

# Nanoparticle preparation

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## Ball Milling Method

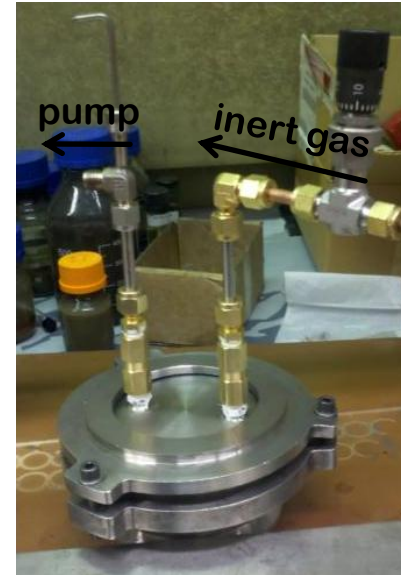
Physically grind micron size feedstock down to the nanoscale regime using planetary ball milling with WC balls

Particles are coated with various ligands/capping agents to promote suspension in a variety of fuels and/or to protect them from unwanted oxidation

## Particle Characterization

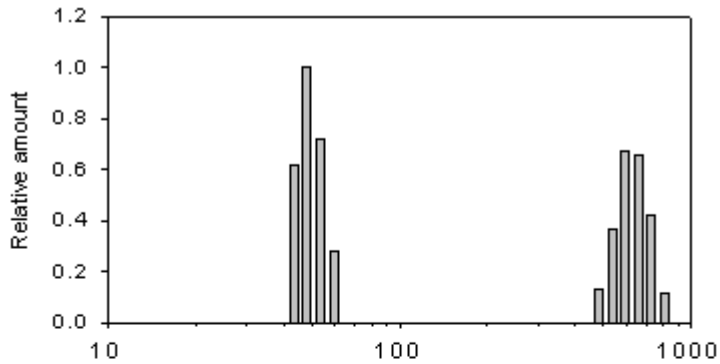
Chemical composition: XPS, FTIR, TGA

Particle Size: DLS, SEM, TEM

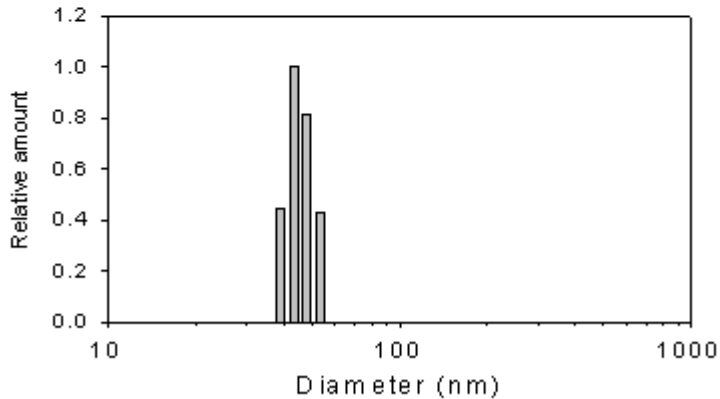


*Retsch PM400 ball mill*

# ~50 nm particles separated by settling big particles

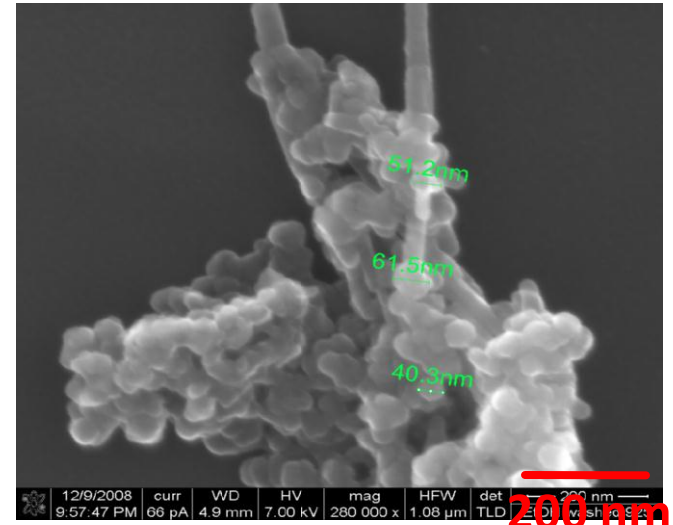


Volume weighted

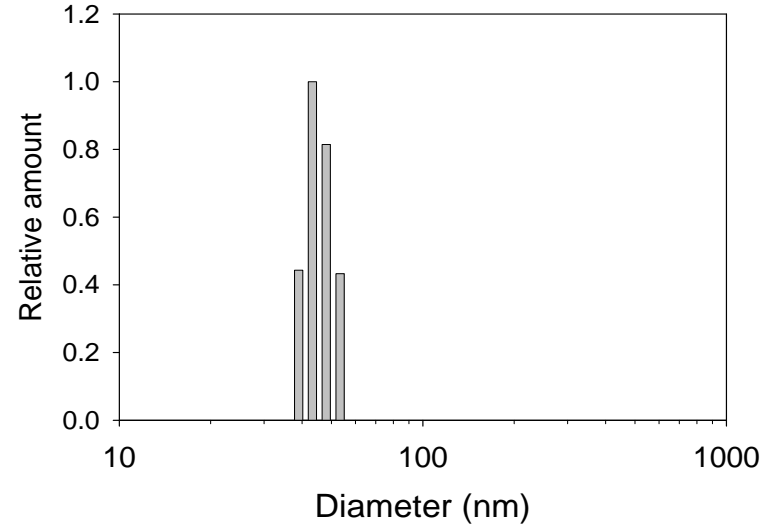


Number weighted

Boron Nanoparticle Size distribution

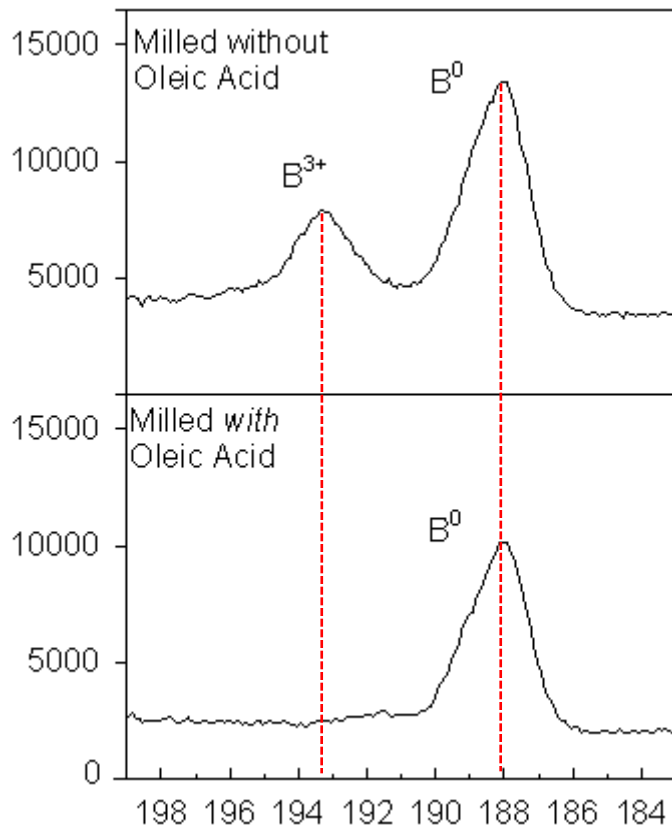


SEM image of <100 nm particles



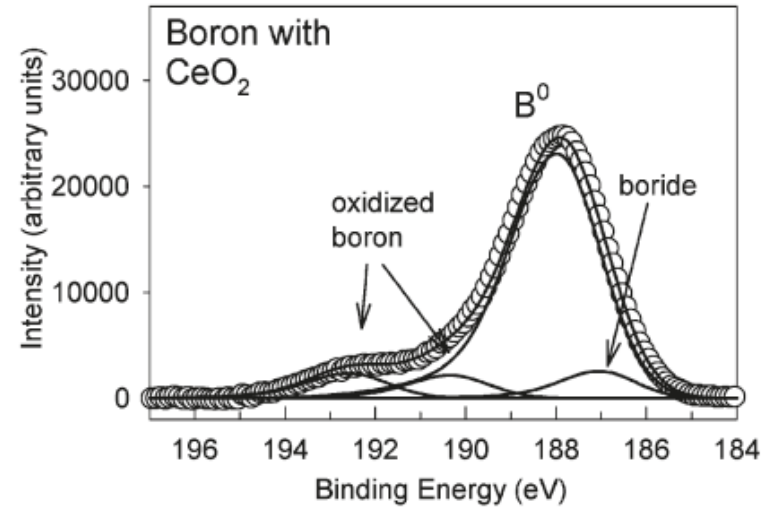
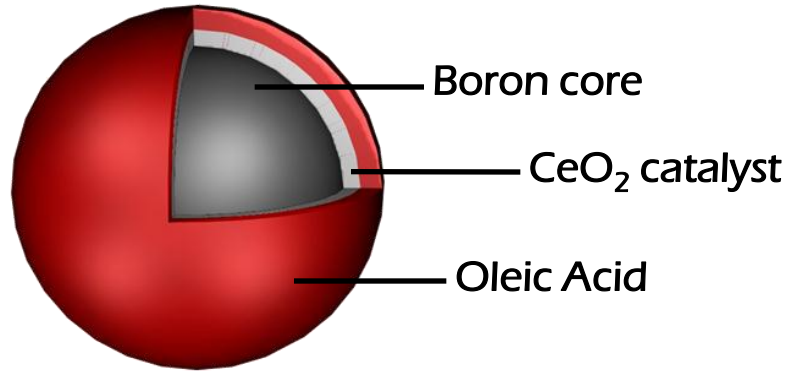
Volume weighted size distribution

# Unoxidized Boron Nanoparticles

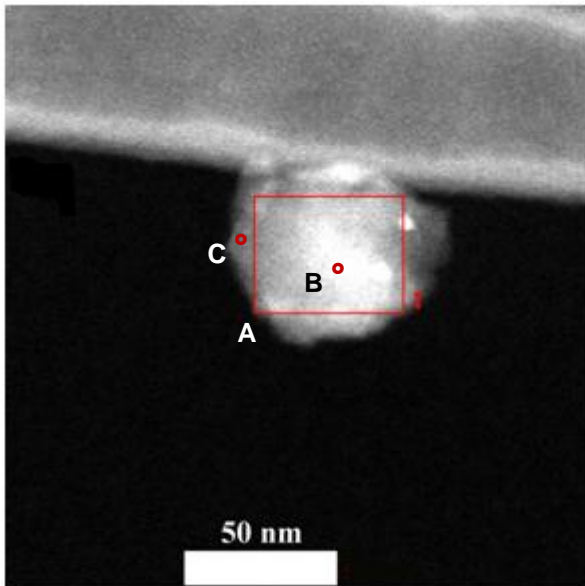


- Elemental  $B^{1s}$  peak observed at  $\sim 188$  eV,  $B^{3+}$  at  $\sim 193$  eV
- Nanosized boron immediately formed oxide upon exposure to air as the XPS samples were prepared.
- Oleic acid prevented oxidation of the fresh boron surfaces formed during the milling process.

# Catalyst Coated, Unoxidized Boron Nanoparticles



XPS



STEM

Table 3. EDX Results Boron Milled with Ceria

EDX atomic composition						
element	area A		area B		area C	
	atomic %	weight %	atomic %	weight %	atomic %	weight %
B	27.8	23.0	30.4	26.1	37.4	33.0
Ce	0.2	1.8	0.2	2.1	0.0	0.3
O	4.8	5.8	2.5	3.2	1.1	1.5
C	65.4	60.3	65.7	62.7	60.2	59.0
Cu	1.9	9.0	1.2	5.9	1.2	6.2

CeO<sub>2</sub> exists as patch of island on a boron nanoparticle

XPS suggests the presence of a low binding energy boride species (Ce<sub>x</sub>B<sub>y</sub>)

# Boron Nanoparticles with Polar Surfactants

Boron Powder +  $\frac{1}{8}$ " WC balls  
80:1 BPR

↓ 3 hrs dry milling

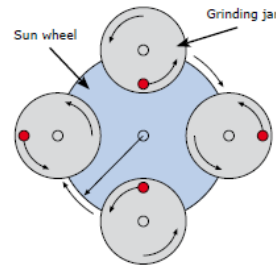
+ Surfactant (Ionic Liquid,  
 $\text{Et}_2\text{NH}$ ,  $\text{Et}_3\text{N}$ , Ethanolamine)  
~10 % total volume

↓ 3 hrs milling

+ Solvent (Ethanol)

↓ 18 hrs milling

Samples washed with EtOH to  
remove excess surfactant



[www.retsch.com](http://www.retsch.com)

**4 x 250 ml capacity**

# Method of Preparation: Ball Milling



B-Ionic Liquid



Et<sub>3</sub>N



Et<sub>2</sub>NH



Boron in EtOH



Wet Milled  
Boron in EtOH

## Solubility / Dispersibility in Ethanol (polar solvent)

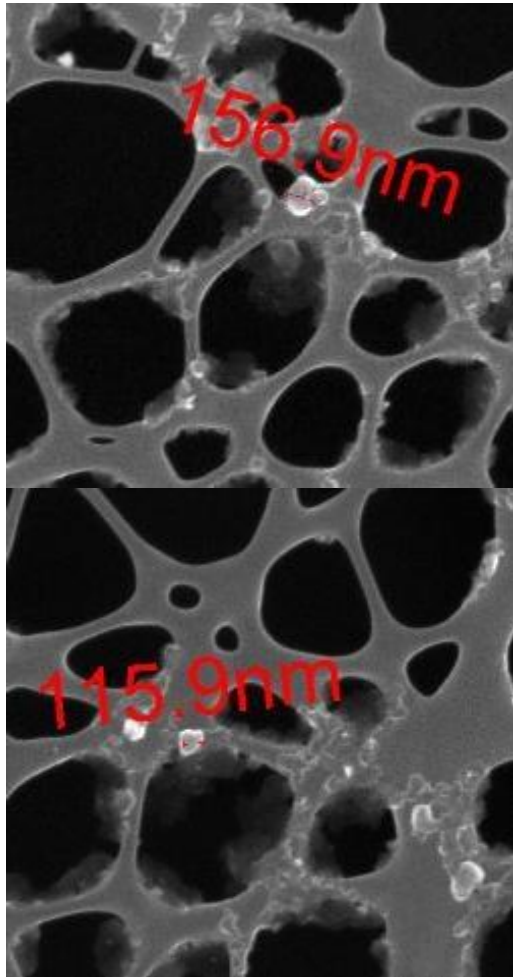
Characterize size of particles using:

SEM, DLS

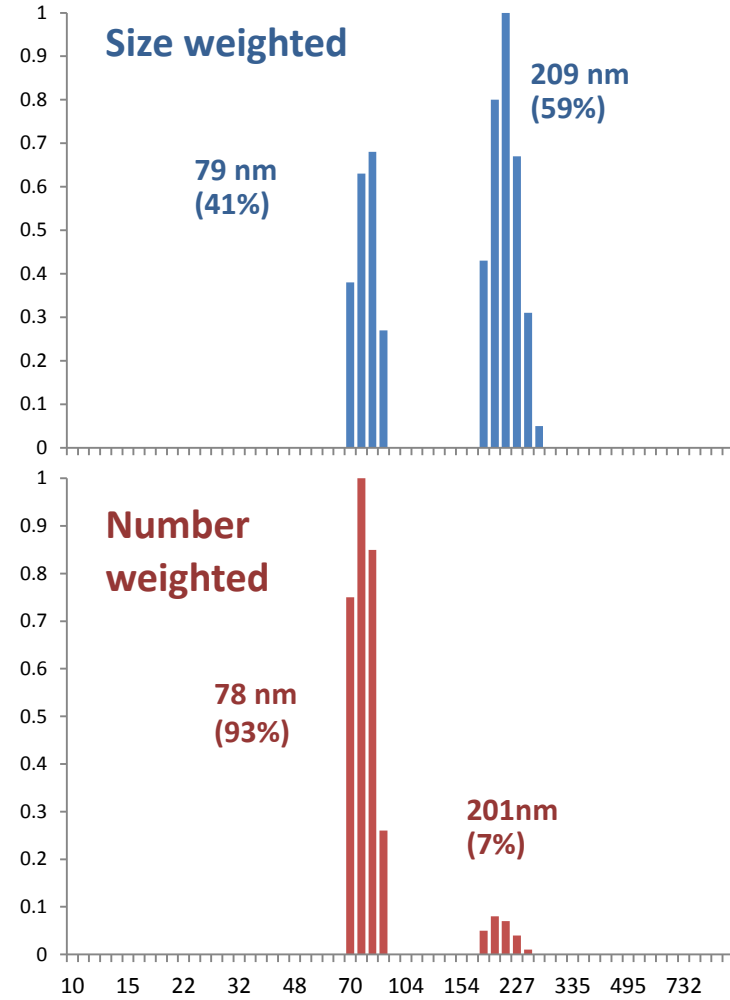
Characterize chemical composition using:

EDX-STEM, XPS, FTIR

# Boron-Ionic Liquid Particle Size



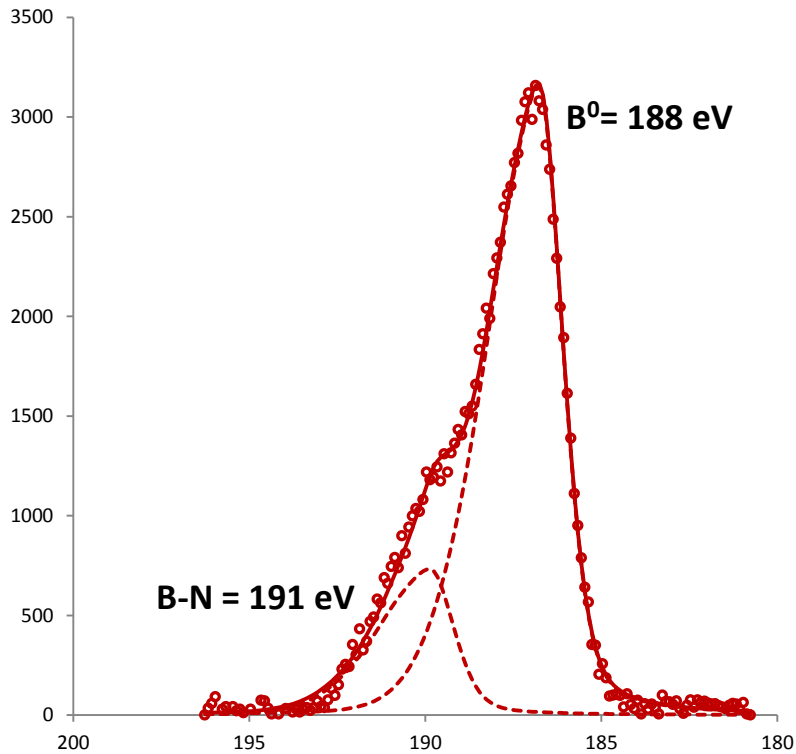
SEM Image of Boron Nanoparticles on TEM Grid



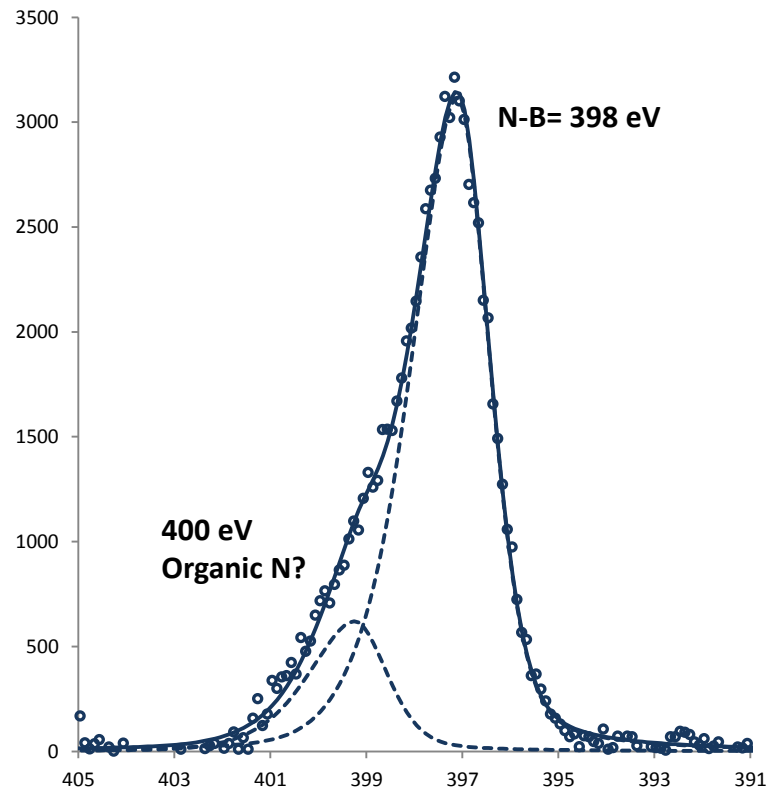
DLS Measurement of Particle Size

# Boron, Nitrogen Species

**B1s** Region XPS of Boron milled with Ionic Liquid in EtOH

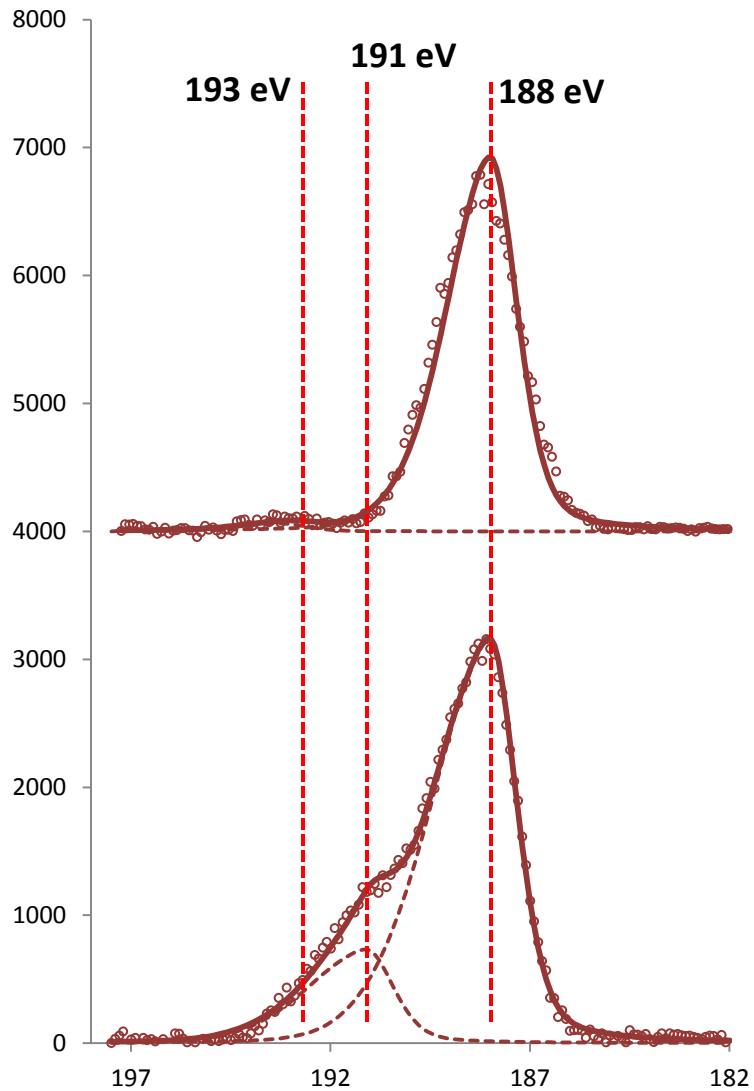


**N1s** Region XPS of Boron milled with Ionic Liquid in EtOH

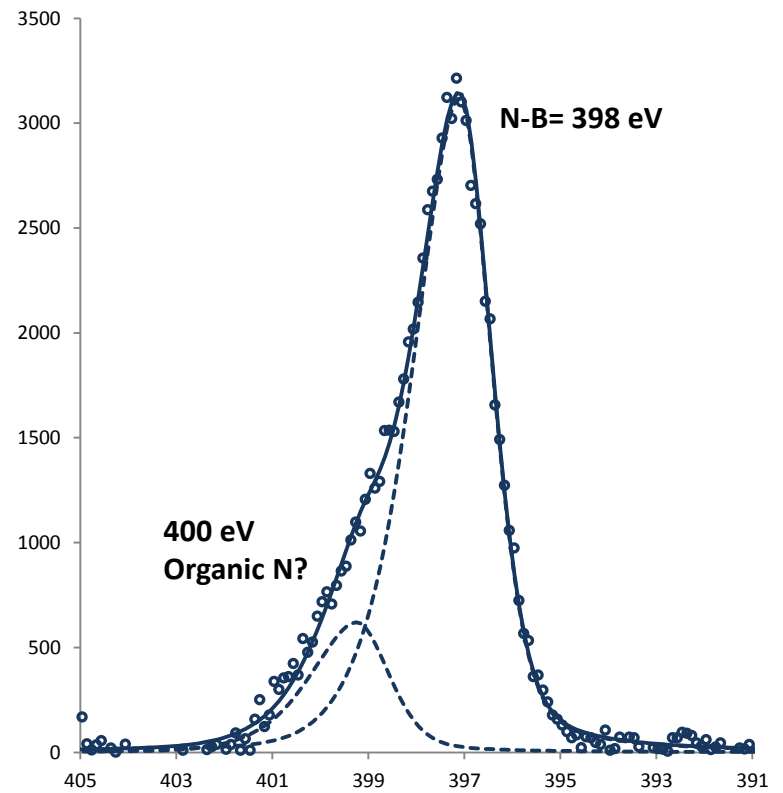




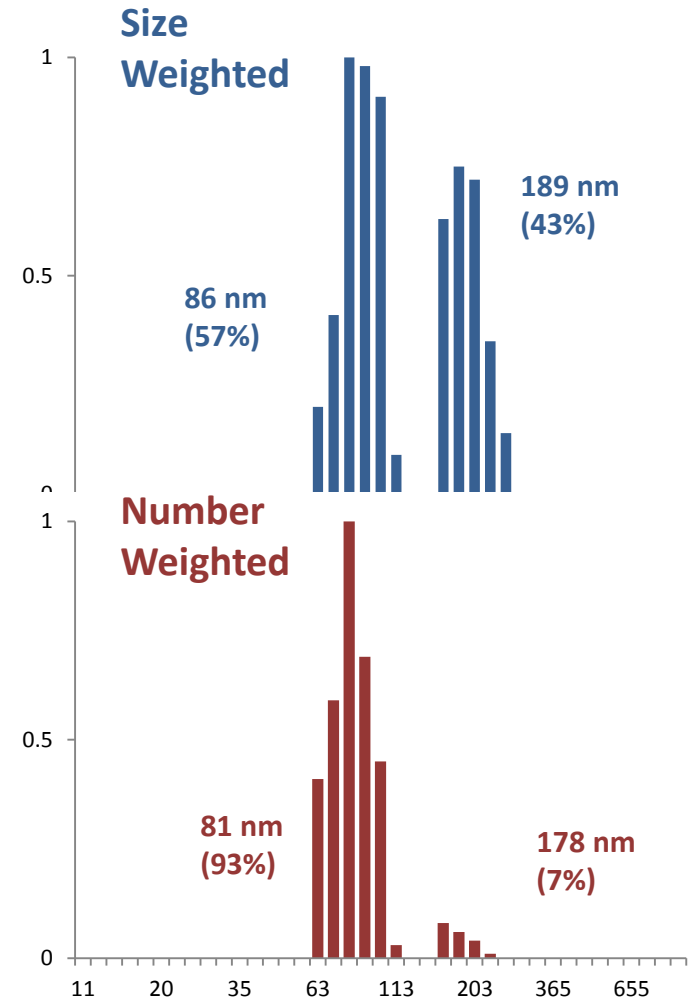
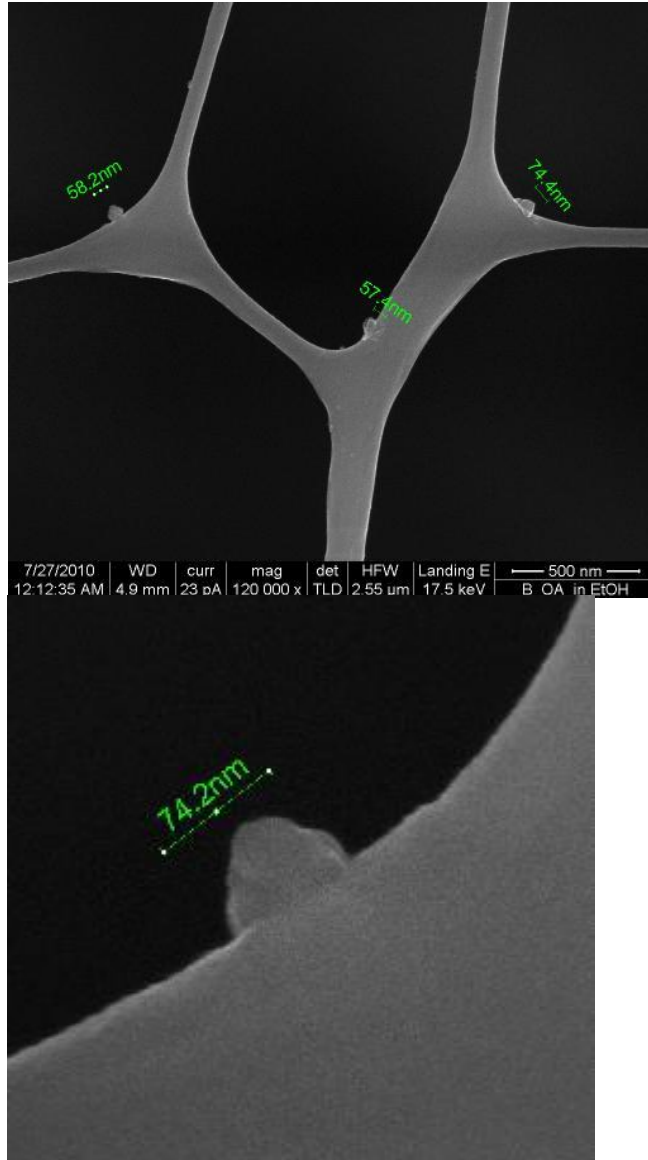
# Boron, Nitrogen Species



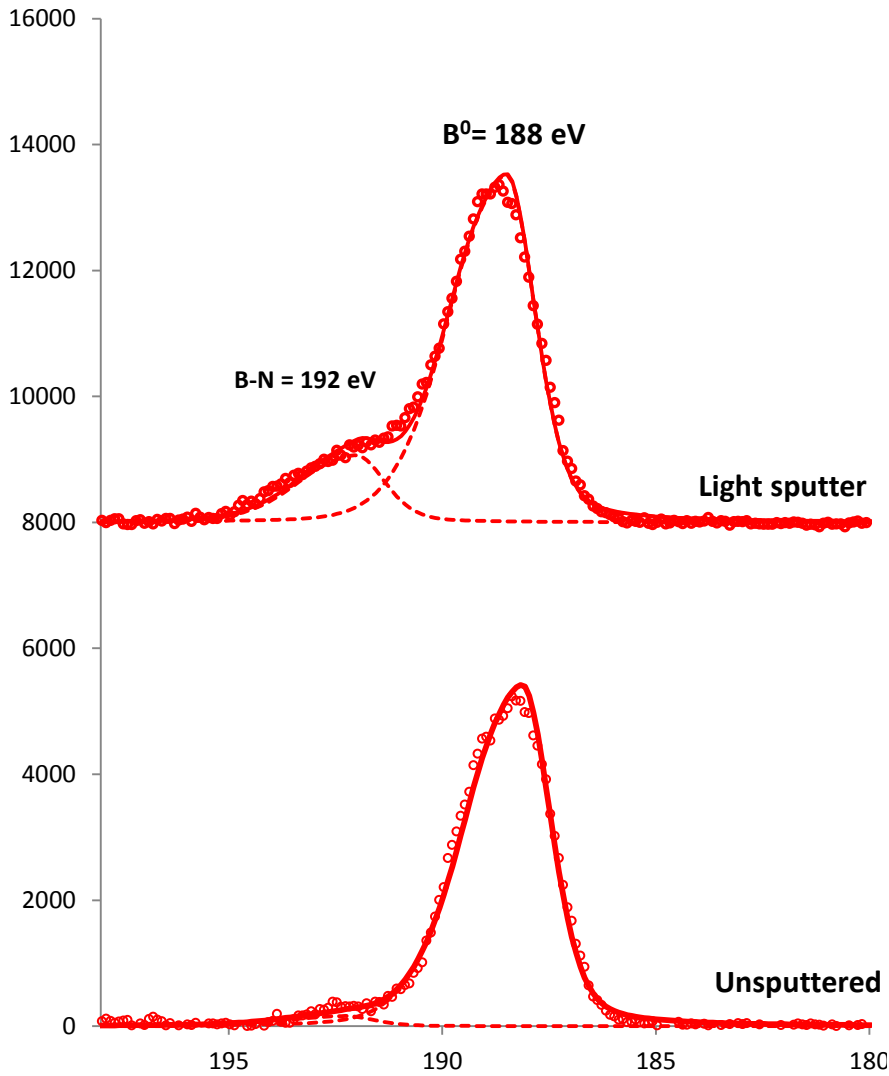
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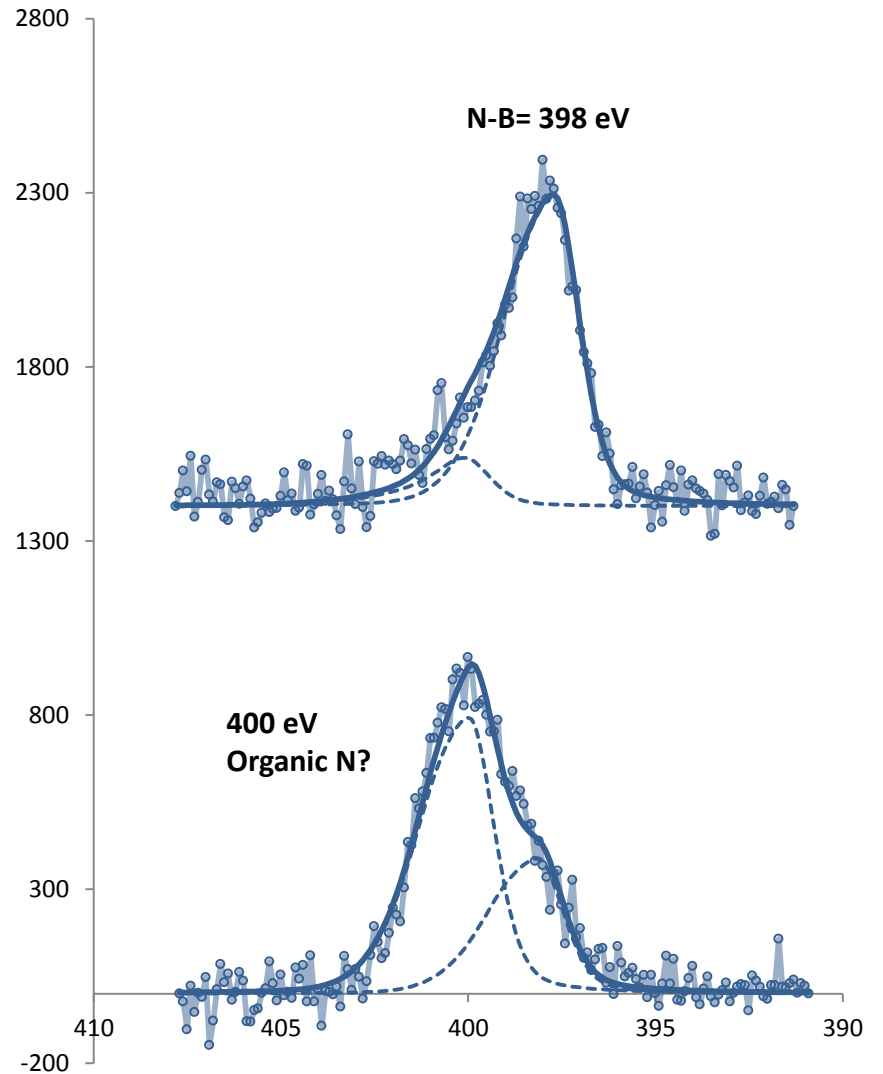
# Diethylamine Surfactants



# XPS

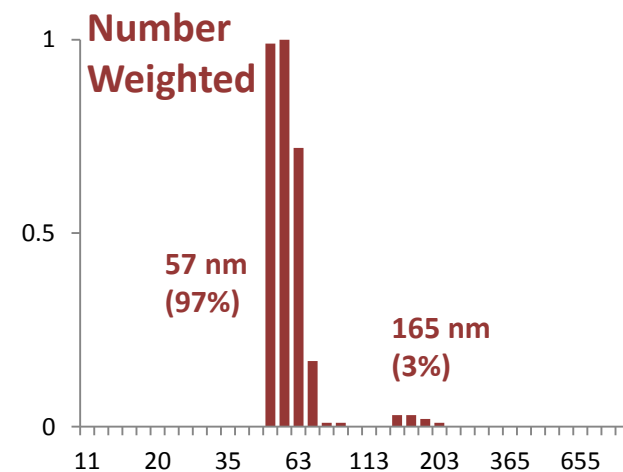
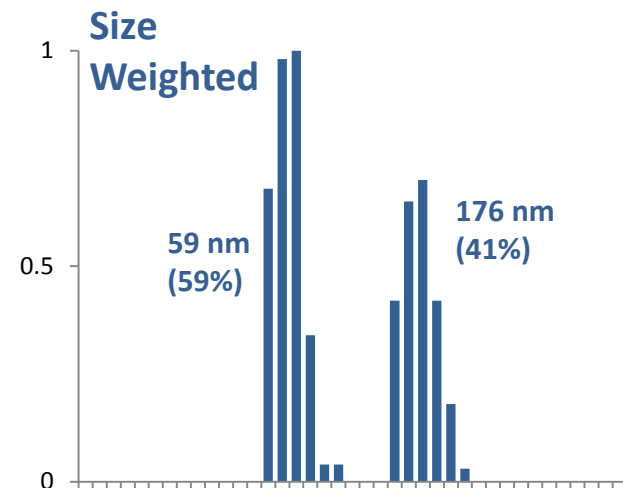
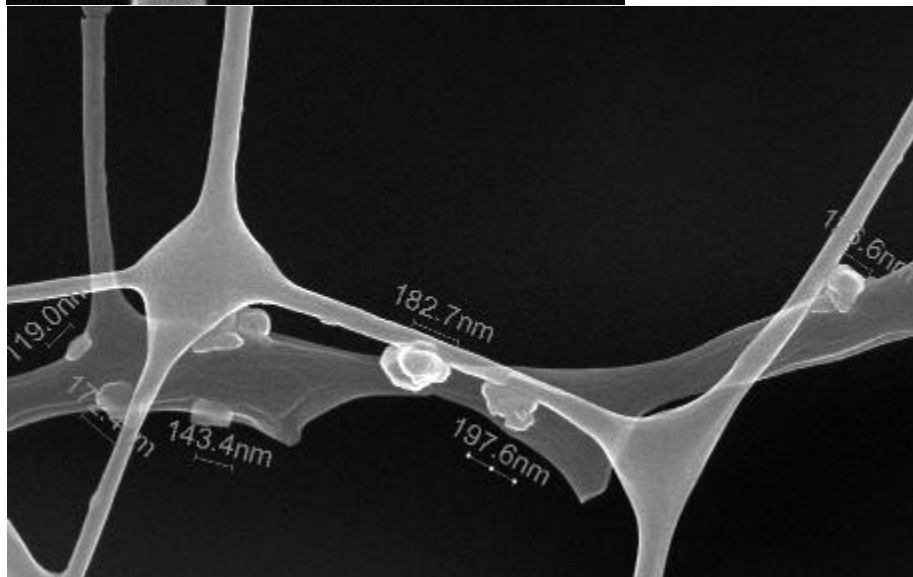
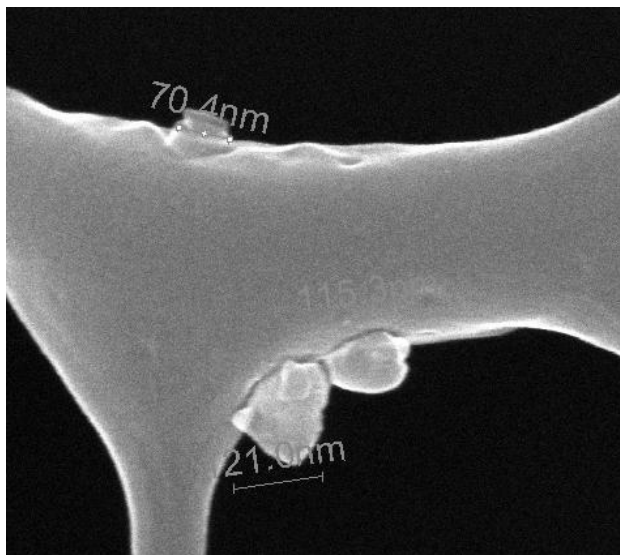


**B1s** Region XPS of Boron milled with Et<sub>2</sub>NH in EtOH

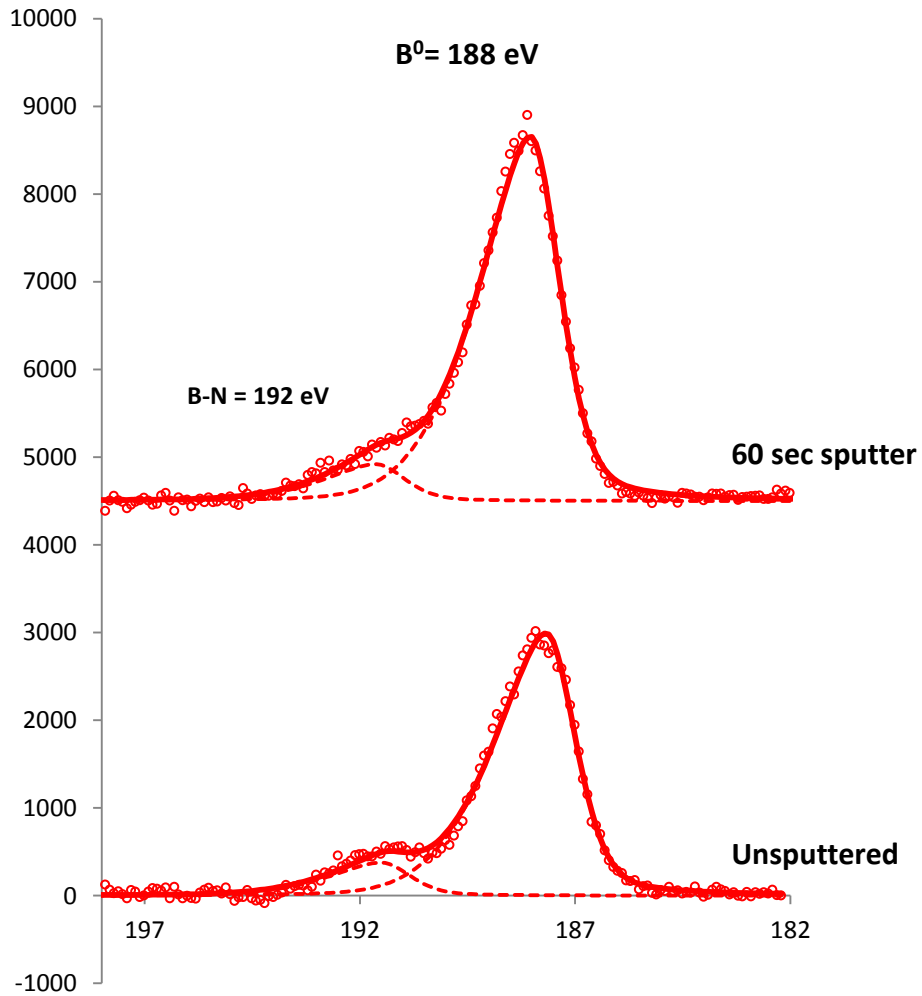


**N1s** Region XPS of Boron milled with Et<sub>2</sub>NH in EtOH

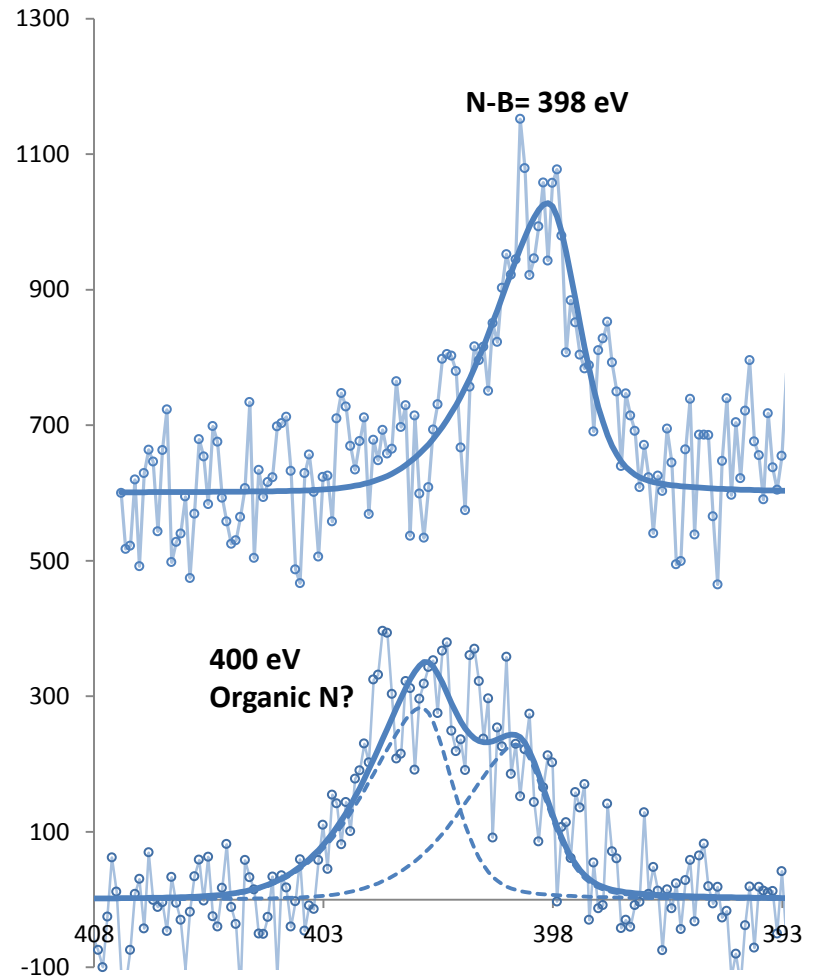
# Triethylamine Surfactants



# XPS



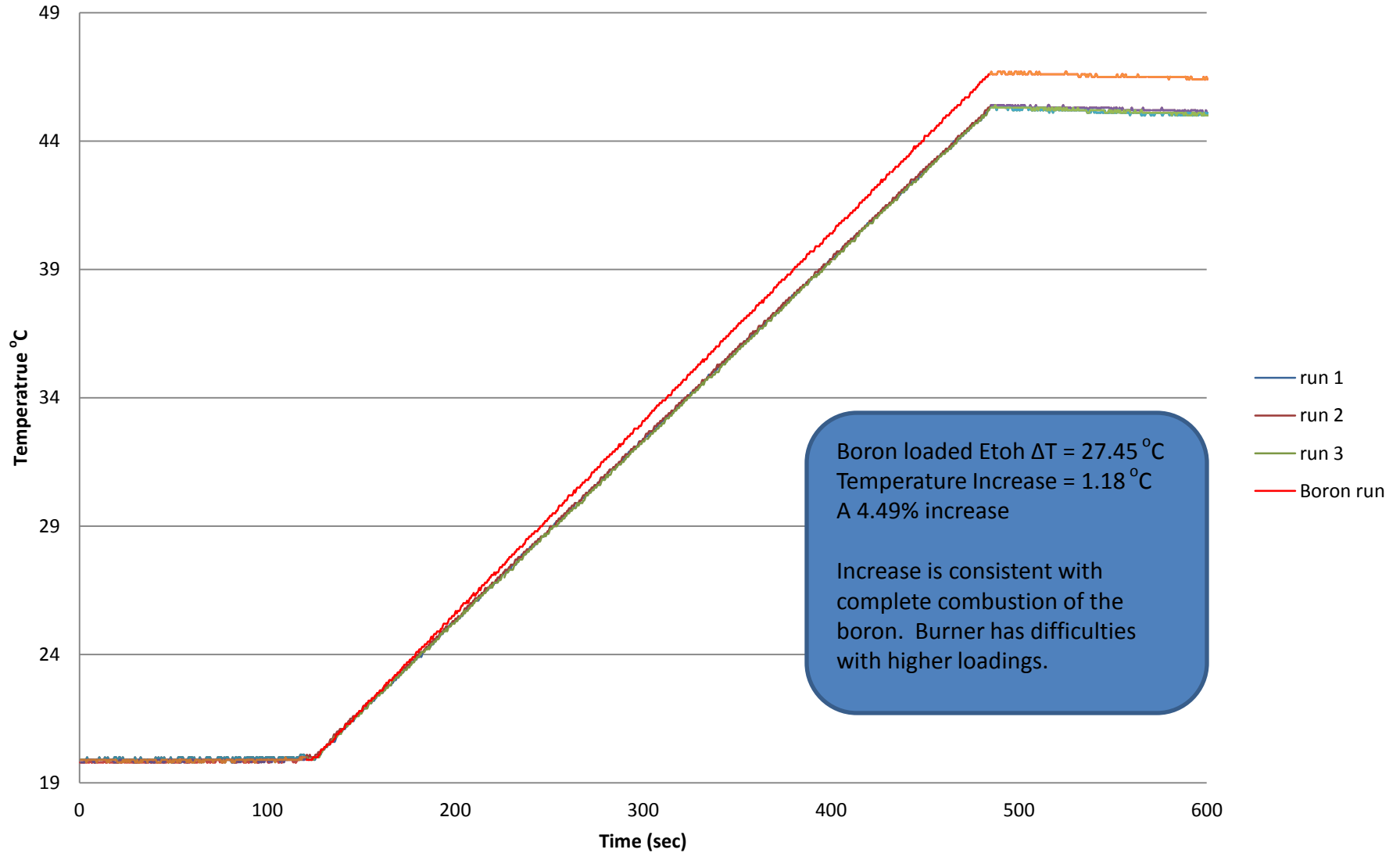
**B1s** Region XPS of Boron milled with Et<sub>3</sub>N in EtOH



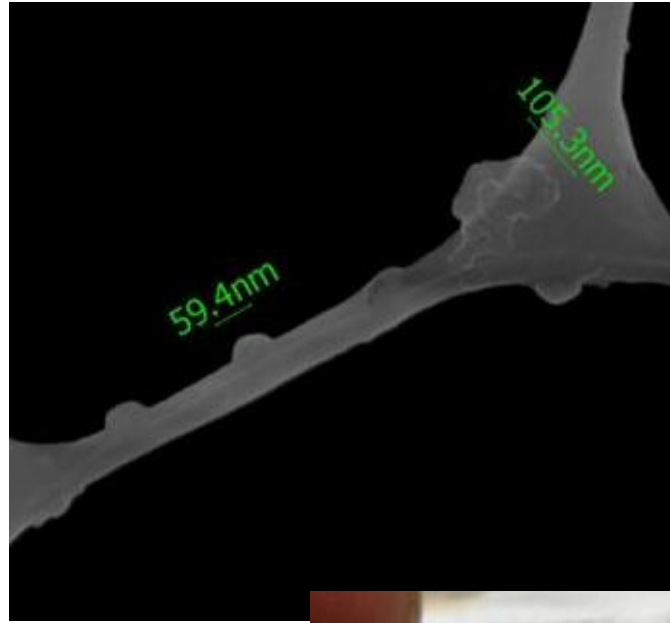
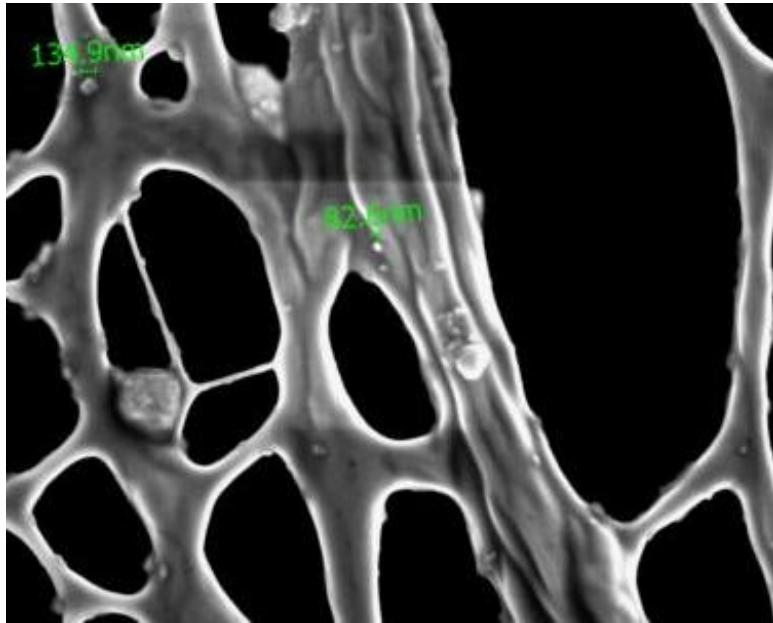
**N1s** Region XPS of Boron milled with Et<sub>3</sub>N in EtOH

# Combustion testing in a turbulent flame calorimeter

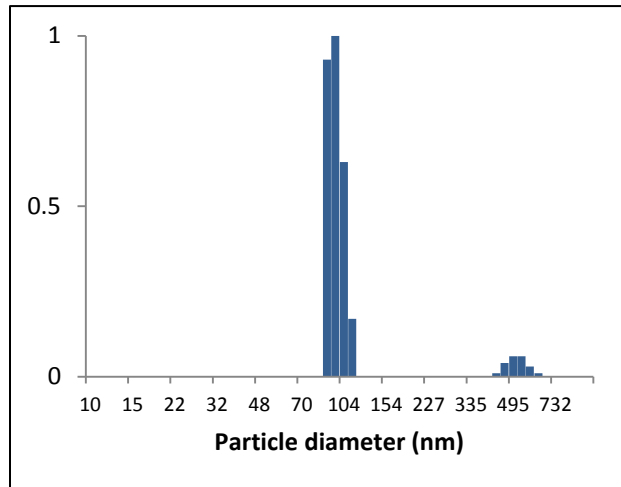
## ~3% Boron Loaded Ethanol vs. Pure Ethanol



# Boron nanoparticles functionalized with ionic liquid



Dynamic Light Scattering (DLS) of particles suspended in EtOH



# Acknowledgment

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