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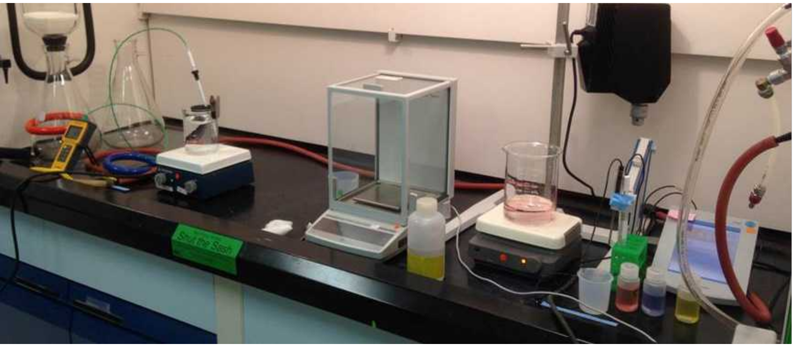

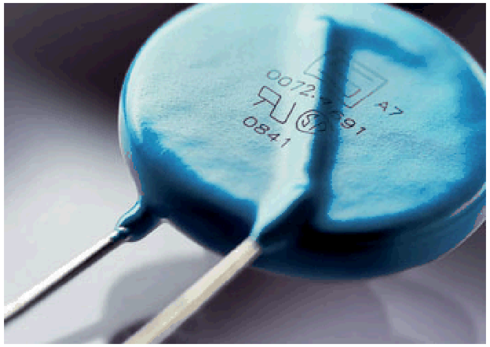

# Two-Stage Synthesis and Processing of High-Field ZnO Varistors


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# Introduction

**What is a Varistor?**

- Variable-Resistor
  - Dense ceramic with variable resistance dependent on applied voltage
- Applications
  - Voltage regulation and suppression
  - Powerlines, surge protectors, etc.

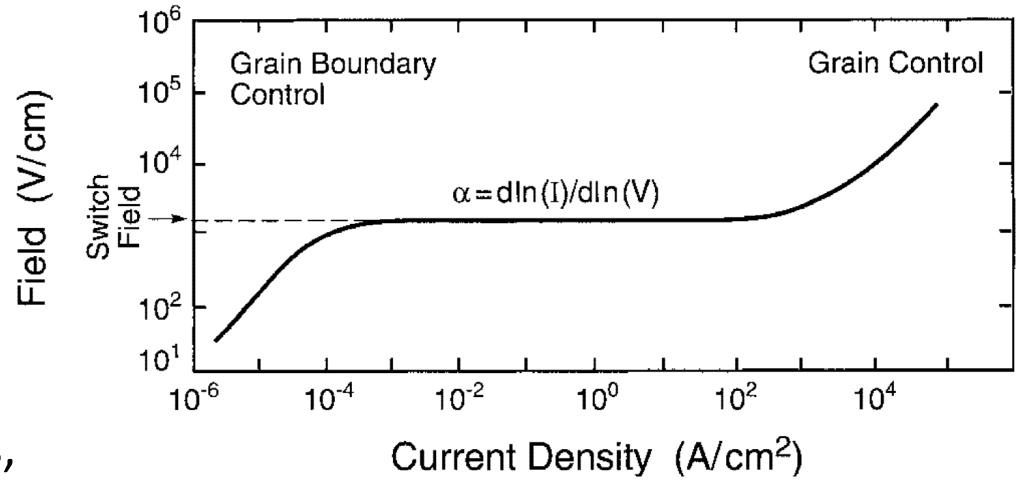


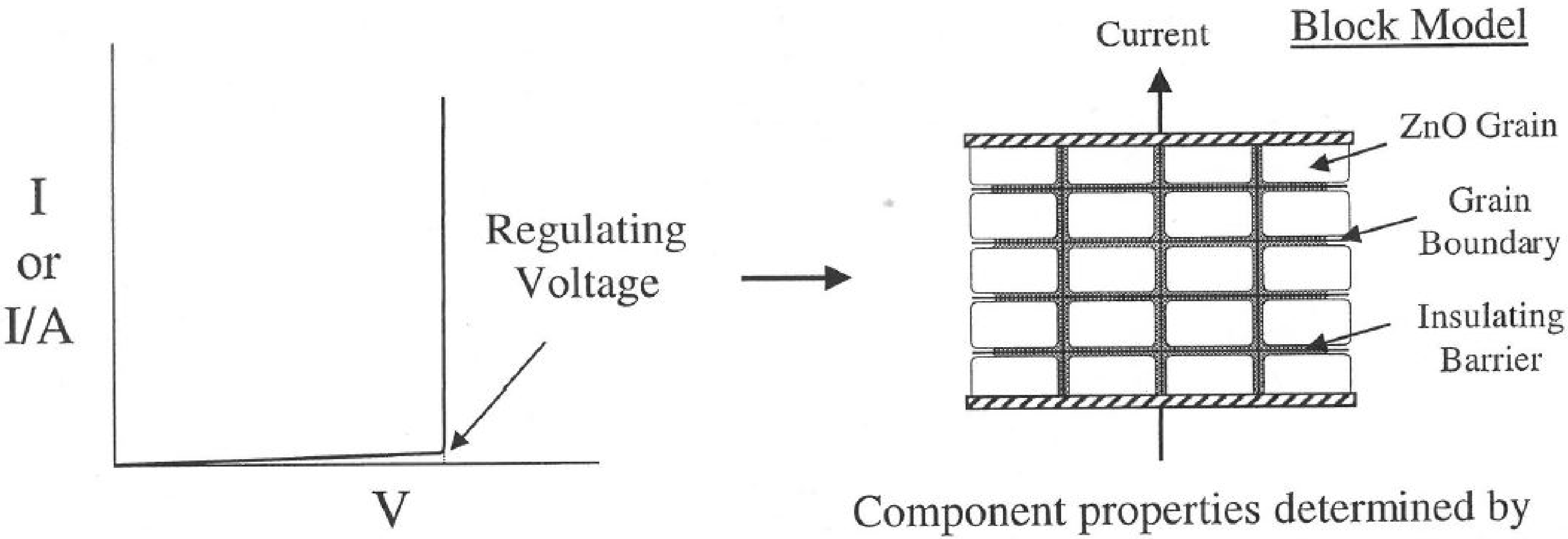

Image: Clarke, D. R. *Journal of the American Ceramics Society*, (1999) **82** (3), 485-500.

# Introduction

**What Affects a Varistor?**

- Preparation methods
  - Mixed oxide vs. chemical synthesis
- Physical and electrical properties
  - Microstructure (e.g., grain growth and size)
  - Composition (including chemical gradients)

# Introduction




Component properties determined by microstructure/composition of ZnO-based ceramic

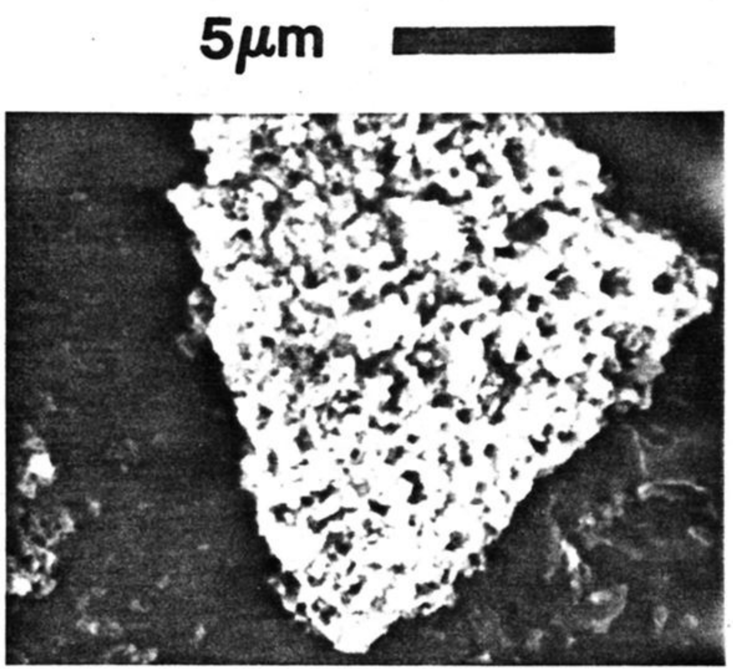
Image: Voigt, J.A. Unpublished work.

# Hydrous Oxide Precipitation

## Scenario 1: NaOH

- Co-precipitates metal salts to hydrous oxides
  - Exothermic reaction
- Particles
  - Electrical properties comparable to baseline
  - Large size complicates physical processing
  - Multiple morphologies exist
  - Lower pressed densities

## Hydrous Oxide Morphology



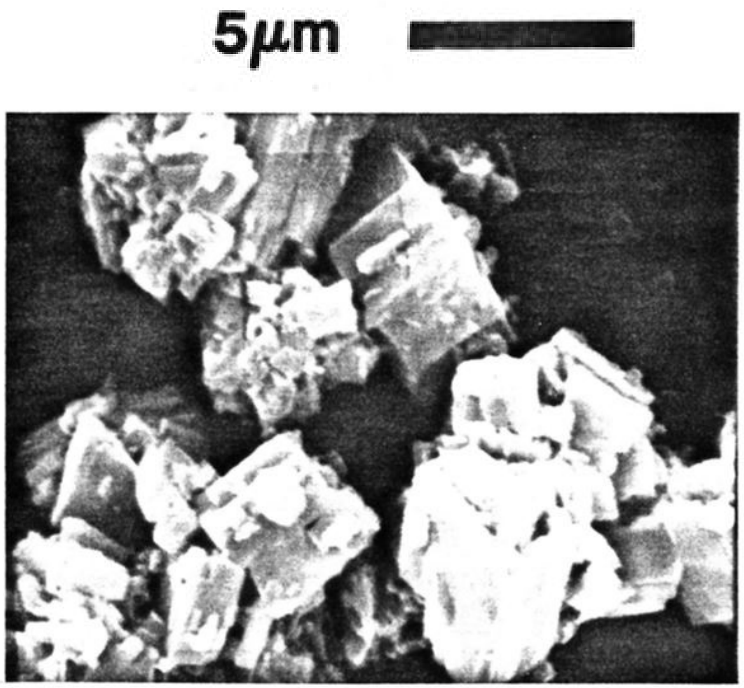

SEM Image: Voigt, J.A. Unpublished work.

# Oxalate Precipitation

**Scenario 2: Oxalic Acid**

- Co-precipitates metal salts to oxalates
  - Exothermic reaction
- Particles
  - Smaller size streamlines washing, filtering, etc.
  - Reproducible processing
  - Electrical and sintering results poor

**Oxalate Morphology**



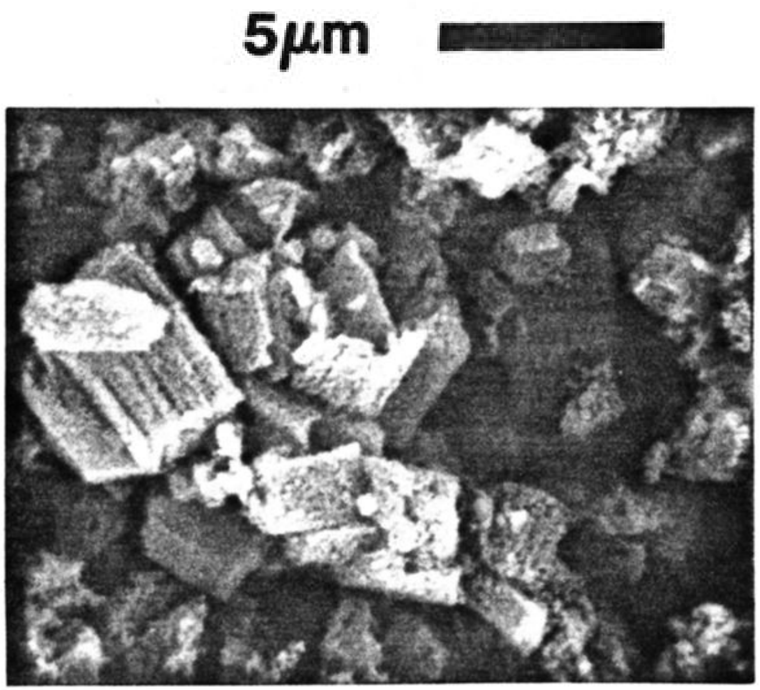

SEM Image: Voigt, J.A. Unpublished work.

# Combined Precipitation

**Problem Solving**

- Solution
  - Combine hydrous oxide and oxalate precipitations
- Precipitation dissolution re-precipitation synthesis
  - Best handling, electrical, and physical properties

**Calcined ZnO Morphology**




SEM Image: Voigt, J.A. Unpublished work.

# Procedure Overview

## Major Steps

- Precursor co-precipitation of metal salts
  - Washing and filtration
  - Initial calcination
- Powder processing
  - Successive doping and calcination stages
  - Pressing and sintering
  - Sputter and electrode for testing

## Process Flowchart

# Two-Stage Chemical Synthesis

**Stage 1: NaOH Addition**

**Stage 2: Oxalic Addition**

# Two-Stage Chemical Synthesis

**Chemistry**

- Combined precipitation-dissolution-reprecipitation synthesis
- Supersaturation of metal salts drives reaction kinetics
  - Oxalate particle formation occurs at site of hydrous oxide dissolution
  - Yields homogenous powder with positive sintering results

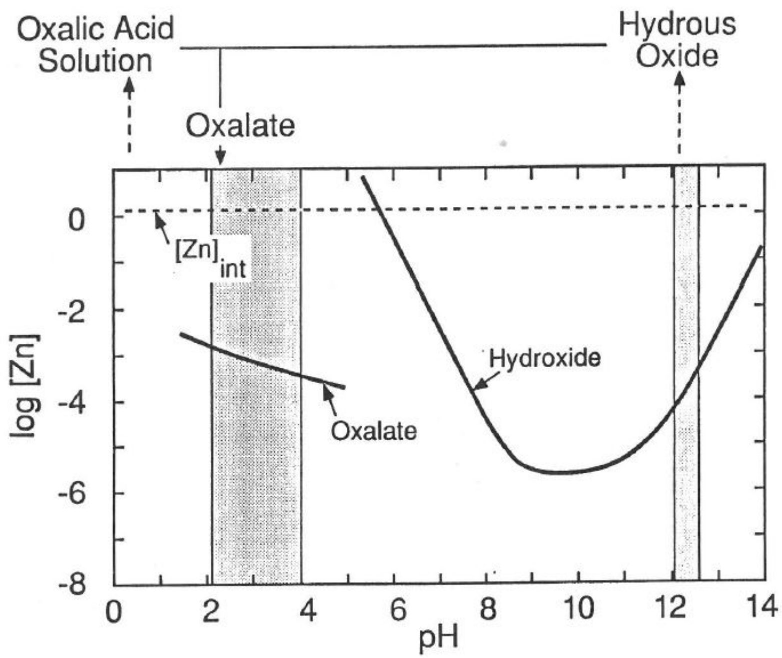



Image: Voigt, J.A. Unpublished work.

# Powder Processing

**Washing and Filtering**

- Washing and filtering
  - Water and solvent rinses
  - Removes excess waste that may be in the filter cake (Chlorides, etc.)
  - Improves performance of final varistor

**Filter Cake**

# Powder Processing

## Calcination

- Thermal profiles
  - Burn off carbon and impurities in first calcine
  - Converts zinc oxalate into zinc oxide

## Calcined ZnO Powder

# Powder Processing

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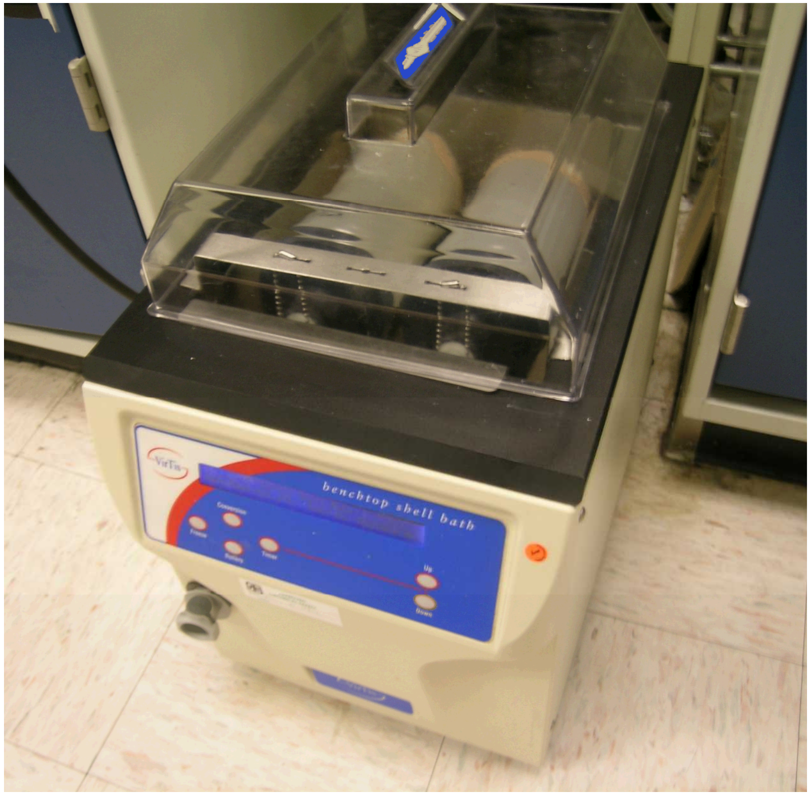
## Doping and Drying

- Doping stages
  - Bismuth metal added to aid with matrix stability
  - Additional doping stages can be added as desired
- Drying process
  - Spray-dry, freeze-dry, etc.
  - Ensures even dopant coating
  - Prevents formation of hard aggregates

## Lyophilizing Powders



# Powder Processing

### Pressing and Sintering

- Optimal target of over 90% densification
  - Combined uniaxial and isostatic pressing
- Fired samples ready for electrode sputtering and electrical testing
  - Grinding/polishing may be necessary to remove defects and surface coating

### Electroded Samples

# Summary

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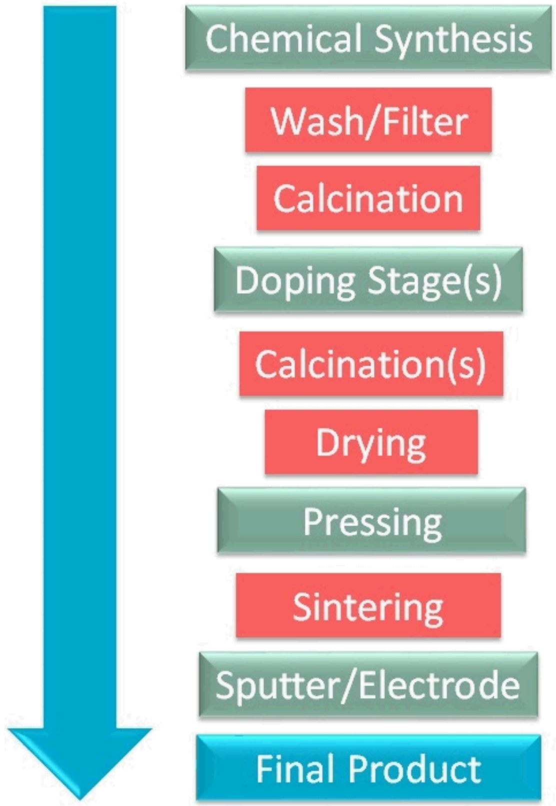
### Take-Home Message

- Chemical-preparation synthesis
  - Create a varistor from start to finish
  - Control composition and microstructure
- "Intelligent chemical preparation of powders allows us to control complex materials properties in advanced ceramics."

### Process Flowchart



# Acknowledgments

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  - Varistor IV figure (Slide 2)

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Thank you!


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# Questions?