## NIST Particle Characterization Laboratories

#### Vince Hackley Ceramic Manufacturing Program, MSEL



## **Ceramic Materials**

#### *Ceramics* : non-metallic inorganic materials

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Structural Electronic Refractory Bioceramic Structural Clay White Wares Cement



## Ceramic Manufacturing

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Ceramic manufacturing can involve a number of complex processes, each of which is impacted by the previous step.

Raw Materials					Finished Products	
Preparation of Starting Powder	Powder Processing	Shape Forming	Green Machining	Densification	Machining	
Comminution Particle Sizing Refining Filtration Chem. Synthesis Calcination Classification	Binders Additives Mixing/Milling Homogenization Classification Spray Drying	Dry Pressing Plastic Forming Slip Casting Tape Casting Extrusion Injection Molding	Drilling Cutting	Pressureless Hot Iso. Pressi Reaction Sinter Infiltration	s Grinding ing Lapping ring Polishing Laser Drilling EDM Ultrasonic	

## Particulates in Ceramic Processing

- The vast majority of ceramics are manufactured starting from powders
- Wet processing (dispersion) is widely used in industry
- Particle characteristics have a profound impact on the properties of consolidated ceramics

## **Particulate Properties**

Particle characterization is a key enabler for reliable cost-effective manufacture of advanced ceramics

#### Physical Properties

• <u>Density</u>

- <u>Porosity</u>
- <u>Surface Area</u>
- <u>Shape</u>
- Size Distribution

#### **Dispersion Properties**

- <u>Surface Charge</u>
- <u>Zeta Potential</u>
- <u>Surface Chemistry</u>
- <u>Rheology</u>
- <u>Agglomeration</u>

## Ultrasonic Measurement Methods

The most important feature of ultrasonics is its capacity to provide useful information on concentrated suspensions

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#### **Advantages**

- concentrated systems
- non-destructive
- robust sensors
- during flow
- rapid

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high precision

#### **Properties**

• particle size distribution

- zeta potential
- solids concentration

## Zeta Potential



## Measuring Zeta Potential

Zeta is typically measured using electrokinetic methods

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In an electric field, charged particles will move with a velocity dependant on Zeta and the applied field



## Electroacoustic Measurements

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## Based on the coupling between electric and acoustic fields in a suspension of charged particles



### **Cement Hydration**

#### CVP Measurements Provide Information on Cement Particle Chemistry

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Relative Acoustic Mobility (RAM) of Portland cement as a function of hydration time and solids loading.

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Reflects physical and chemical changes that impact the cement particles.

Flatt & Ferraris (unpublished)



## Conclusion

#### The Ceramic Manufacturing Program at NIST has:

- world class laboratories for the characterization of fine particles
  - expertise in working with inorganic particulates
  - unique ultrasonics facility for characterizing concentrated suspensions

## Available Instrumentation

• AMRAY 1830 SEM

- Beckman J2-HC Centrifuge
- Brookfield DVII+ Viscometer
- Colloidal Dynamics Acoustosizer
- Coulter LS230 Laser Diffraction
- Coulter Multisizer II Zone Sensing
- Dispersion Technology DT1200
  Acoustic Spectrometer
- Horiba LA900 Laser Diffraction
- Micromeritics Sedigraph 5100
- Micromeritics AccuPyc 1330

- Malvern Zetasizer 3000HS Doppler Laser Scattering
- Matec ESA8000 Electroacoustic Analyzer
- Perkin-Elmer 330 UV-VIS Spectrophotometer
- Quantachrome AUTOSORB-1
  Multipoint Gas Adsorption
- Quantachrome AUTOSORB-60
  Mercury Porosimeter
- Rheometrics RS2000 Rheometer
- Rosemount-Dohrmann DC80 TOC

## Particle Shape Measurement

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 Scanning Electron Microscopy/Auto-Digitization

#### **AMRAY 1830**



## **Rheological Measurements**

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• Shear Viscosity

- Storage Modulus
- Shear Modulus
- Yield Stress

#### Rheometrics RS-2000



## Surface Charge Measurement

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# • Potentiometric Titration

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#### Matec ESA-8000



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## Agglomeration Measurements



- Electroacoustic Spectrometry
- Acoustic Attenuation
  Spectrometry

#### Colloidal Dynamics Acoustosizer



## Surface Chemistry Measurements

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Polymer Adsorption

- Isoelectric Point
- Potentiometric Titrations

#### Techmar-Dohrman DC-80 TOC



## Zeta Potential Measurement

- Doppler Microelectrophoresis
- Colloid Vibration
  Current (CVI)

• Electrokinetic Sonic Amplitude (ESA)

#### Malvern Zetasizer 3000HS



## Surface Area & Porosity Measurement

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- N<sub>2</sub> Gas Adsorption
- Hg Porosimetry

#### Quantachrome AUTOSCAN 60



## Particle Size Distribution Measurement

- Scanning Electron Microscopy (SEM)
- Doppler Laser Scattering (DLS)
- Laser Diffraction
- X-Ray Sedimentation
- Centrifugal Sedimentation
- Electrical Zone Sensing

#### Micromeretics Sedigraph 5100



## Density Measurement

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## • He-Pycnometry

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#### Micromeritics AccuPyc 1330



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