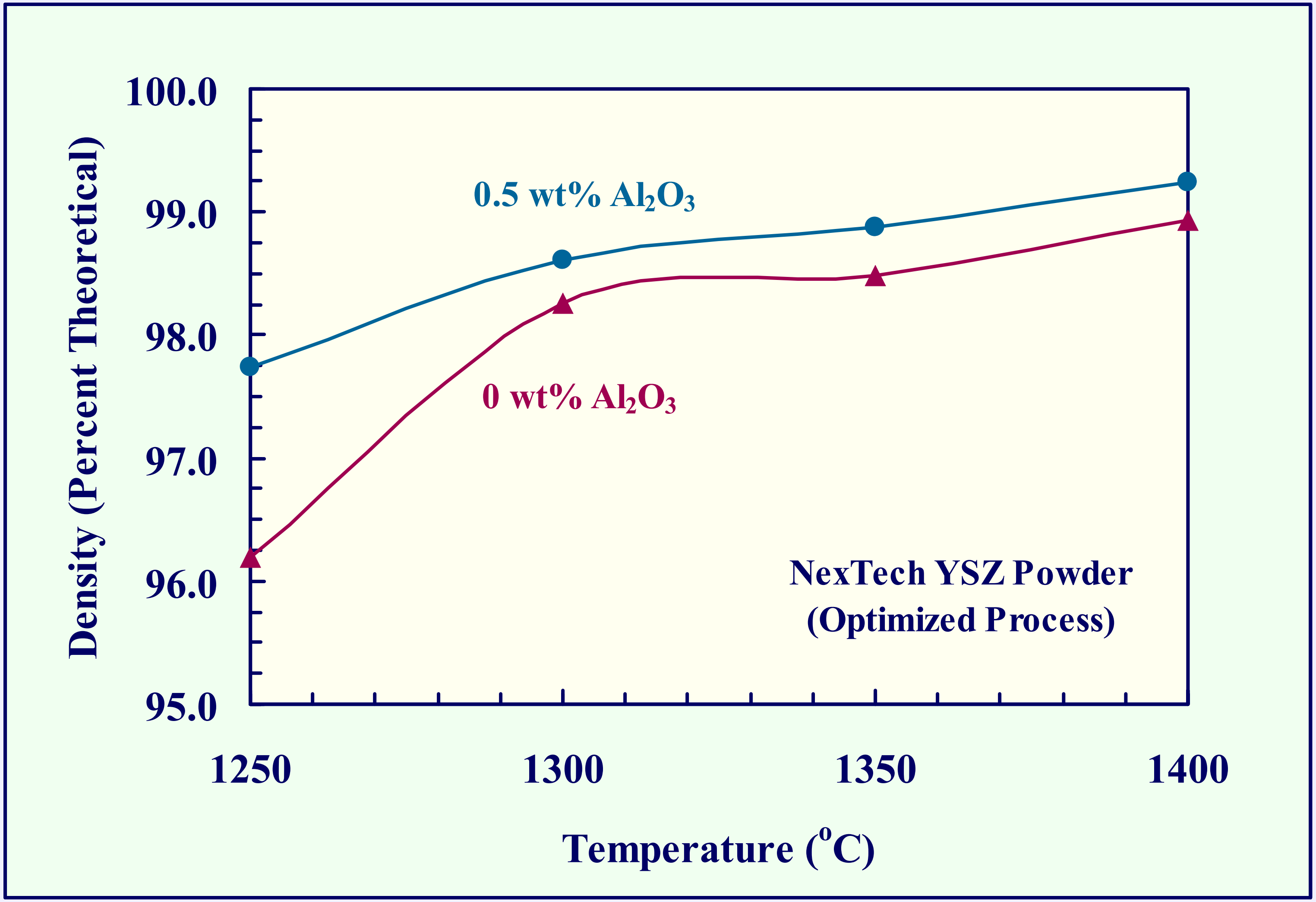
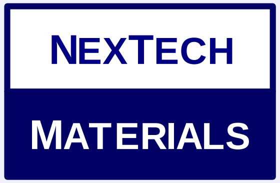
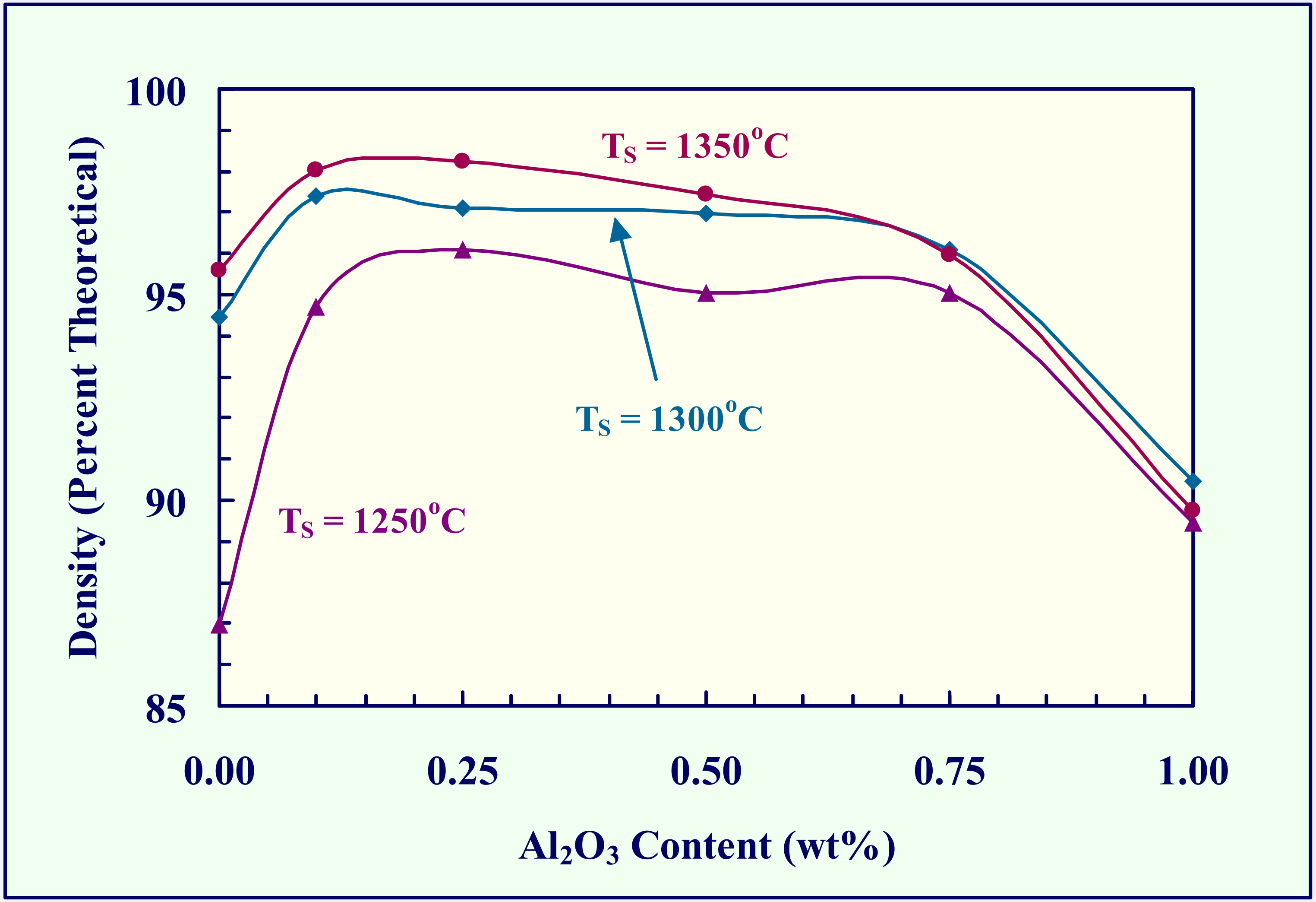
Sintering: Effect of Al_2O_3

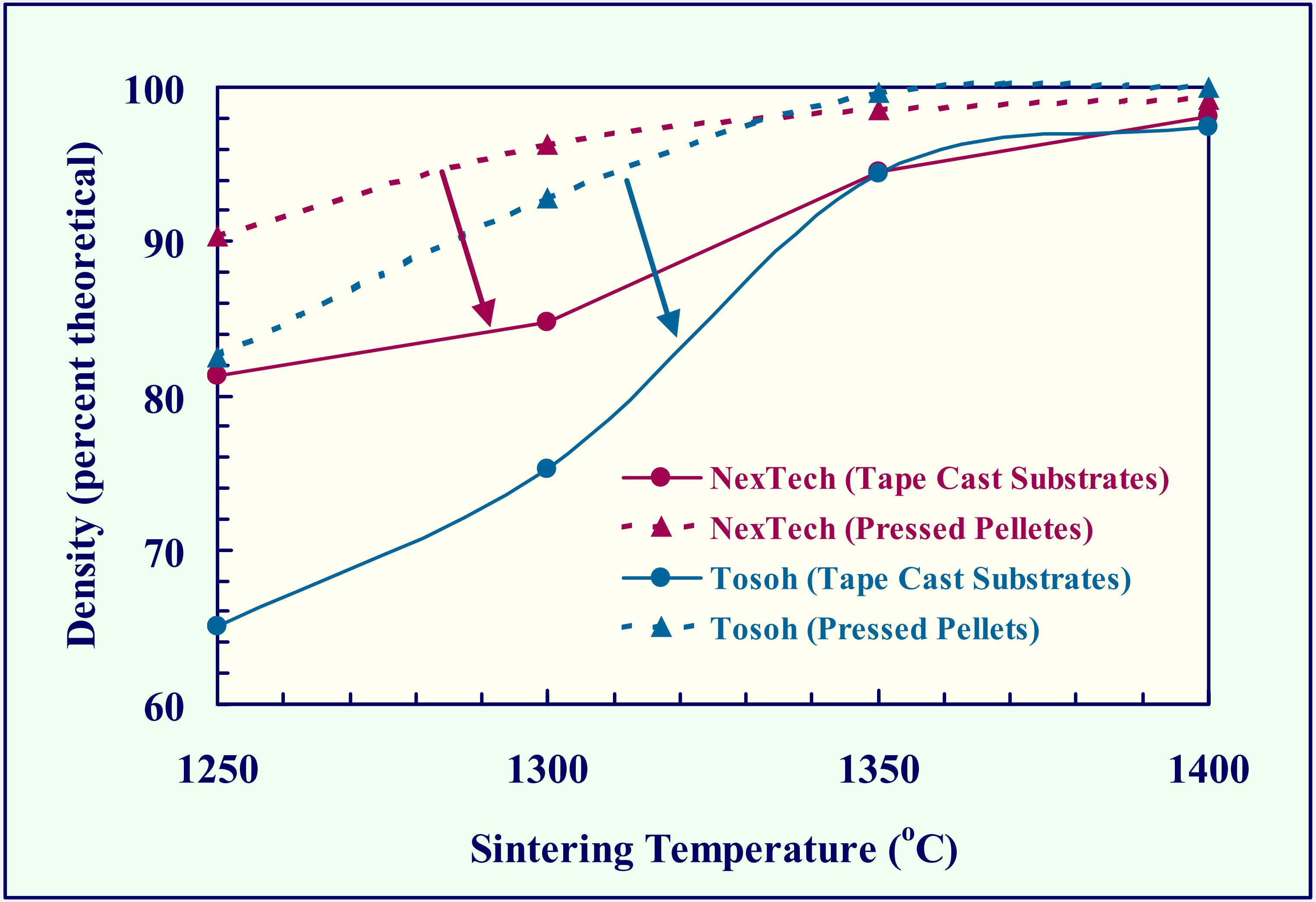


Sintering: Effect of Al_2O_3 Content





Sintering: Effect of Fabrication



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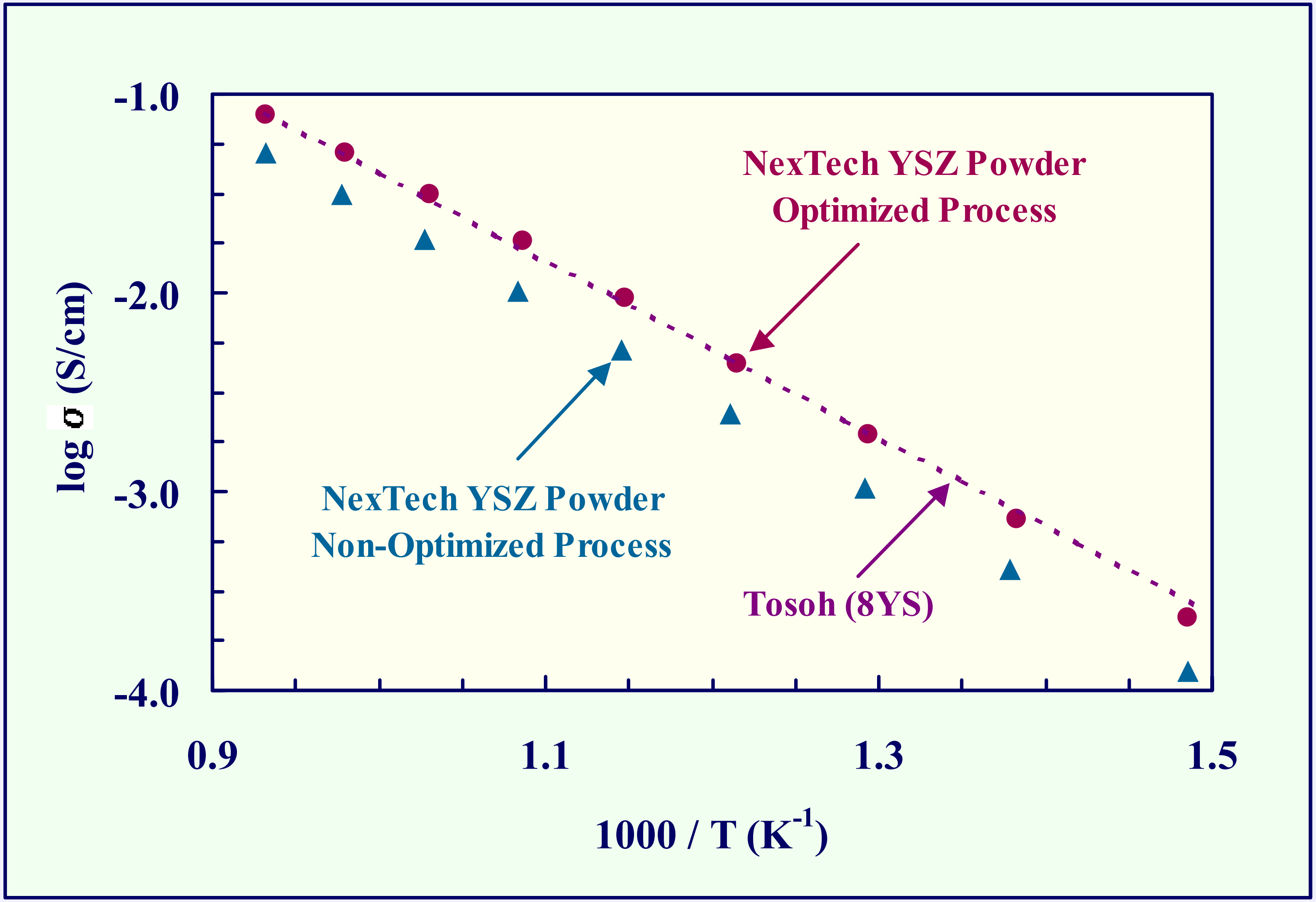
Conductivity: Effect of Synthesis



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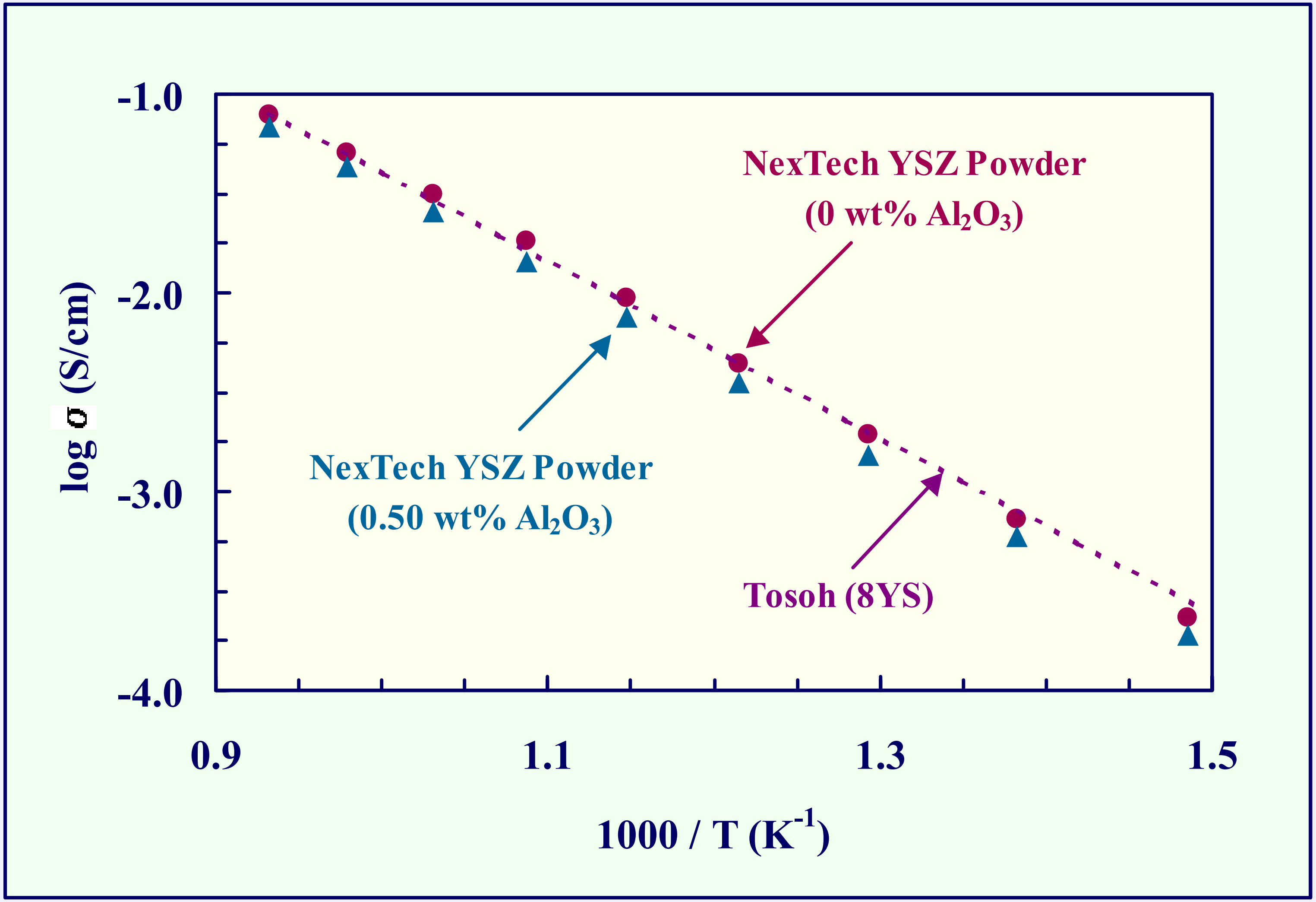
Conductivity: Effect of Al_2O_3



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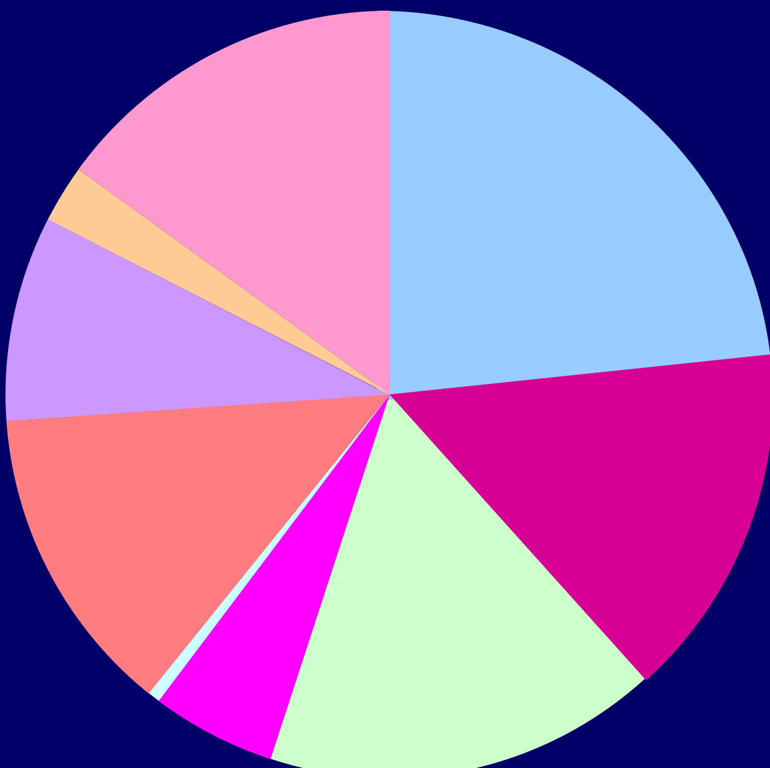
Manufacturing Cost Estimate

- **Basis of Calculations:**
  - **Plant size: 500 MT/year**
  - **Fixed capital investment: $11.2 M**
  - **Cost per kilogram of YSZ: $23.56**



- **Raw Materials: $5.27**
- **Utilities : $3.37**
- **Labor: $3.75**
- **Maintenance & Repairs: $1.39**
- **Operating Supplies: $0.17**
- **Plant Overhead: $2.93**
- **Depreciation: $1.90**
- **Local Taxes and Insurance : $0.57**
- **General Expenses: $3.37**

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Applicability to SOFC Commercialization

YSZ powder must be tailored for different manufacturing processes used for anodes and electrolyte layers.

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	Slurry Coating
Fuel Cell Energy	Screen Printing	Tape Casting
Accumentrics	Colloidal Deposition	Extrusion

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Applicability (continued)

Agile processing will allow tailoring to requirements of SOFC fabrication methods and different developers.

- **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 powder morphology are required for optimum flow characteristics.**
- **Extrusion:** **Lower surface areas needed for dimensional control and green strength; particle size requirements vary by developer.**

Batch-to-batch reproducibility is essential for all processes!

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Phase II Work (Year 1)

- Survey of SECA Industry Teams
- Process Development and Scale-Up
  - Process refinements (especially washing and drying steps)
  - Chemical analyses through all processing steps
  - Scale-up to 10-20 kg batch sizes
  - Evaluation of batch-to-batch reproducibility
  - Electrical and mechanical property testing
- Validation of Alumina Doping Strategies
  - Evaluation of dopant incorporation methods
  - Chemical analyses
  - Comprehensive microstructural analyses
  - Electrical and mechanical property testing
  - Long-term testing

Phase II Work (Year 2)

- **Demonstrations in SOFC Fabrication Processes**
  - **Preparation of composite (NiO/YSZ) anode powders**
  - **Tape casting of anode substrates**
  - **Co-sintering of anode-supported cells**
  - **Screen-printed anode coatings**
  - ***Special Requests***
- **Production of Evaluation Samples**
  - **YSZ electrolyte powder**
  - **NiO/YSZ anode powder**
  - **Fabricated components**
- **Manufacturing Cost Analyses**

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FY'04 Milestones

- Q1 Milestones (December 31, 2003)
  - Final list of specifications
  - Refined list of validation experiments
  - Plan for producing evaluation samples
- Q2 Milestones (March 31, 2004)
  - Define baseline YSZ precipitation process
  - Specify alumina dopant amount and incorporation method
- Q3 Milestones (June 30, 2004)
  - Achieve YSZ performance metrics in 3-5 kg batches
  - Demonstrate long-term stability of Al_2O_3-doped YSZ
- Q4 Milestones (September 30, 2004)
  - Achieve YSZ performance metrics in 10-20 kg batches
  - Demonstrate specific advantages for SOFC fabrication

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Acknowledgments

Ribbon Cutting (September 10, 2003)