

# Improved Microstructure of Silver Acetylide-Silver Nitrate Explosive and Subsequent Effects to Detonation Performance

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Preliminary PhD Research Proposal

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Research Advisor: Dr. Crystal Glen (SNL)

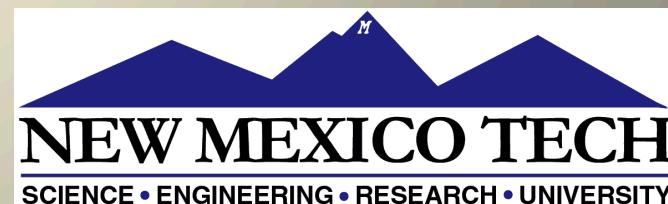
MATE592 Graduate Seminar

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# Motivation

Variability in Explosive Performance

Hypotheses:

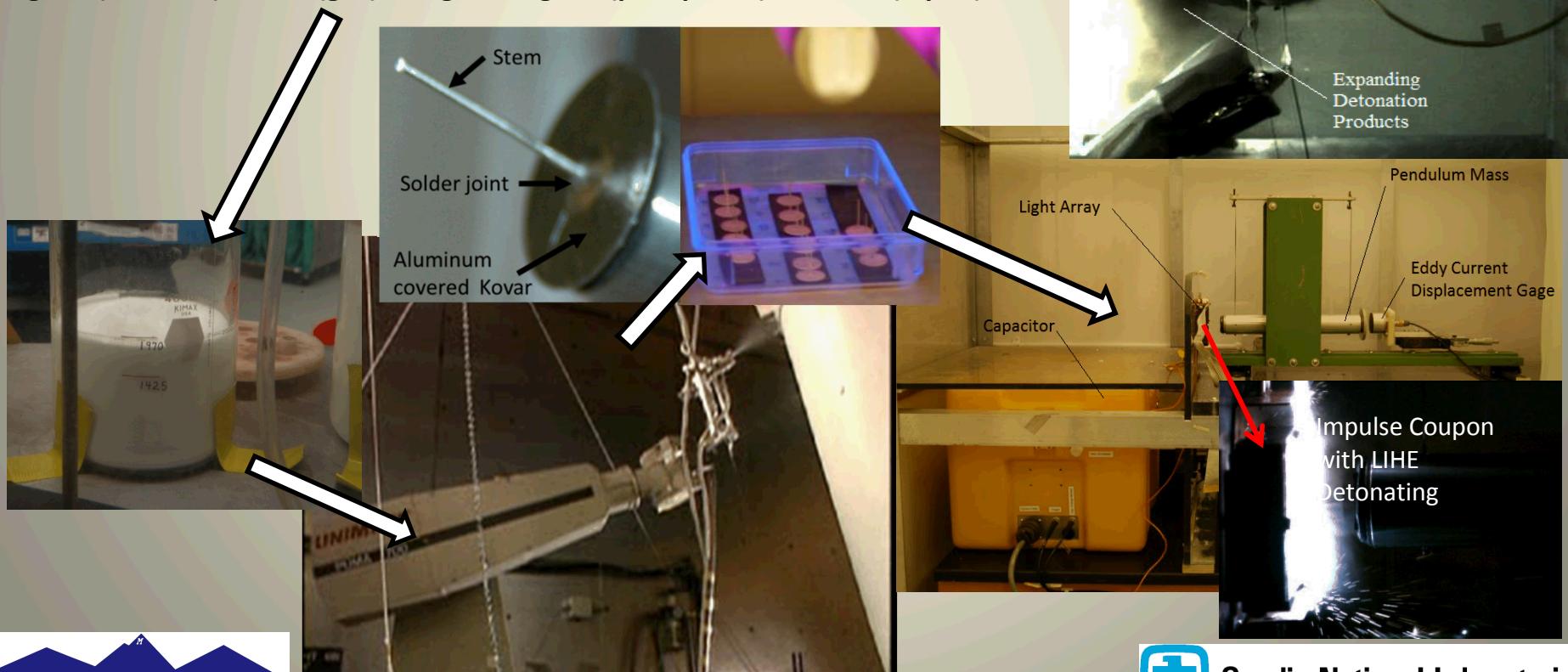
1. Energetic materials (EMs) require processing to control subsequent explosive behavior, especially during manufacturing processes
2. The explosive phenomena can be modeled by time-delayed logistic differential equations and guide engineering of EMs, but requires knowledge of physical and chemical properties

Goal:

Control explosive behavior and variation; i.e. peak pressure, impulse, deflagration-to-detonation transition, etc. and variation in all behaviors and relate to material's properties via mathematical description

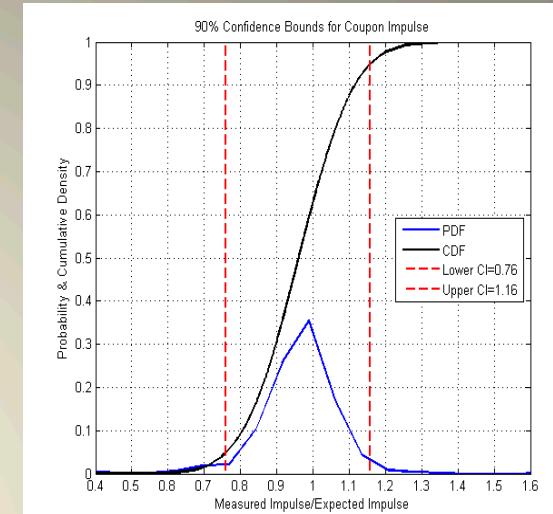
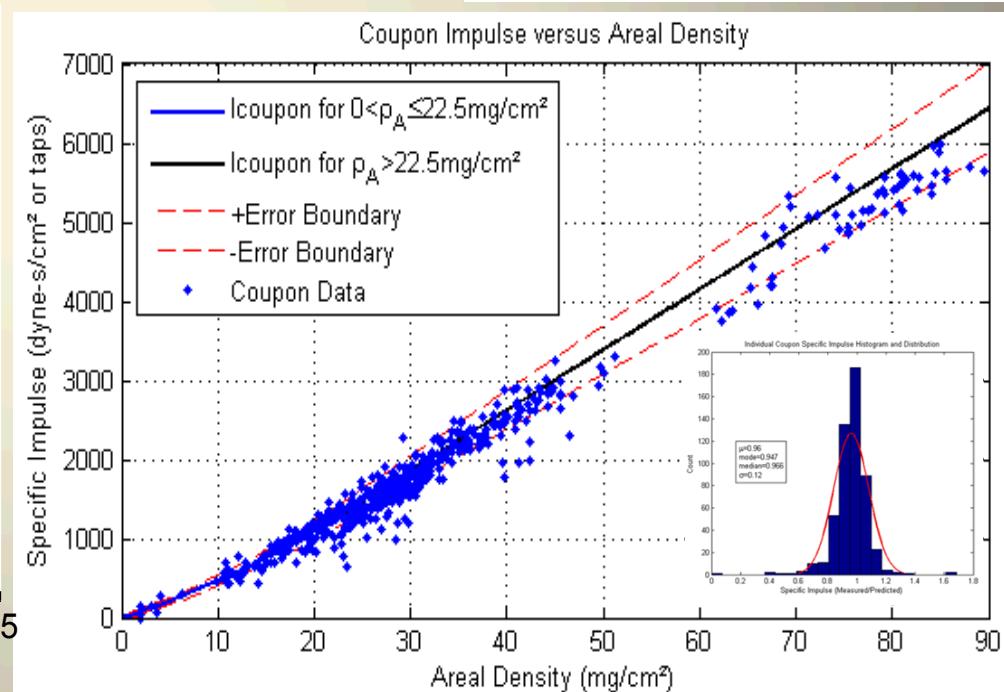
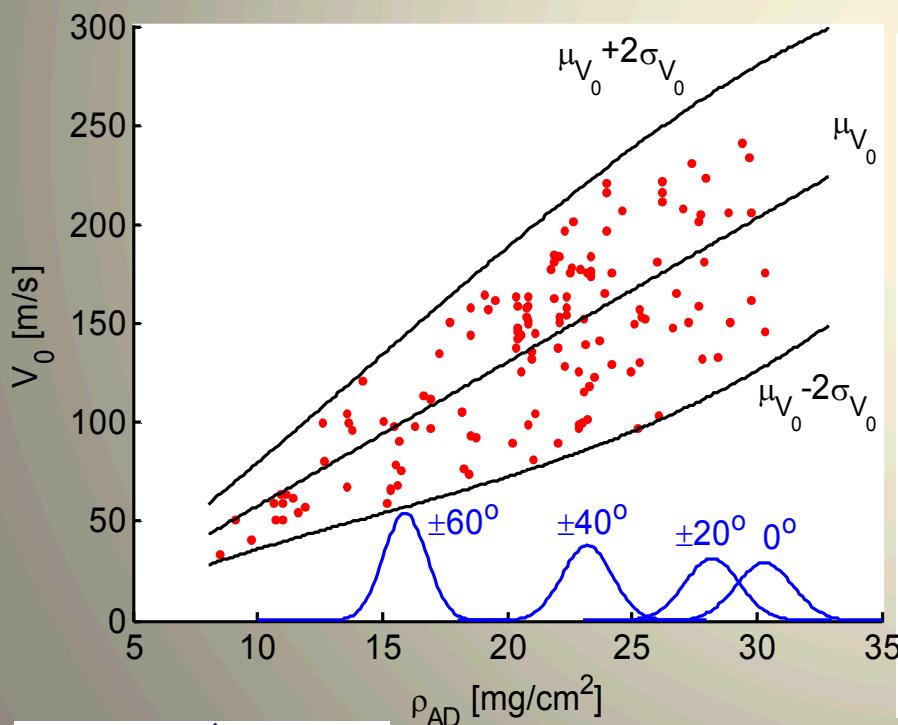
# Background: SASN

- Silver acetylide-silver nitrate (SASN)
  - Light sensitive primary explosive
  - Used for surface impulsive loading of materials & structures in unique environments



# Background:

- Unacceptable variability,  $\pm 20\text{-}50\%$ 
  - Manifests in all quantitative properties measured:
    - Areal density, thickness, & bulk density
    - Flyer plate terminal velocity & specific impulse
    - Peak pressure, pulse duration, & waveform



Ref: Radtke, G.A., Chavez, M.A., Covert, T.T., SAND2014-3450, April 2014.

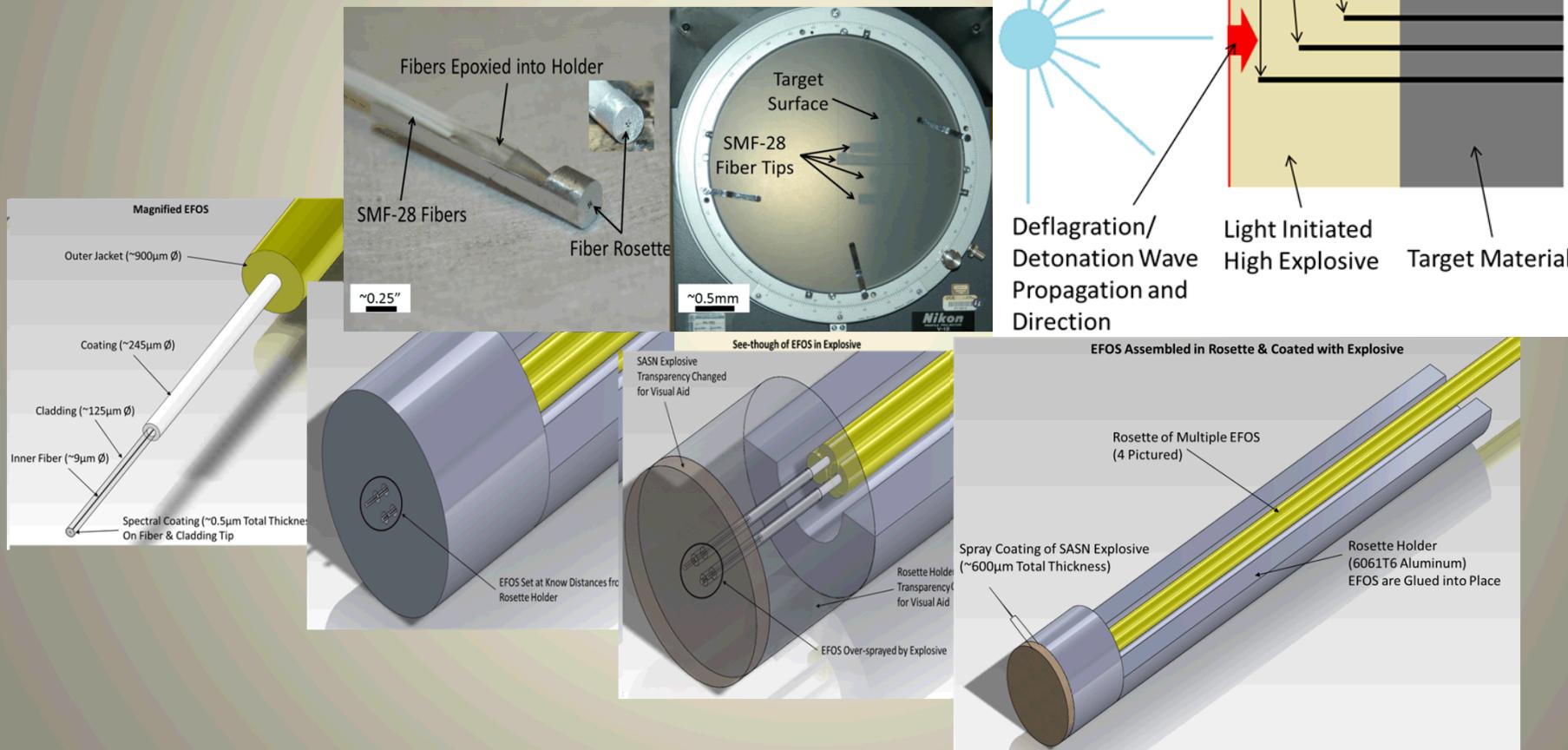
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# Past and Present Research

1. Non-uniformity deemed to be underlying issue, techniques attempted to stabilize slurry and engineer deposition
2. Detonation phenomena contributes to variation in output, techniques attempted to address phenomena
3. Impurities are present and contribute to variability
4. Sensitive primary explosive that is manufactured to produce microscopic to sub-millimeter dimensions for thickness is difficult to characterize

# Past and Present Research

- Embedded Fiber Optics (EFOS) development for deflagration/detonation velocity, data mining, & peer reviews



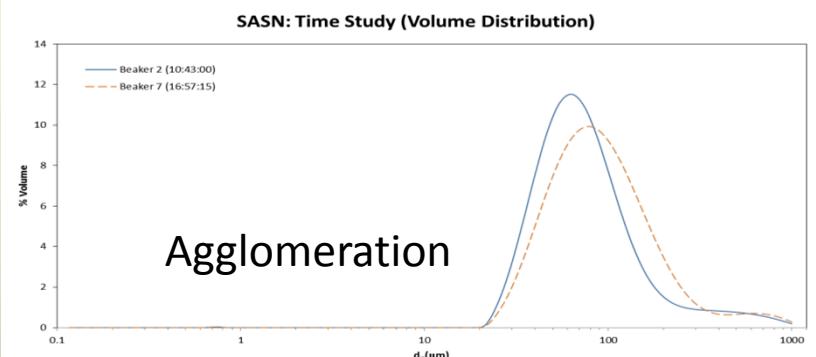
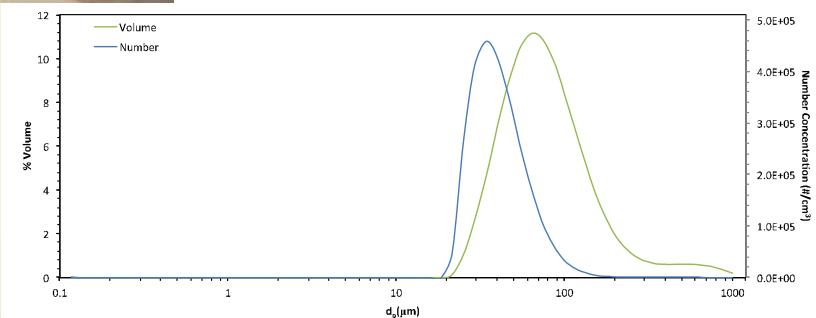
Ref: Chavez, M.A., Willis, M.D., Covert, T.T., SAND2014-17359, September 2014.

# Past and Present Research

- Collection of particle size distribution (PSD)

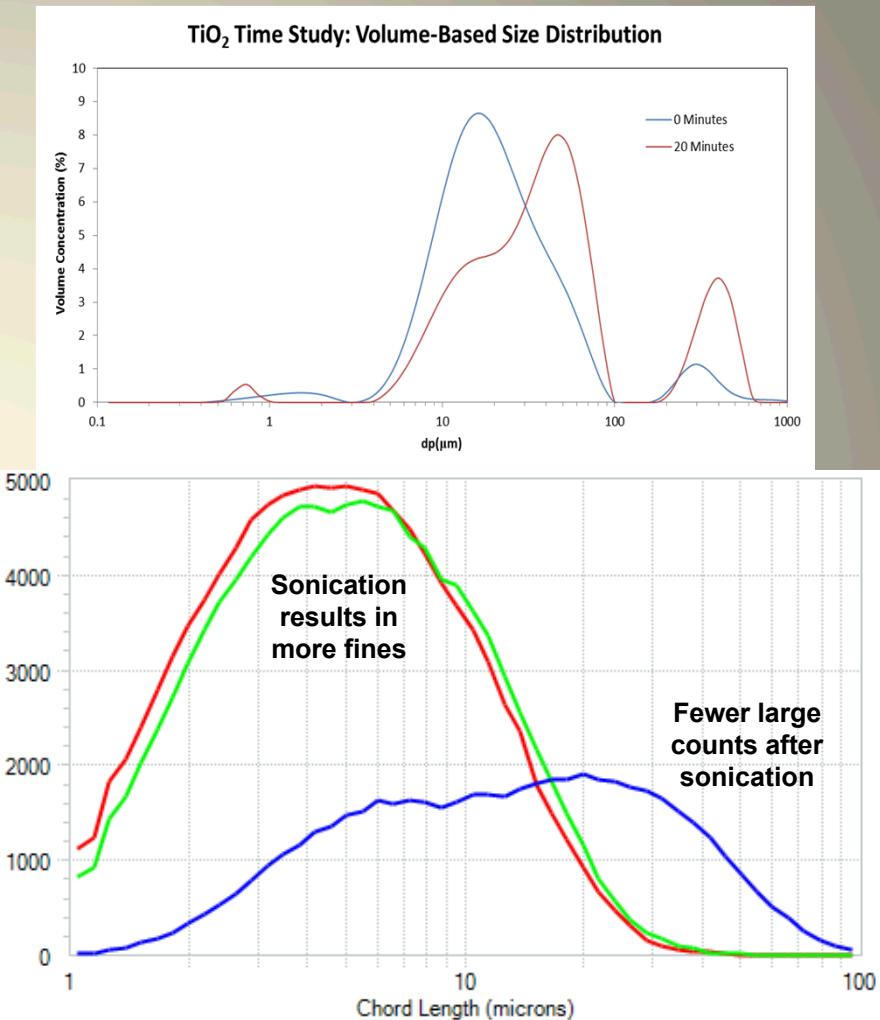
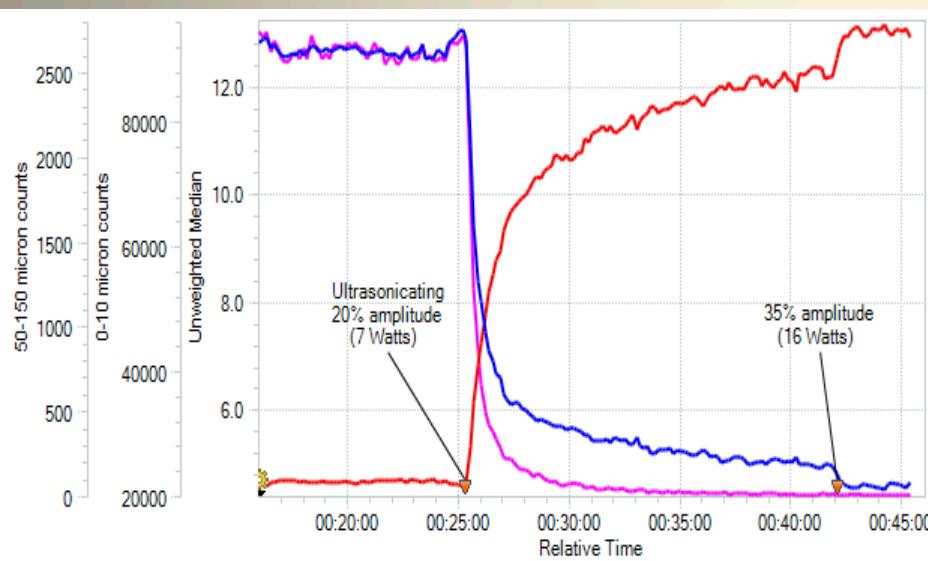


SASN: Original Sample



# Past and Present Research

- Surrogate/mock explosive search
  - $\text{MgO}$ ,  $\text{AgC}_2\text{H}_3\text{O}_2$ ,  $\text{AgNO}_3$ , &  $\text{AgCl}_2$
- $\text{TiO}_2$  is suitable surrogate
  - Application of ultrasonication



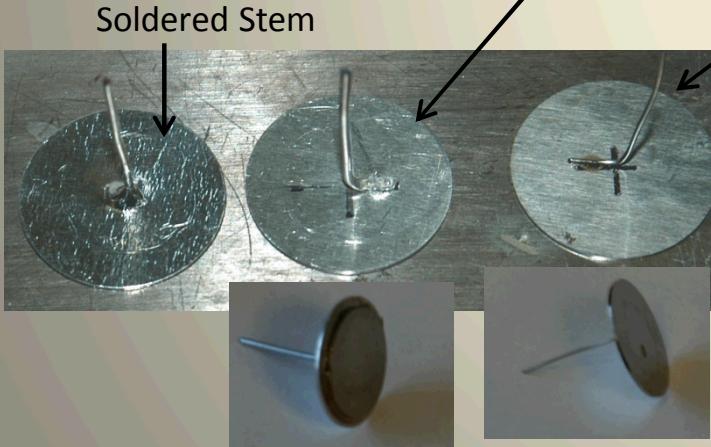
Ref: Sanchez, A., Storch, S., Tezak, M., LIHE Collaboration with Aerosol Science Team, Internal FY15 Memo, September 2015.

Seliga, C., Dycus, E., Chavez, M., Ensure Optimal and Repeatable Flyer Plate Testing by Controlling Propellant Size/Count with ParticleTrack and Particle View, Internal Presentation, Mettler-Toledo/SNL collaboration, October 2014.

# Past and Present Research

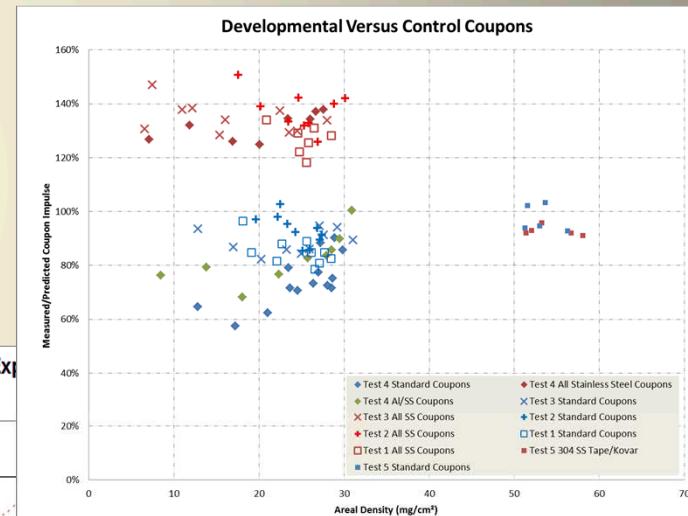
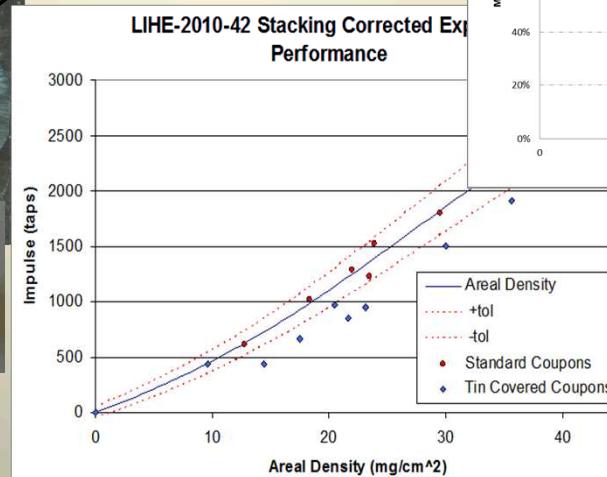
- Substrate compatibility discovery
  - 440A stainless steel (SS) shows 40-50% increase in specific impulse
  - 1100-O aluminum/440A SS backing shows similar impulse as standard coupon
  - 304 SS covered standard Kovar coupon backing shows conflicting results
- SASN found to react with Sn, Cu, Al, and Kovar

“Standard Coupon” 1100-O Aluminum Tape/Kovar Steel Backing With Sn coated Cu



1100-O  
Aluminum/440A  
SS Backing  
With Al Resistance  
Welded Stem

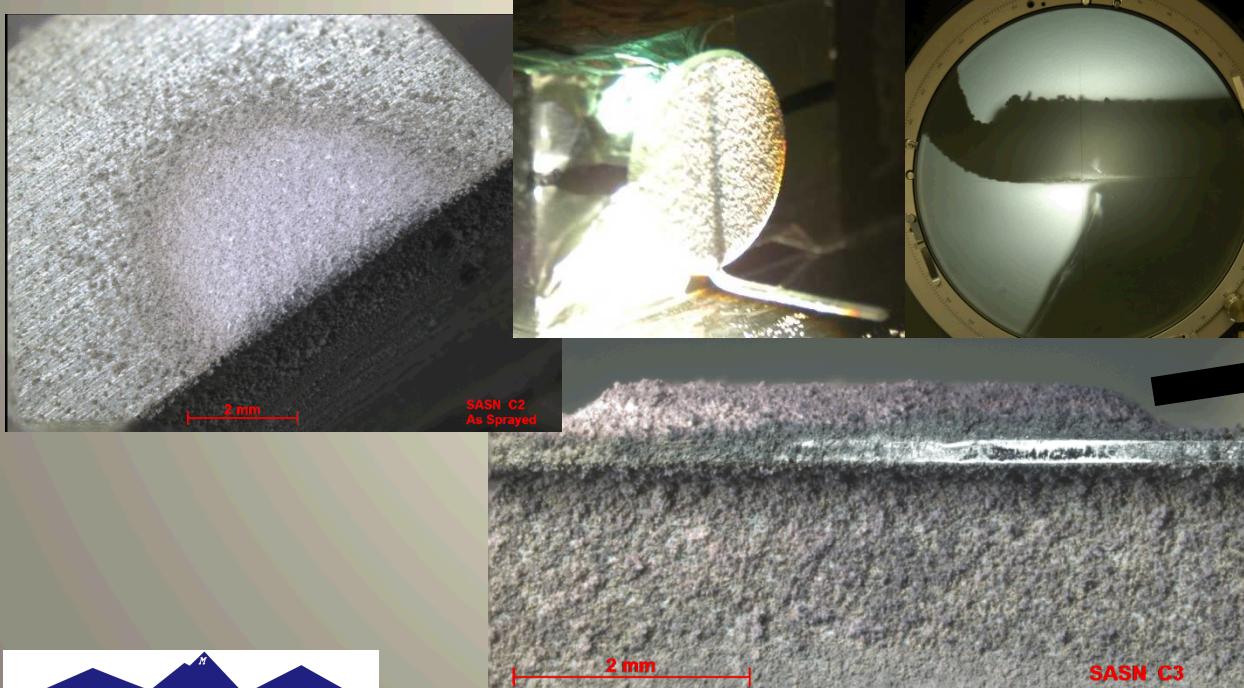
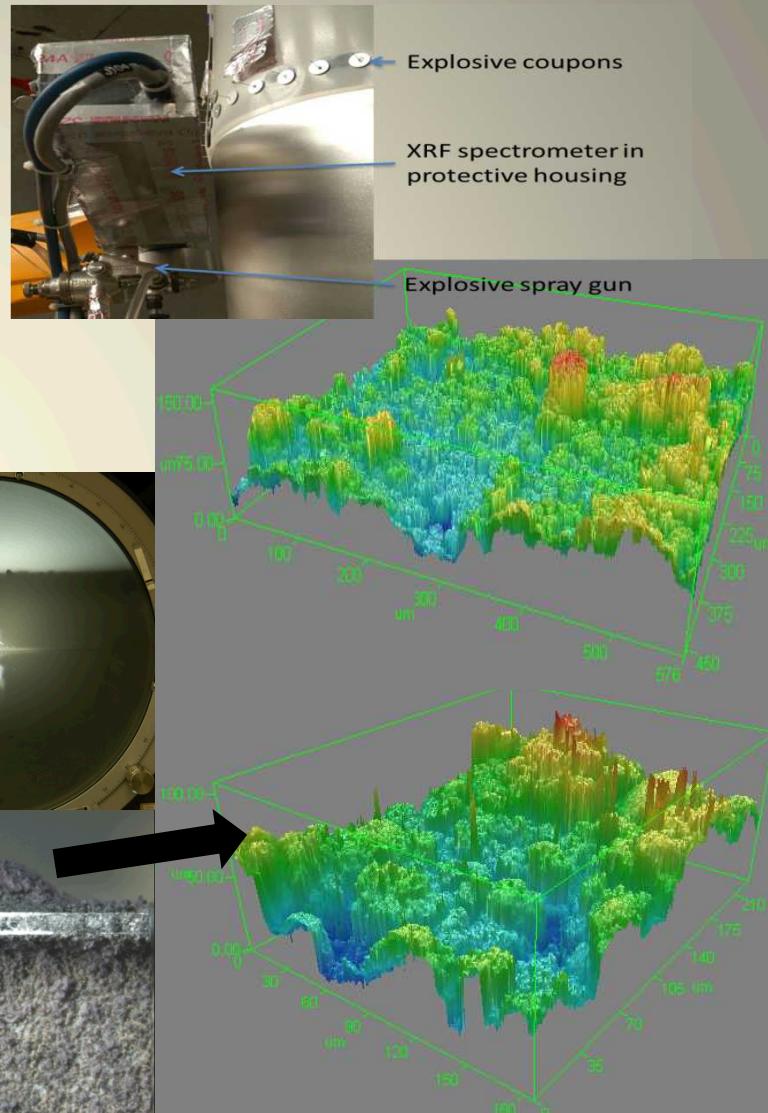
440A SS  
With 17-4PH SS  
Resistance  
Welded Stem



# Past and Present Research: Diagnostics

## Deposition Determination Techniques

1. Areal density deposition via mass balance
2. Thickness of deposition via optical comparator
3. Surface roughness via confocal microscopy
4. High resolution photographs from camera and SEM coupled with image analysis
5. X-ray fluorescence (XRF) calibrated for Ag:Sn ratio

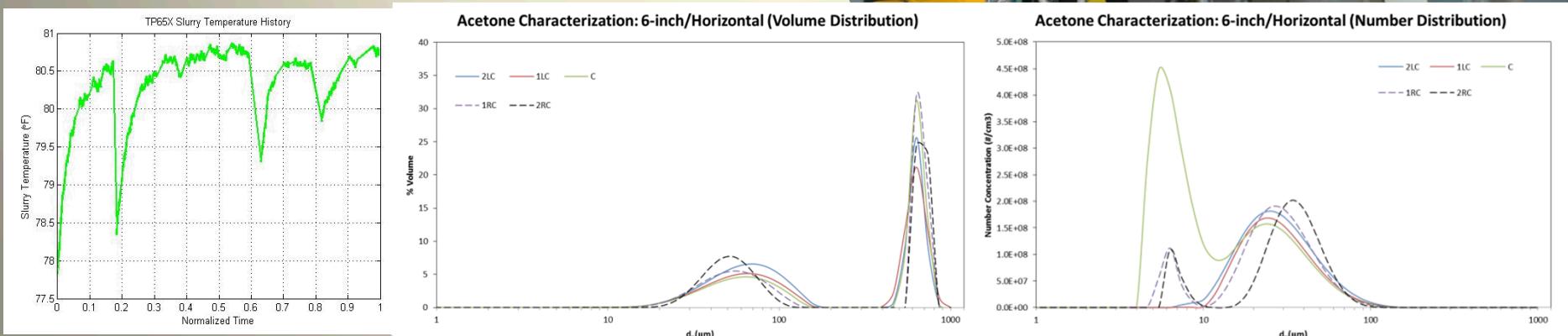
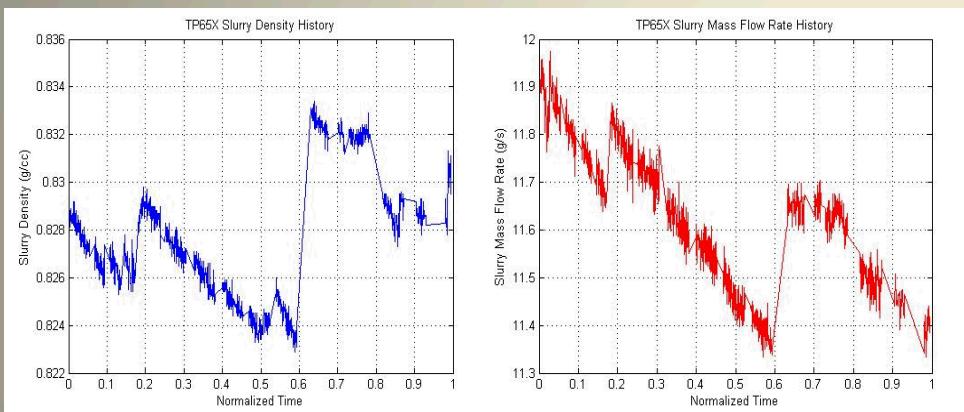


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# Past and Present Research: Diagnostics

## Slurry and Spray Characterization Techniques

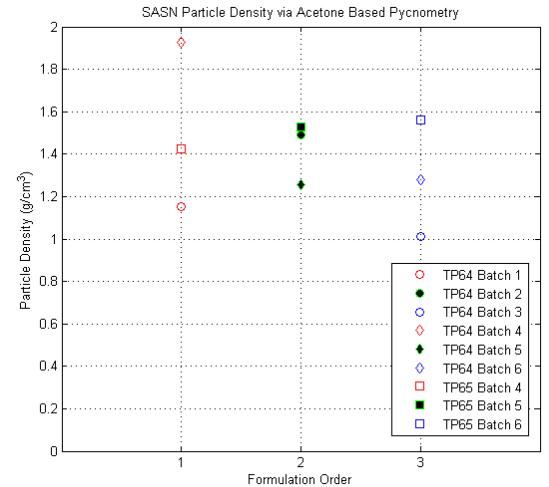
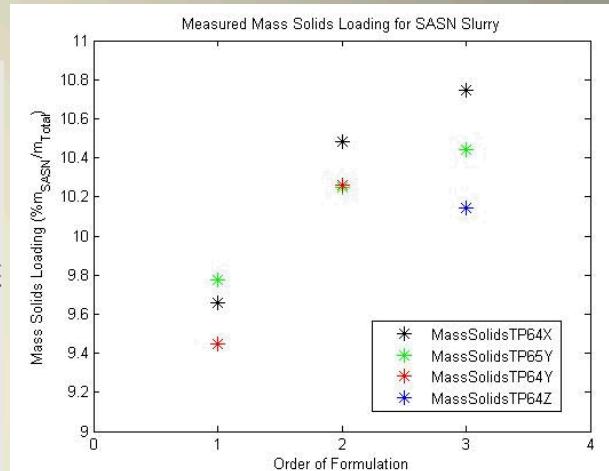
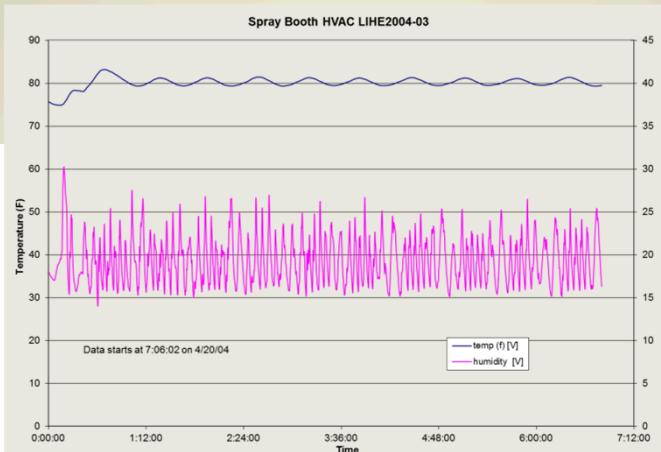
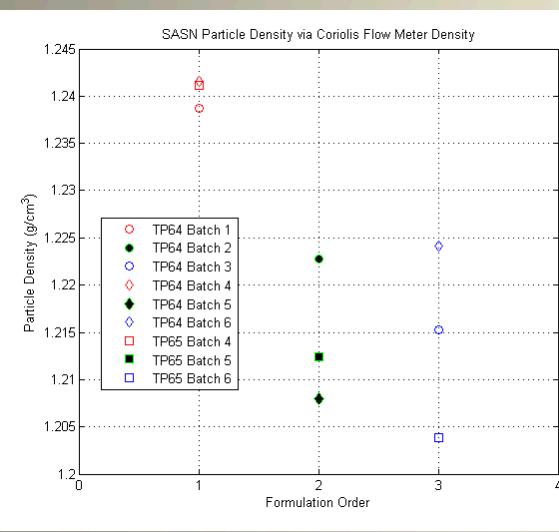
1. Simultaneous mass flow rate, temperature, and density via coriolis flow meter
2. Laser diffraction for PSD and droplet size distribution (DSD)



# Past and Present Research: Diagnostics

## Slurry and Spray Characterization Techniques

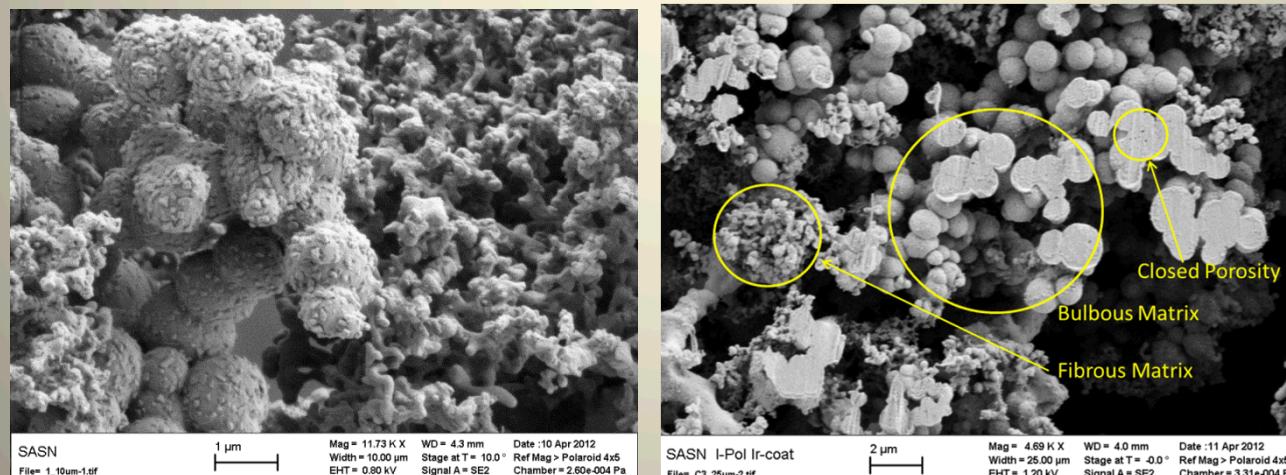
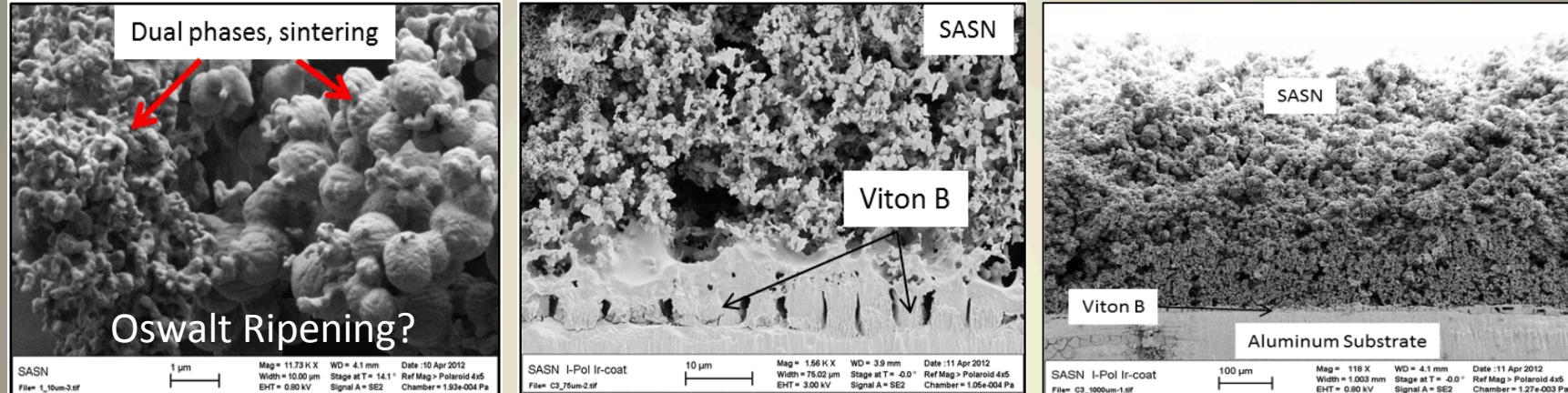
3. Mass fraction and acetone based pycnometry
4. Spray booth humidity and temperature during manufacture



# Past and Present Research: Diagnostics

## Microstructure and Molecular Signature Characterization Techniques

### 1. Scanning Electron Microscopy (SEM) with Focused Ion Beam (FIB)



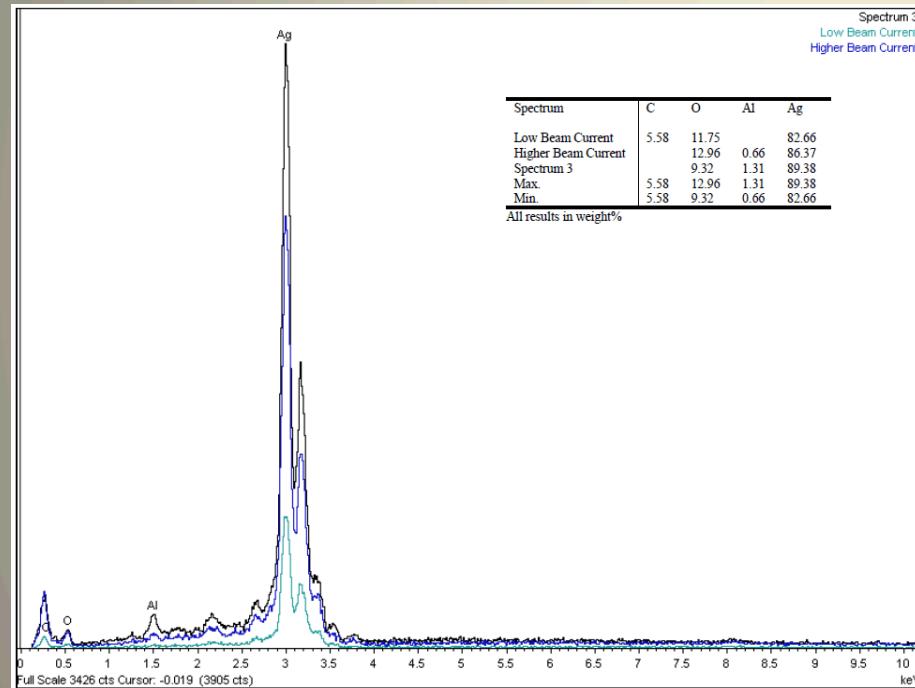
Ref: Chavez, M.A., SAND2012-3438P, April 2012.

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# Past and Present Research: Diagnostics

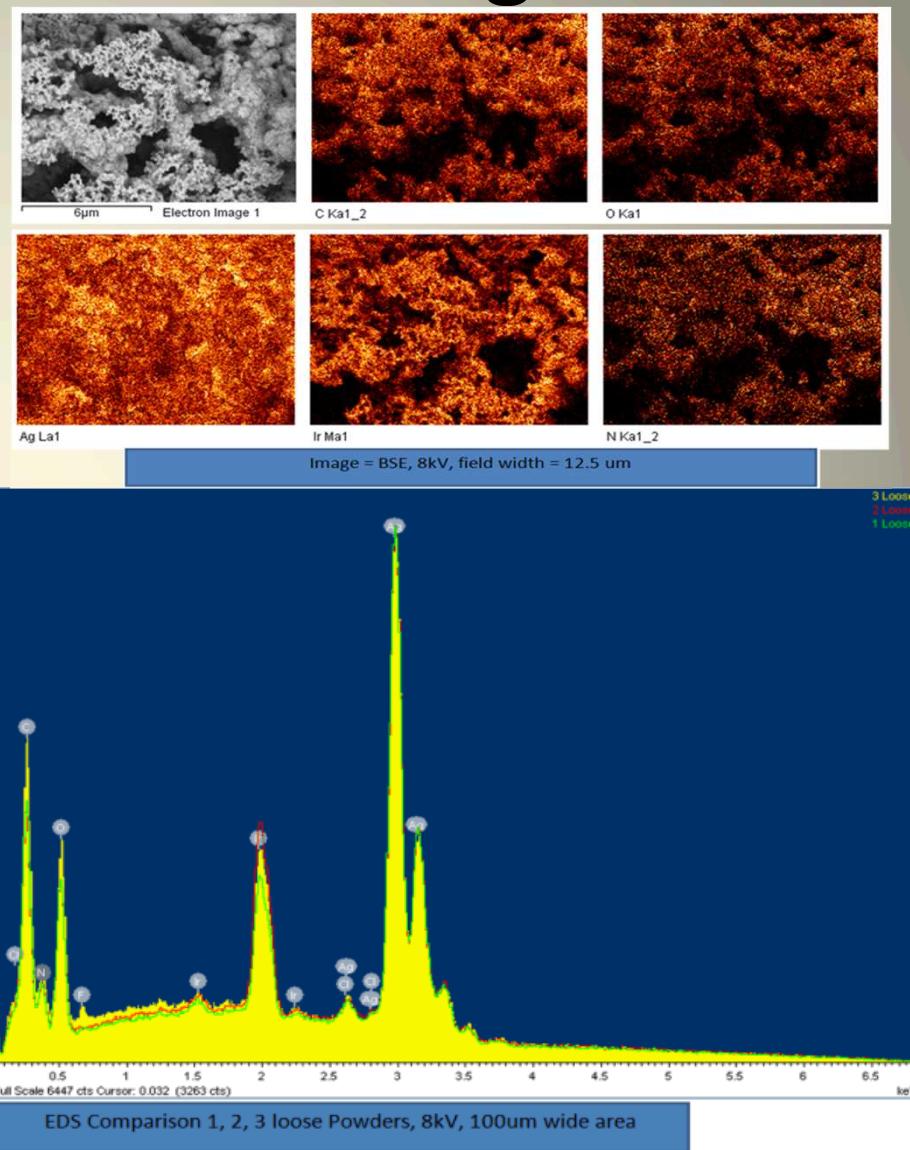
## Microstructure and Molecular Signature Characterization Techniques

### 2. Energy Dispersive X-ray Spectroscopy (EDS or EDX)



Ref: Chavez, M.A., SAND2012-3438P, April 2012.

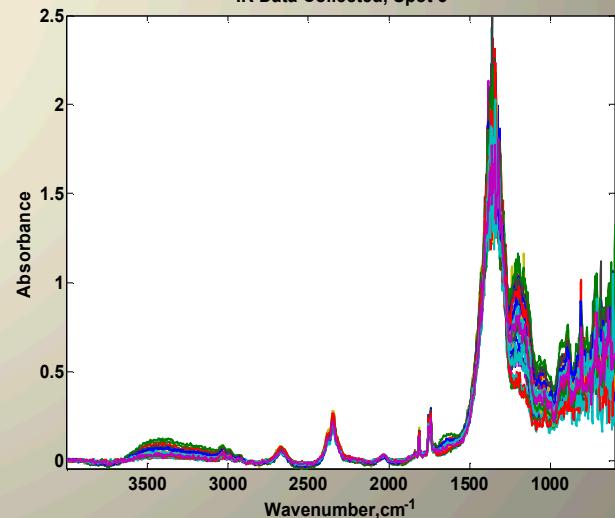
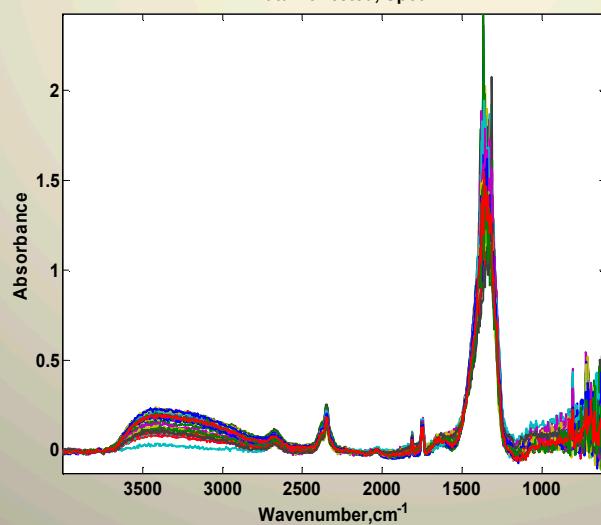
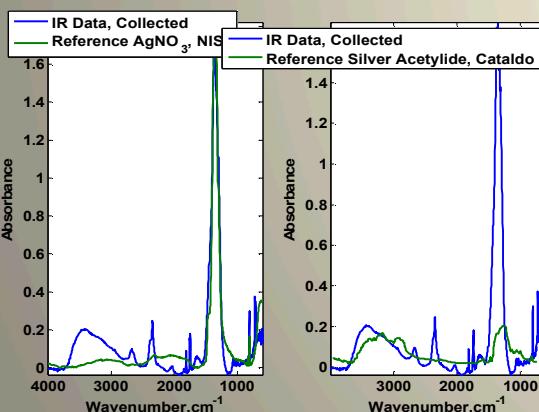
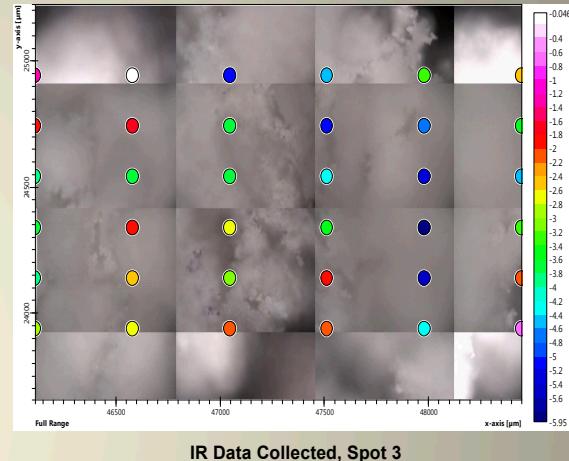
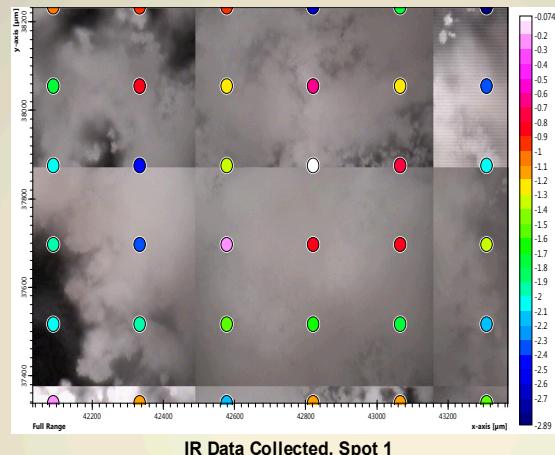
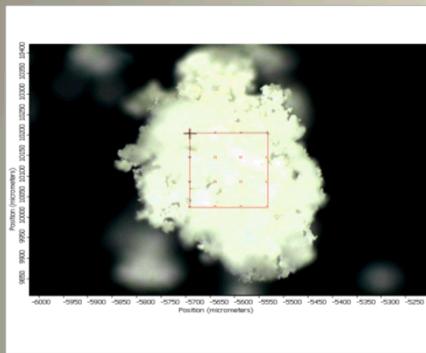
Sandstrom, M.M., Preston, D., Archuleta, J.G., Roemer, E.L., Safety Analysis of Silver Acetylide-Silver Nitrate Explosive by Los Alamos National Laboratory's WX-7 High Explosives Science & Technology Group, March 2013.



# Past and Present Research: Diagnostics

## Microstructure and Molecular Signature Characterization Techniques

### 3. Fourier Transform Infrared spectroscopy (FTIR)



Ref: Chavez, M.A., Covert, T.T., SAND2013-3088C, April 2013.

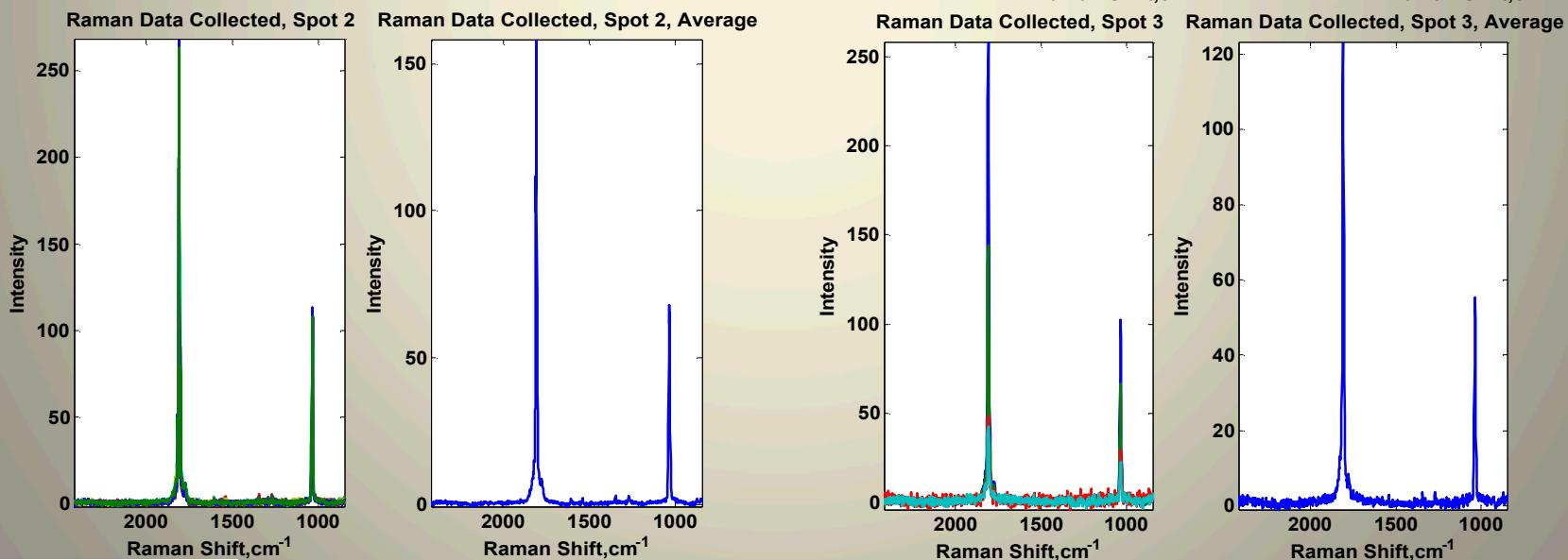
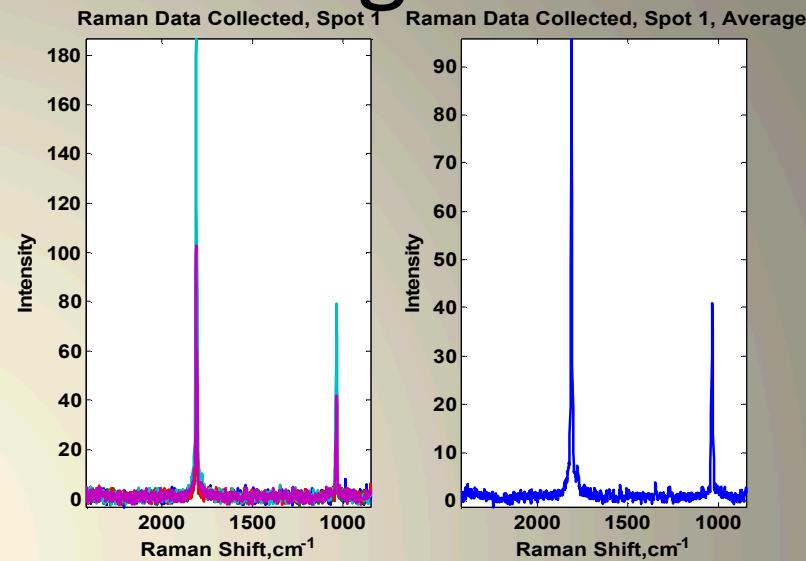
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# Past and Present Research: Diagnostics

## Microstructure and Molecular Signature Characterization Techniques

### 4. Raman spectroscopy

- Guo et. al. reports the Raman stretching frequency of the  $\text{C}\equiv\text{C}$  bond at  $2069\text{ cm}^{-1}$ , but not seen
- $\text{NO}_3$  at  $1040\text{ cm}^{-1}$  (from  $\text{AgNO}_3$ )
- $\text{C}=\text{C}$  bond at  $1812\text{ cm}^{-1}$  (from  $\text{Ag}_2\text{C}_2$ )
- May indicate  $\text{Ag}_2\text{C}_2$  and  $\text{AgNO}_3$  mixture instead of double salt



Ref: Chavez, M.A., Covert, T.T., SAND2013-3088C, April 2013.

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# Past and Present Research: Diagnostics

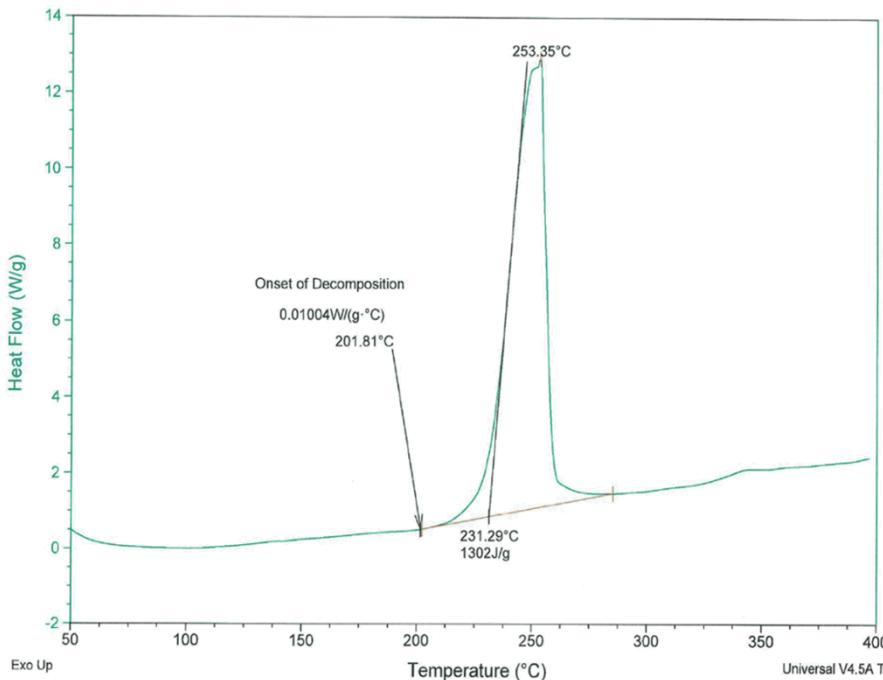
## Thermal Characterization Techniques

### 1. Differential Scanning Calorimetry

Sample: SASN  
Size: 0.1310 mg  
Method: 10C Ramp

DSC

File: C:\TA\Data\DSC\51706\SASN.002  
Operator: MMS  
Run Date: 15-Feb-2013 14:10  
Instrument: DSC Q2000 V24.10 Build 122



Ref: Sandstrom, M.M., Preston, D., Archuleta, J.G., Roemer, E.L., Safety Analysis of Silver Acetylide-Silver Nitrate Explosive by Los Alamos National Laboratory's WX-7 High Explosives Science & Technology Group, March 2013.

Sample: SASN  
Size: .61 mg  
Rate: 10° C/min  
Program: Interactive DSC V3.0 (Quantitative Data)

Date: 8-Feb-85 Time: 12:45:00  
File: SASNB.01.GENERAL.02  
Operator: TMM  
Plotted: 8-Feb-85 13:37:51

DSC

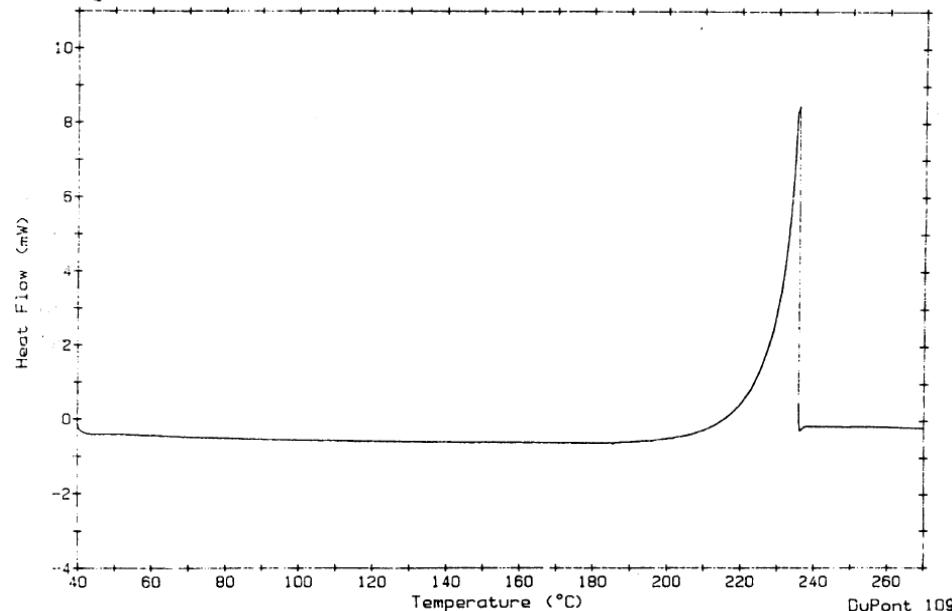


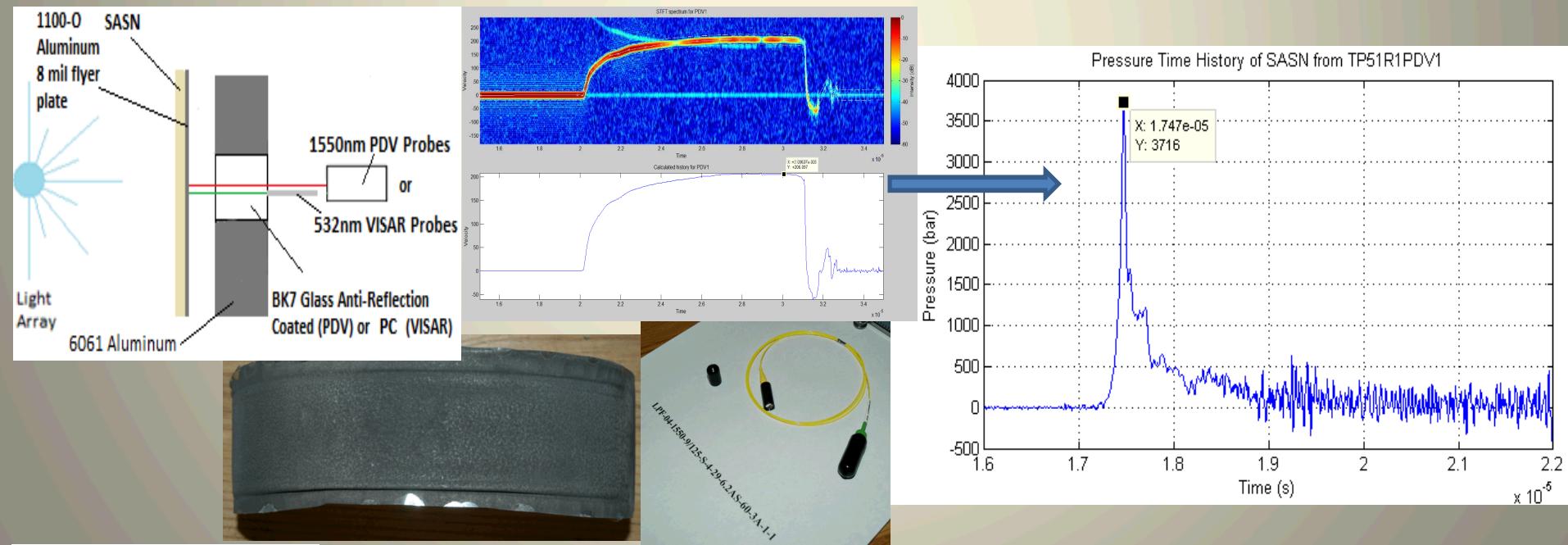
Figure 6. Thermal Energy Output from Sprayed SASN as a Function of Temperature for Heating at 10°C/min. (Measurement made with a differential thermal analyzer).

Wilden, M.W., *Investigations Pertinent to Ignition of Sprayed Layers of Silver Acetylide-Silver Nitrate*. Albuquerque, NM: Sandia National Laboratories, May 1986. SAND85-1859.

# Past and Present Research: Diagnostics

## Explosive Output Determination Techniques

1. Specific impulse via ballistic pendulum
2. Flyer plate velocity time histories coupled with photonic Doppler velocimetry (PDV)
3. Pressure time histories via PDV, velocity interferometry for any reflective surface (VISAR), and carbon gauges in cut-back experiments



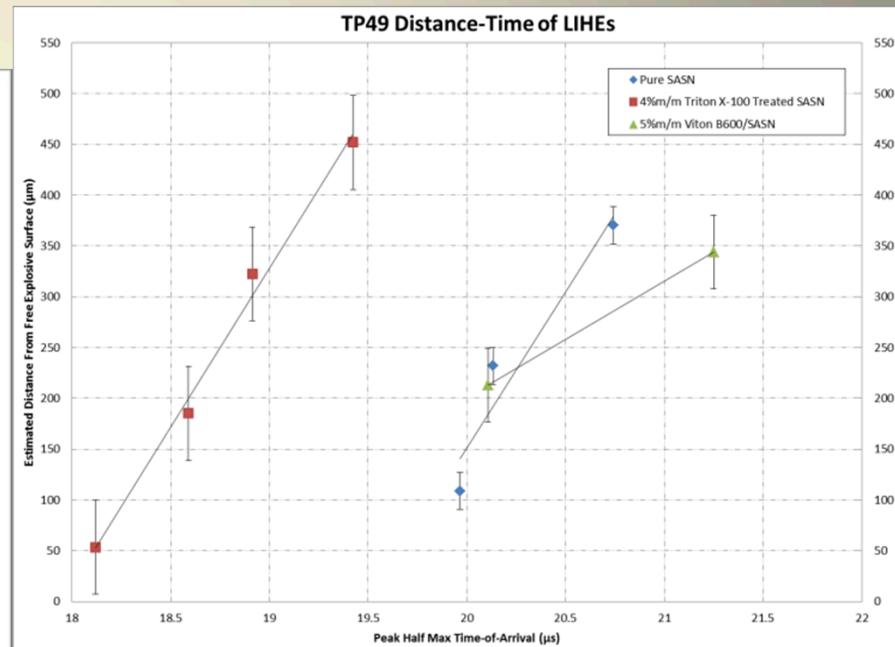
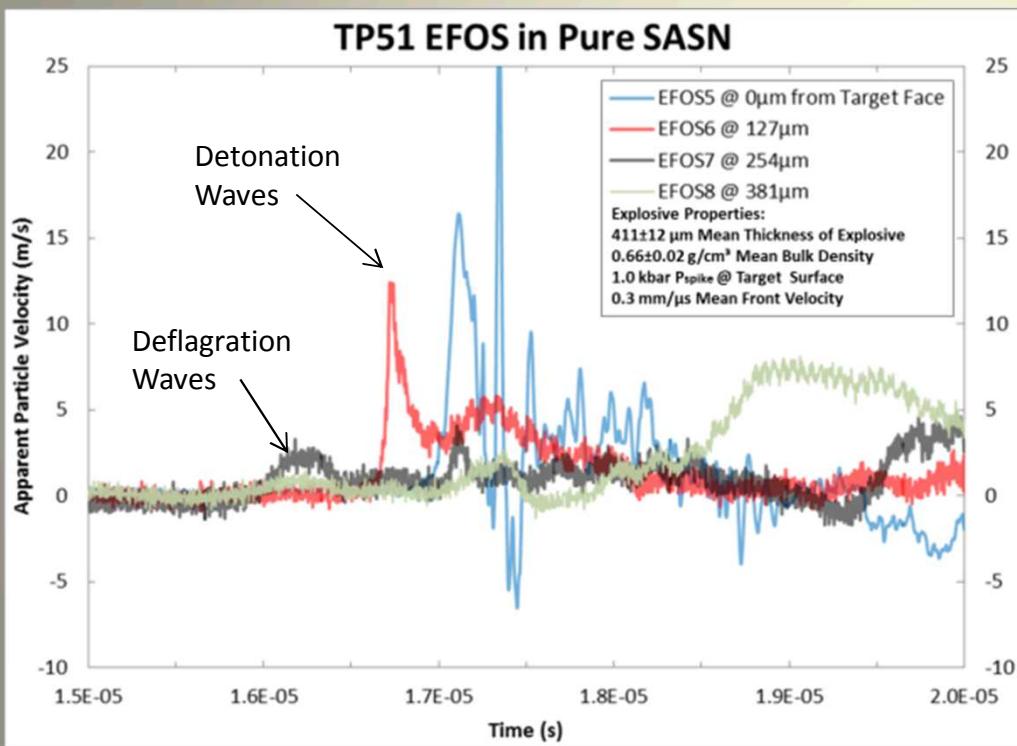
Ref: Chavez, M.A., Covert, T.T., SAND2013-3088C, April 2013.

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# Past and Present Research: Diagnostics

## Explosive Output Determination Techniques

4. Interface (particle) velocity time histories and deflagration/detonation velocities via PDV and VISAR with cut-back and embedded gauge techniques (EFOS)



Detonation velocity for traversing wave=1.2mm/μs as compared to through the thickness of 0.3mm/μs

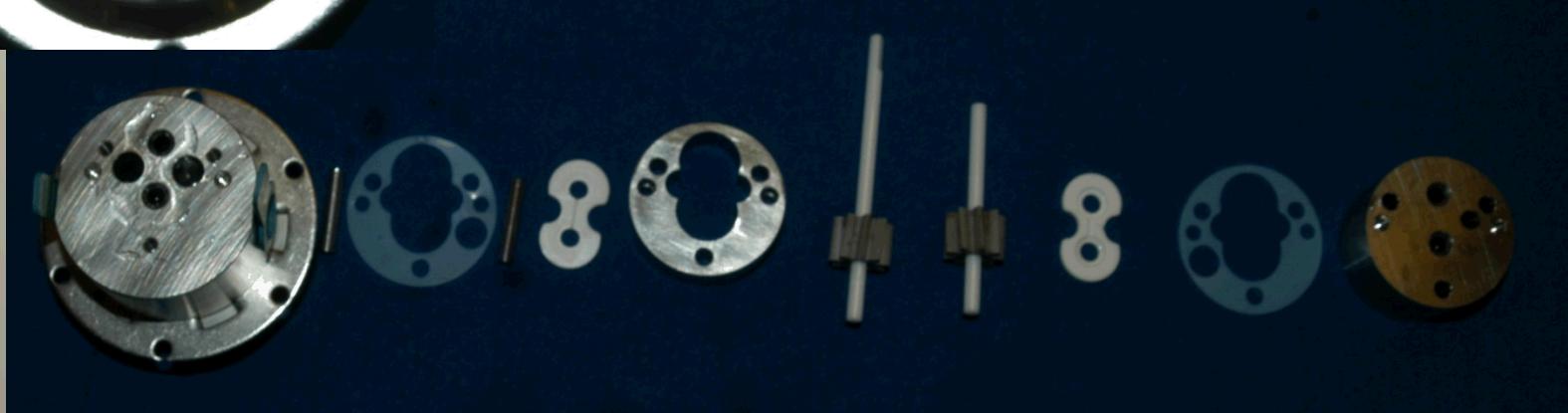
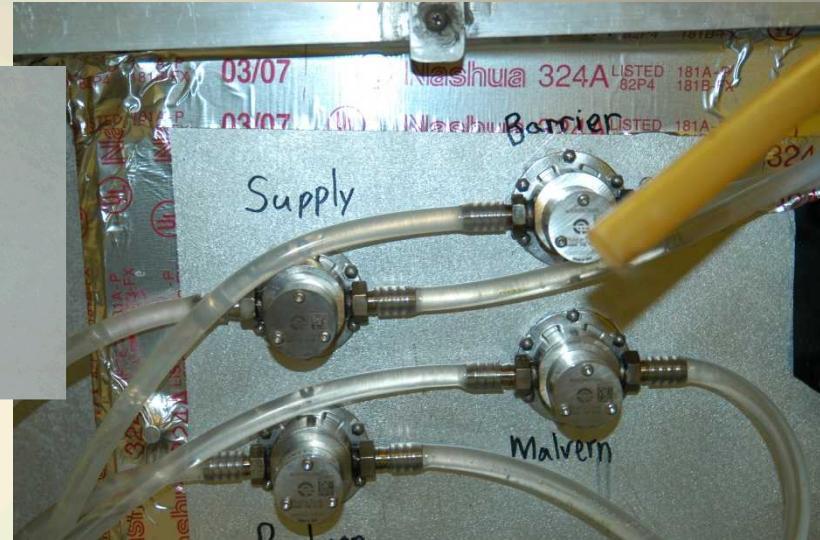
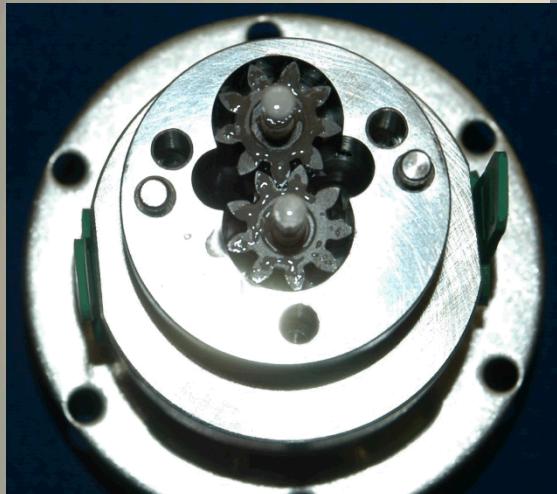
# Proposed Research and Activities

- Reduction and control of PSD via high shear mixing (Recently employed)
  - Increase specific surface area (SSA)
  - Subsequent densification expected
  - Potentially employ during synthesis or use ultrasonic horn



# Proposed Research and Activities

- Reduction of pulsation in mass flow rate to spray gun via magnetically coupled gear pumps  
(Recently employed)



# Proposed Research and Activities

- New or modern deposition technologies for higher deposition uniformity and densification control
  - Modern conventional spray gun (Employed now due to unavailability of old models)
  - Ultrasonic spray nozzles (Acquired a conical spray gun)
  - Electrostatic spray nozzles (arcing dangerous, but can be overcome)
  - 3D printing of explosive (Other SNL teams are researching this)
  - Optimize spray parameters
  - Investigate Viton B adherence layer contribution

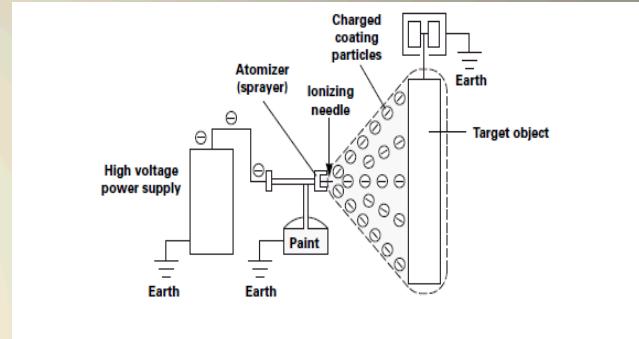
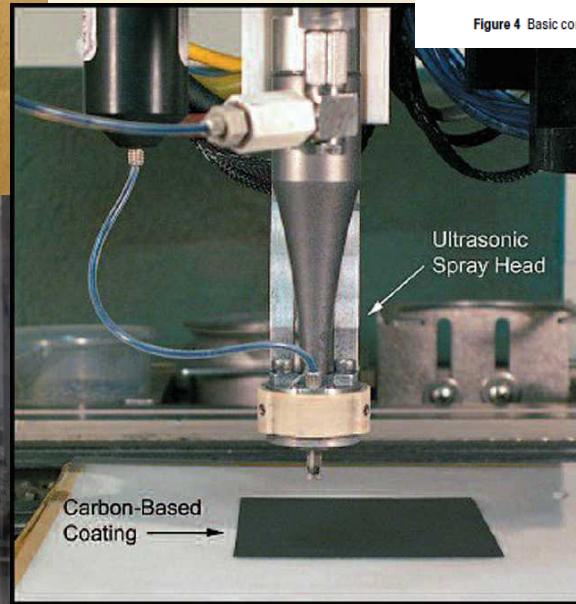
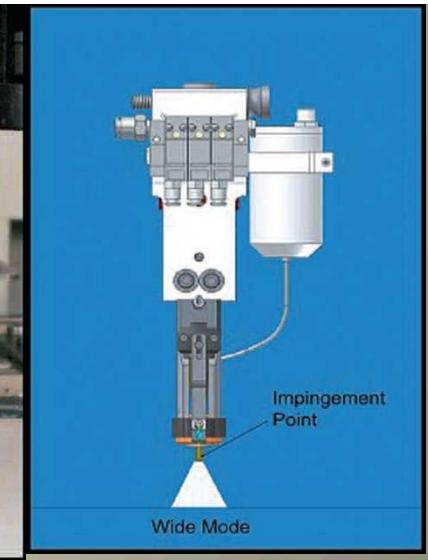


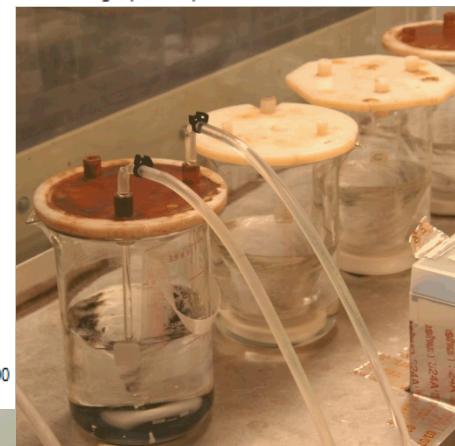
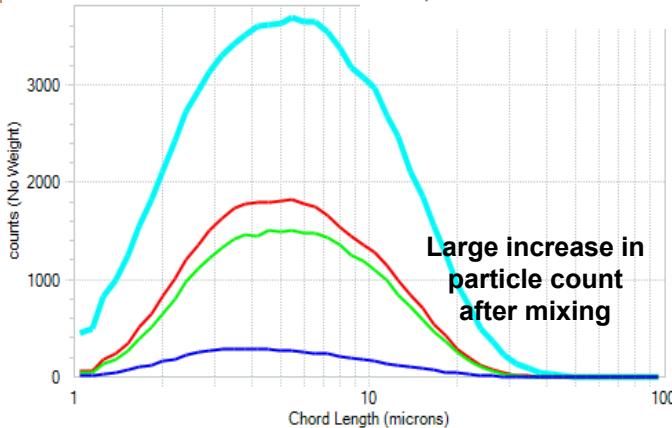
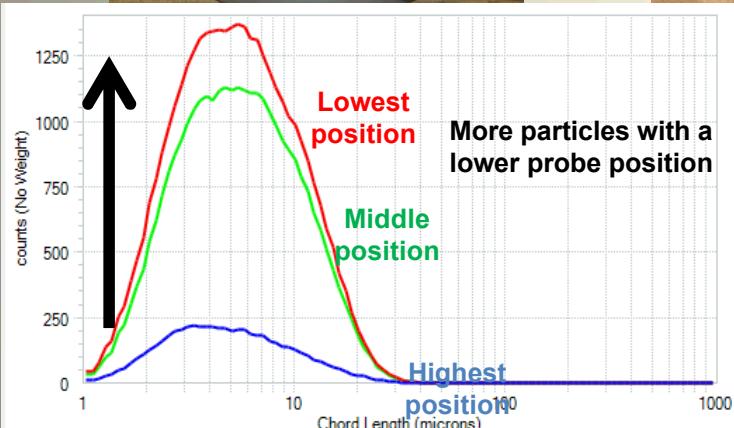
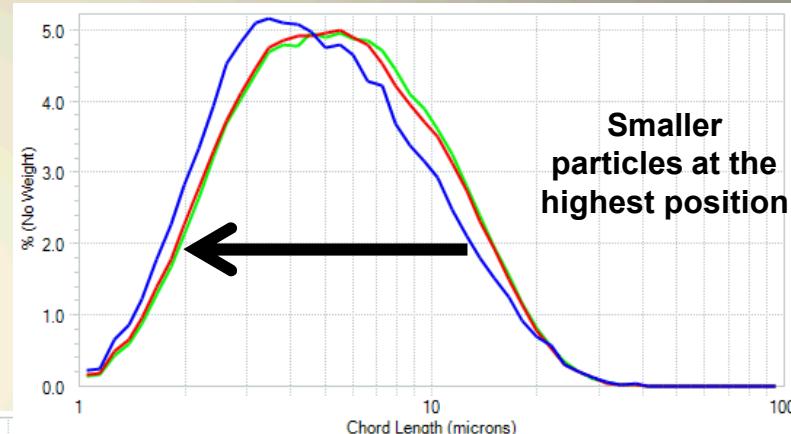
Figure 4 Basic components of electrostatic spray equipment



Ref: Erickson, S., Updates in Nozzle-less Ultrasonic Coating Technology, March 2007.

# Proposed Research and Activities

- Higher degree of uniformity for slurry in beakers
  - Magnetically coupled high shear blenders (Employed now)
  - Optimize mass solids loading for deposition technology employed
  - Acoustic mixing (ideal)



Ref: Seliga, C., Dycus, E., Chavez, M., Ensure Optimal and Repeatable Flyer Plate Testing by Controlling Propellant Size/Count with ParticleTrack and Particle View, Internal Presentation, Mettler-Toledo/SNL collaboration, October 2014.

# Proposed Research and Activities

- Substrate compatibility
  - Ensure uniform samples in terms of geometry and ability to control materials in contact (Recently employed)
  - Chemical inerting of explosive

**Standard Coupon**

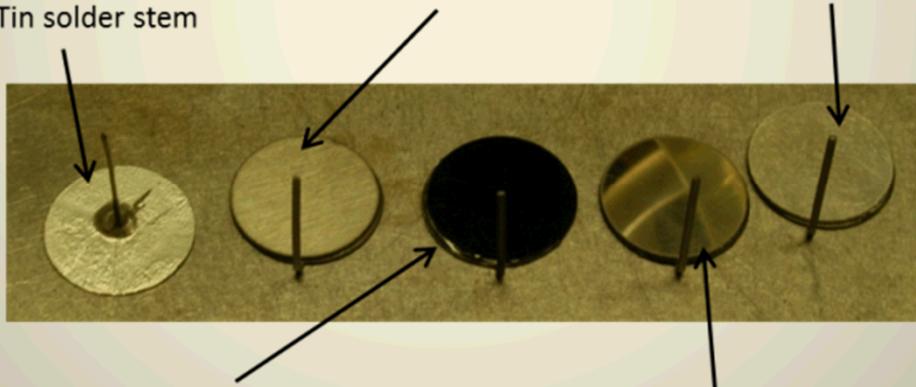
1100-O aluminum tape  
substrate (adhesive  
backed)  
Kovar steel base  
Copper/Tin solder stem

**S Coupon**

304 stainless steel substrate  
3-minute epoxy adhesive  
430 stainless steel base

**A Coupon**

1100-O aluminum substrate  
3-minute epoxy adhesive  
430 stainless steel base



**K Coupon**

Kapton B substrate  
3-minute epoxy adhesive  
430 stainless steel base

**SS Coupon**

430 stainless steel substrate  
3-minute epoxy adhesive  
430 stainless steel base

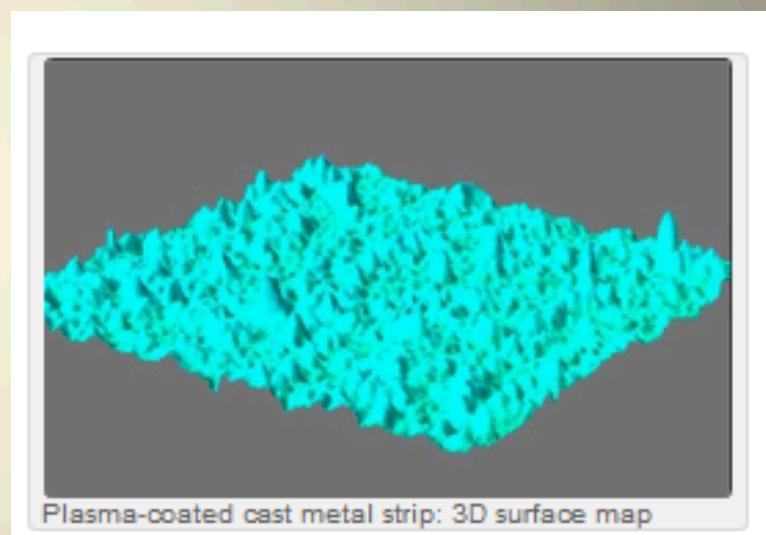
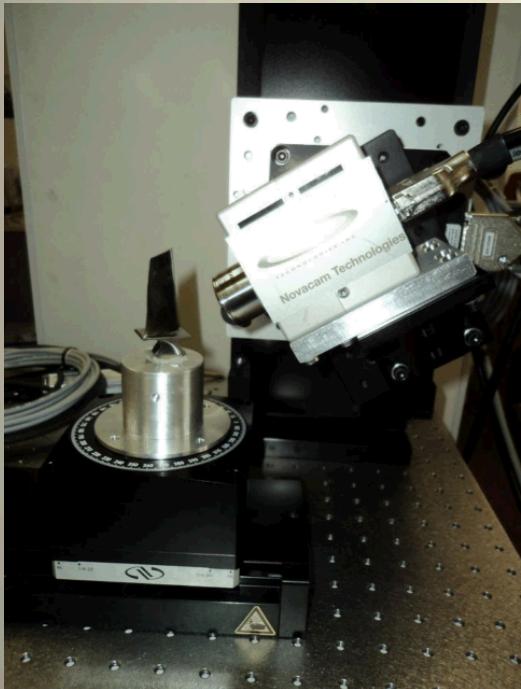
# Proposed Research and Activities

- Compilation of data and formation of appropriate mathematical model (logistic model for energetic material behavior)
  - Fit explosive performance data with measured parameters
  - Predict outcomes of processing techniques to validate
  - Apply to other well-characterized energetic materials
- Addition of sensitizers/phlegmatizers to matrix to tailor explosive behavior
  - Hollow glass microspheres with appropriate porosity size and spatial distribution for sensitization
  - Viton B600 for phlegmatization (Employed in past)
- Improve light initiation
  - Higher irradiance photon source
  - Optimize spectral output of photon source
  - Create photon trap on surface of energetic material

# Proposed Research: New Diagnostics

## Deposition Determination Techniques

1. Infrared laser interferometer based surface profilometer mounted to robotic arm for in-situ monitoring (Expected to purchase before end of September 2016)
2. XRF calibrated with stainless steel based substrates (backup)

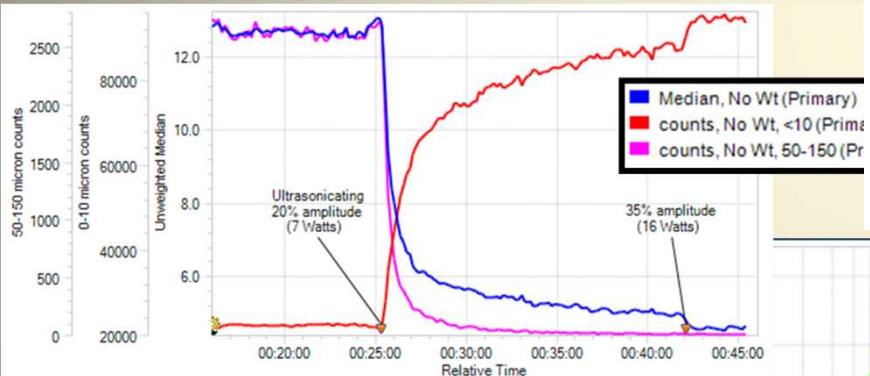


# Proposed Research: New Diagnostics

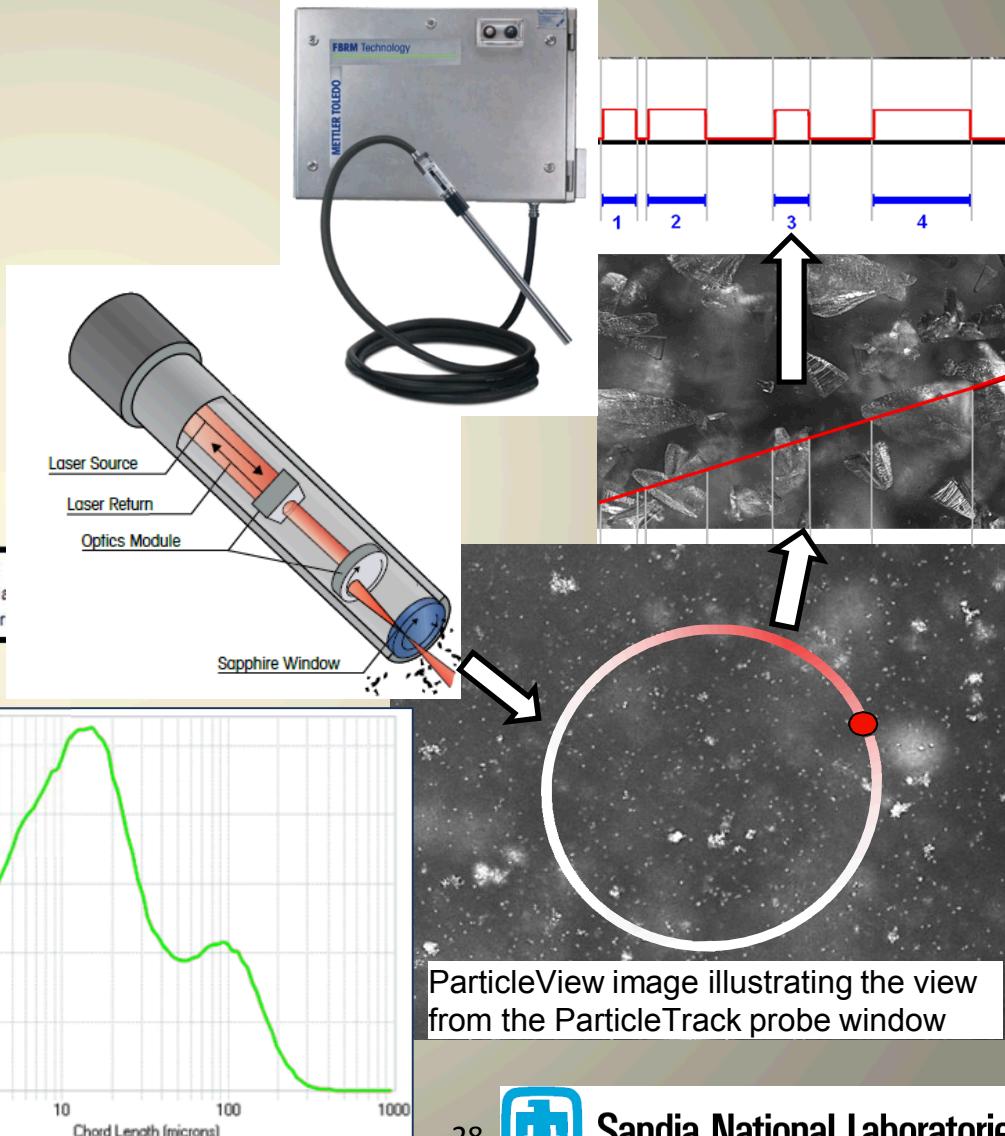
Mettler-Toledo ParticleTrack® Technology

## Synthesis and Slurry Characterization Techniques

1. Focused Beam Reflectance Measurement (FBRM) for in-situ full-concentration PSD (used for surrogate exploration & expected to be employed in June 2016)



Ref: Seliga, C., Dycus, E., Chavez, M., Ensure Optimal and Repeatable Flyer Plate Testing by Controlling Propellant Size/Count with ParticleTrack and Particle View, Internal Presentation, Mettler-Toledo/SNL collaboration, October 2014.



# Proposed Research: New Diagnostics

## Spray Characterization Techniques

1. High speed photometrics coupled with laser diffraction for DSD and tracking

## Microstructure and Molecular Signature Characterization Techniques

1. Cross-sectional spatial mapping with EDS, FTIR, and Raman (Expected summer 2016)
2. Micro X-ray Computed tomography (CT) for porosity mapping (Expected summer 2016)
3. Helium based pycnometry for density (validation of current particle density data) (Expected summer 2016)
4. Surface area analyzer for SSA and open porosity (backup)

# Summary

- Appropriate diagnostics suite key to characterizing and controlling material
- Past and present research have laid a solid foundation to proceed to proposed study
- Data mining and compilation will continue
- Acquisition of new hardware and diagnostics are in progress
- High shear mixing of explosive are in-progress with preliminary results promising
- Mathematical modeling will occur with proper dataset acquired