



U.S. Army Research, Development and Engineering Command



TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

Nanotechnology Capabilities at Picatinny Arsenal

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Report Documentation Page

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- Background Information
- Nanomaterials
- Picatinny's Production Capabilities
 - Bottom Up
 - Top Down
- Picatinny's Characterization Capabilities

Powder technologies are an integral part of Picatinny's Mission





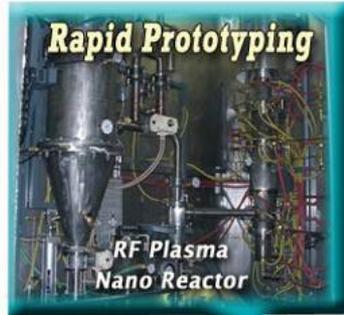
Picatinny Arsenal

- “Home of American Firepower”
- Baldrige Award Winner
- Improving legacy items
- Developing new items
- Prototyping capability
- Fill technology gaps





Nanotechnology Facility



Nanoparticle Reactor Facility at Picatinny Arsenal

Next generation materials for both military and commercial products



North America's largest RF Plasma facility for high performance nanomaterials



An integrated state-of-the-art facility to synthesize, process, and characterize nanophase and nanostructured materials, fully dense near-net shape bulk components, and nanostructured coatings

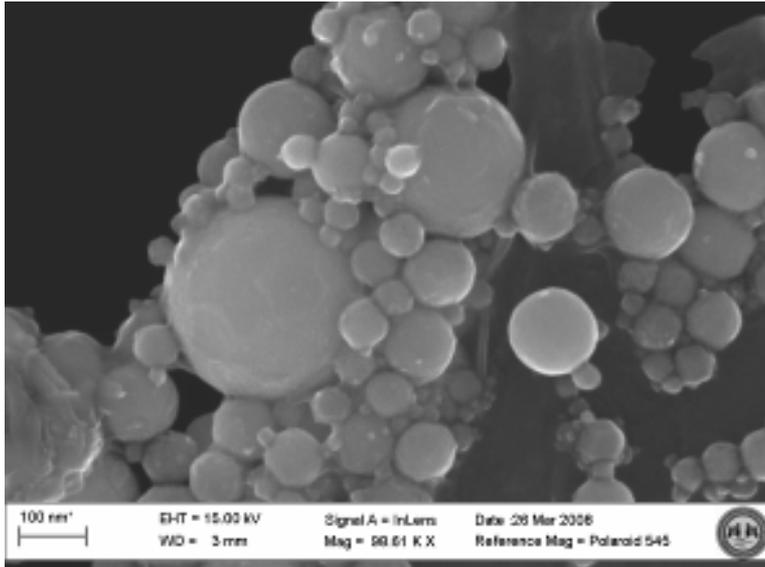


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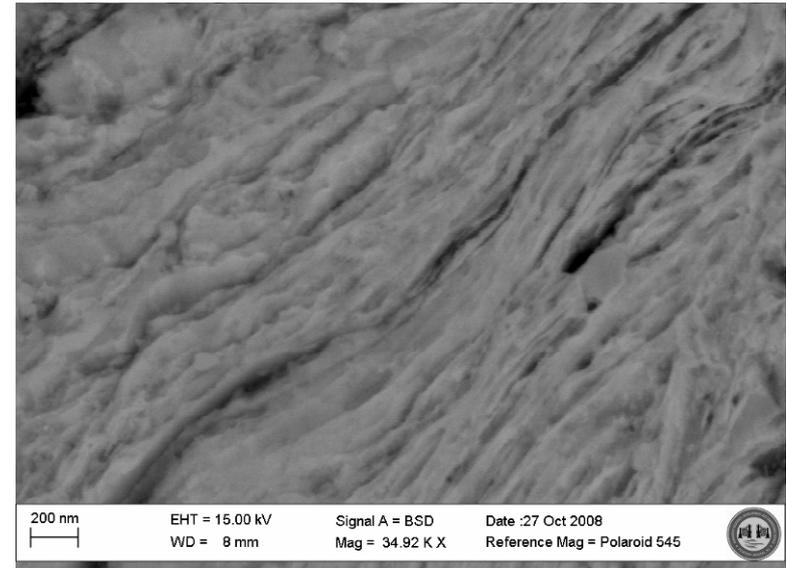
- **Nanophase Materials**

- Materials having nanoscale primary particle size



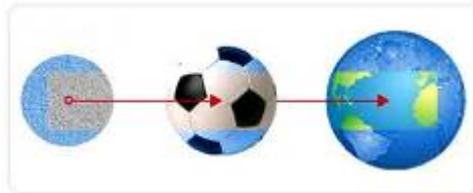
- **Nanostructured Materials**

- Materials which are not necessarily nanoscale but possess features which are on the nanoscale.





- Nanomaterial – a material having at least one dimension in the 1-100nm range
- Thickness of paper = 100,000nm
- Thickness of a human hair = 50,000nm
- Comparing a nanometer to a meter is like comparing a soccer ball to the earth
- There are 25,400,000 nanometers in 1 inch



Nanotechnology is a natural extension of particulate technology.



- What happens on the nano scale

- Tunable properties



Particle morphology

- Higher strength

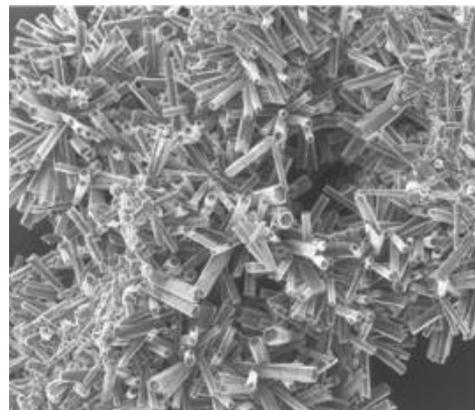
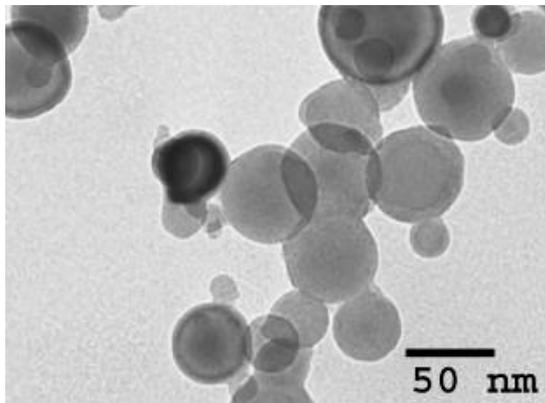
- Reduced Weight

- Increased reactivity



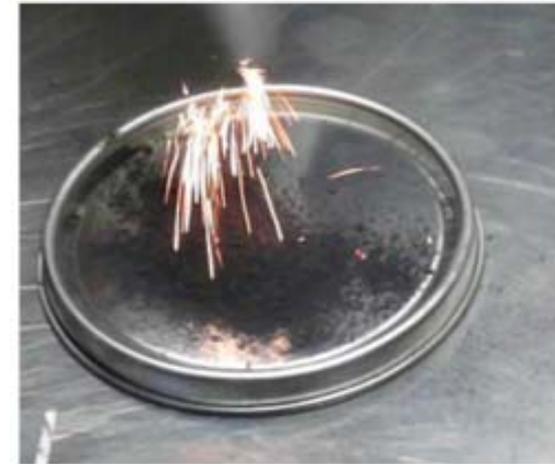
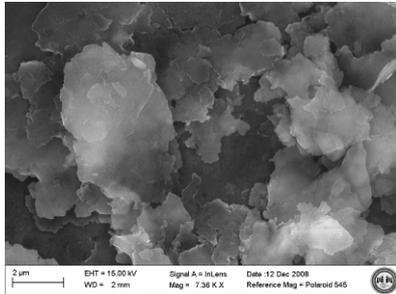
Increased surface area

- Increased durability





- Take conventional anti-corrosion techniques and scale down to the nano level
- Multi functionality as well as improved functionality
- Traditional properties may no longer apply between bulk and nanoscale
 - Materials become transparent
 - Materials become pyrophoric

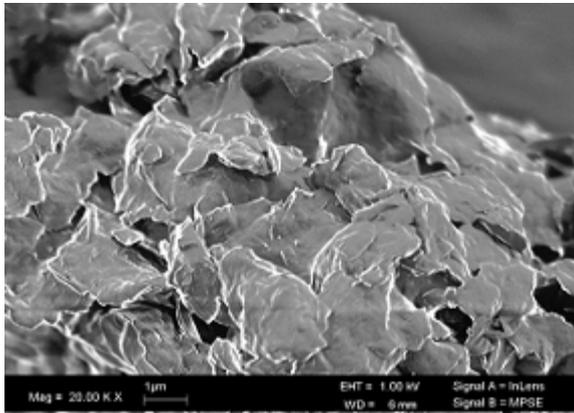


- **Surface modifications, protection, and functional coatings utilize powders and particulate technology**

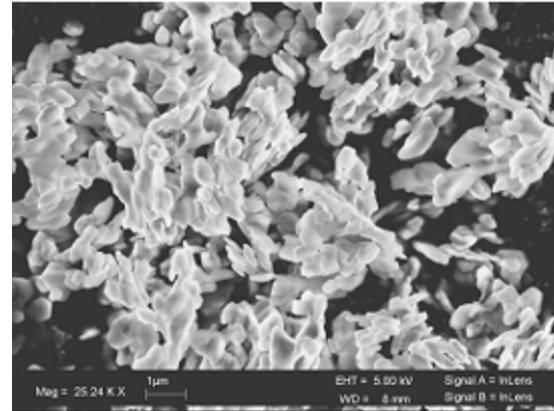




Results after accelerated corrosion testing



**Montmorillonite (Cloisite 15A)
/ 5% Bromothymol Blue**



**Hydrotalcite / 5%
Thymophthalein**





- Bottom Up Approach



- Top Down Approach



Using thermal plasma technology to synthesize nanomaterials is at the heart of our prototyping facility.





Inductively Coupled Plasma

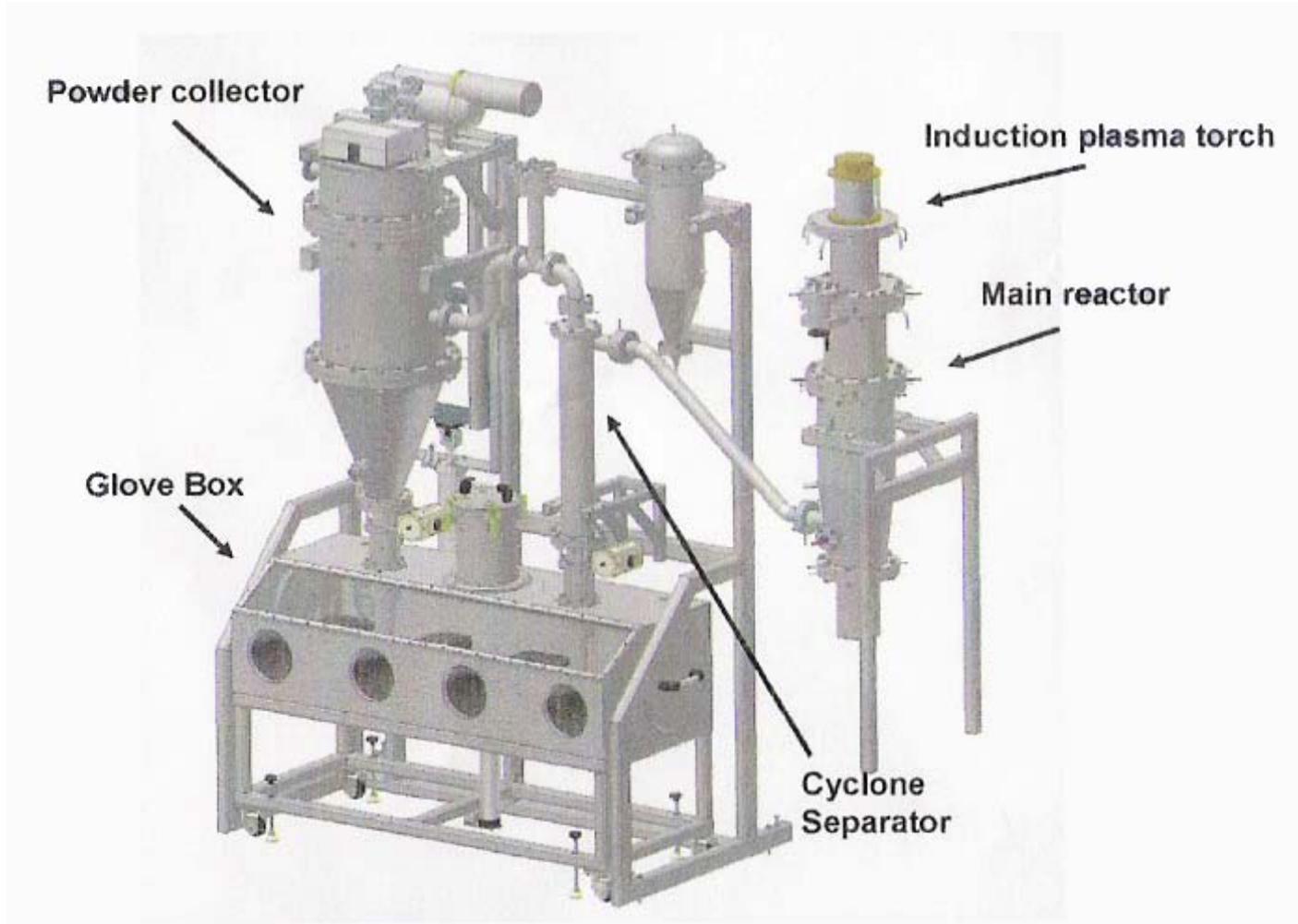


- Induction Plasma is a versatile and high rate technology to synthesize nano scale powders



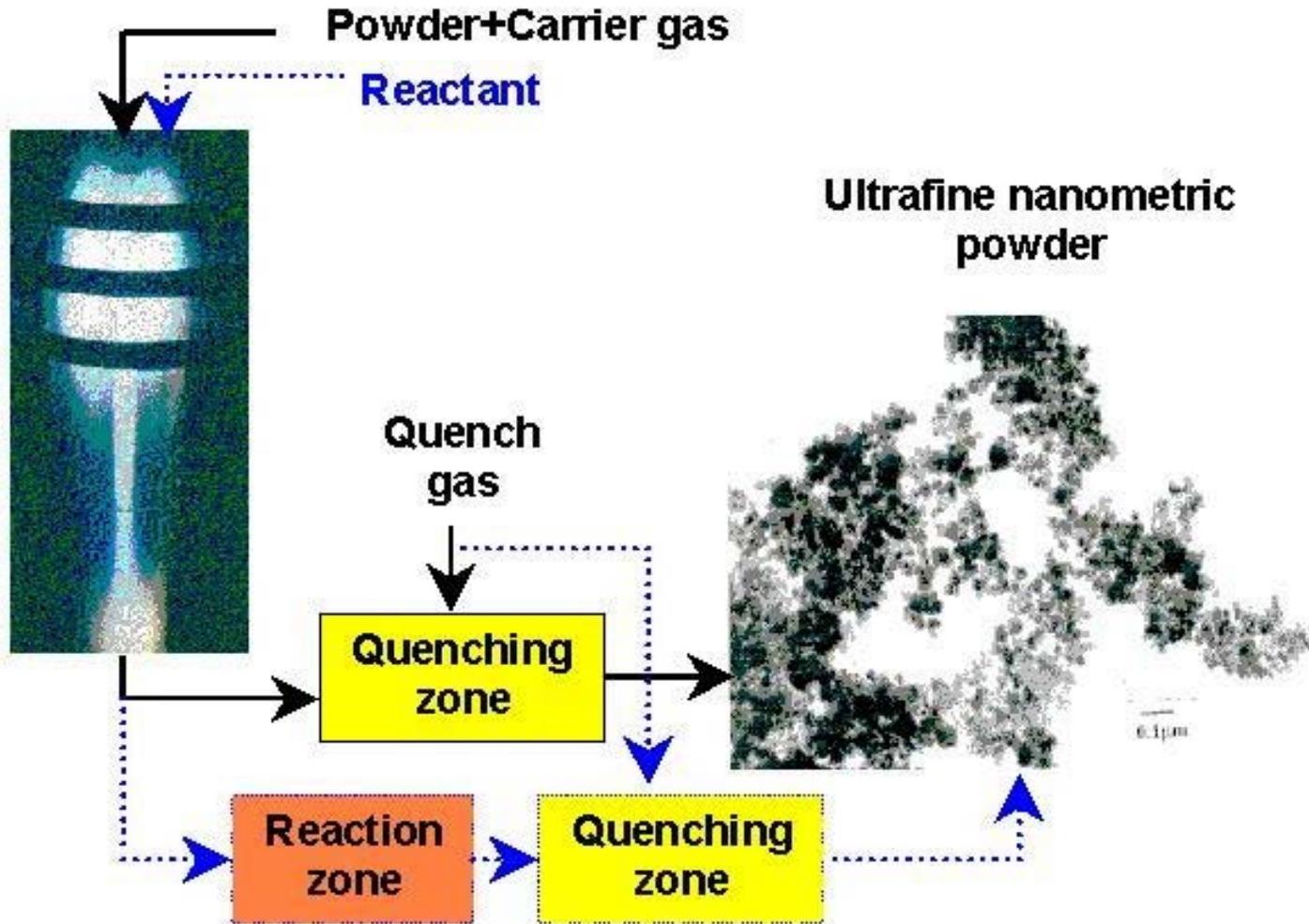


Reactor Layout





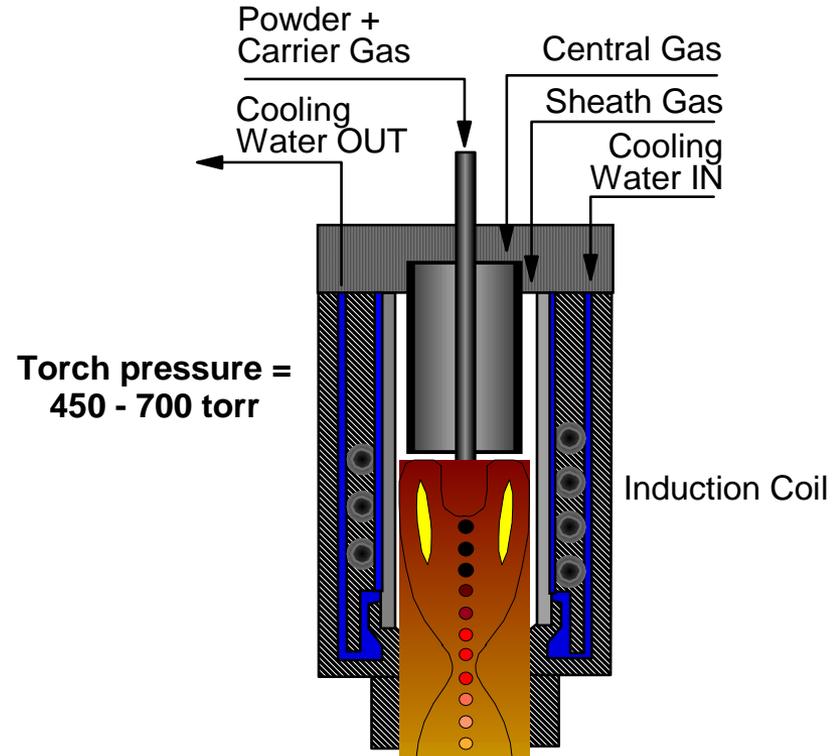
Plasma Theory





- Plasma Technology Benefits

- Powder or liquid feed stock
- No limit on material type
 - Over 10,000k plasma temperature
- High purity production
- Flexibility of operating conditions



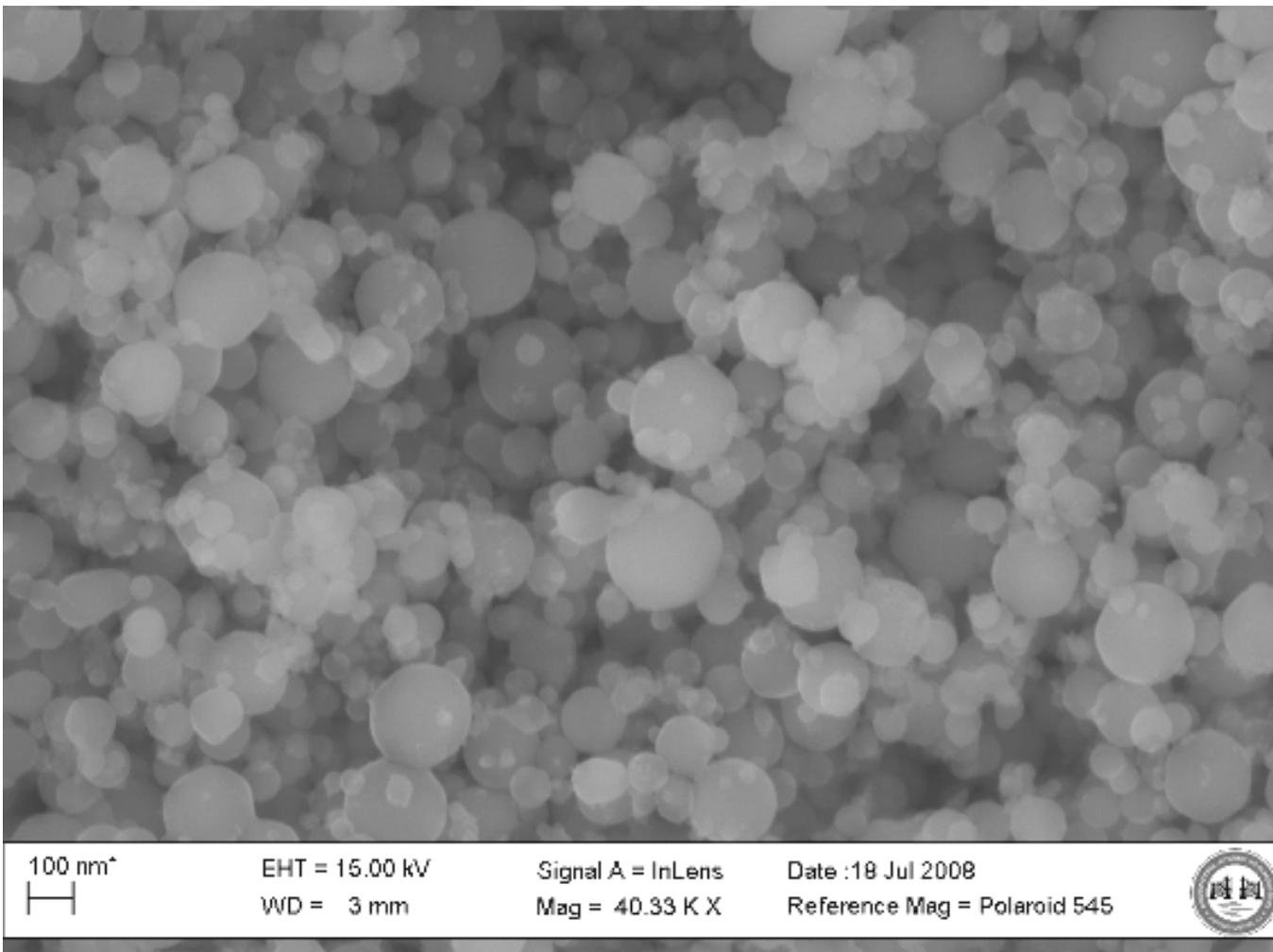


- Two Plasma Reactors
 - One unit for metals only
 - One unit for ceramics and non oxides
 - Production rate: up to 1kg/hr
- Some nanoscale powder examples
 - Aluminum
 - Tungsten
 - Cerium Oxide
 - Boron Carbide
 - Iron



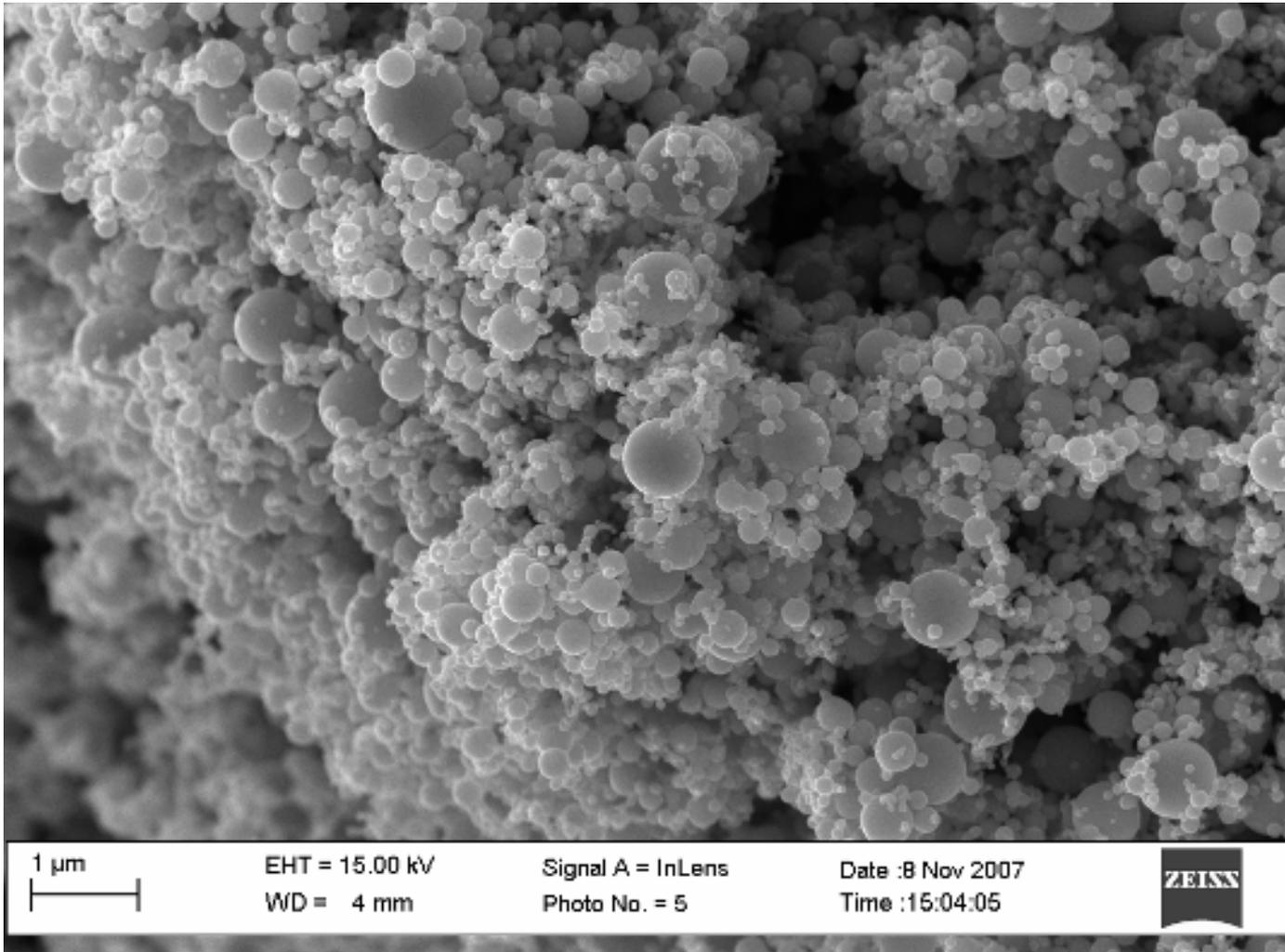


Nano Aluminum



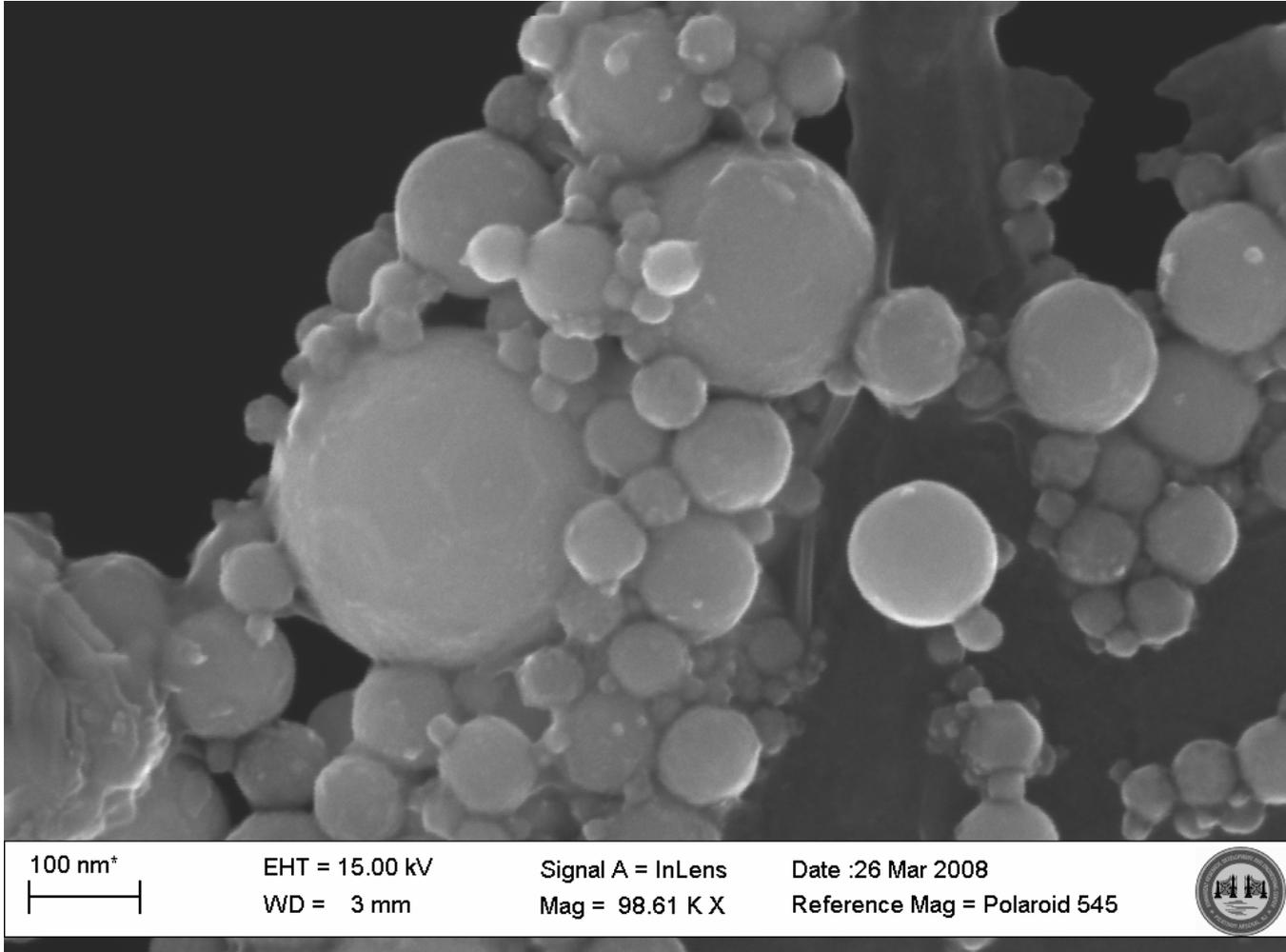


Nano Iron



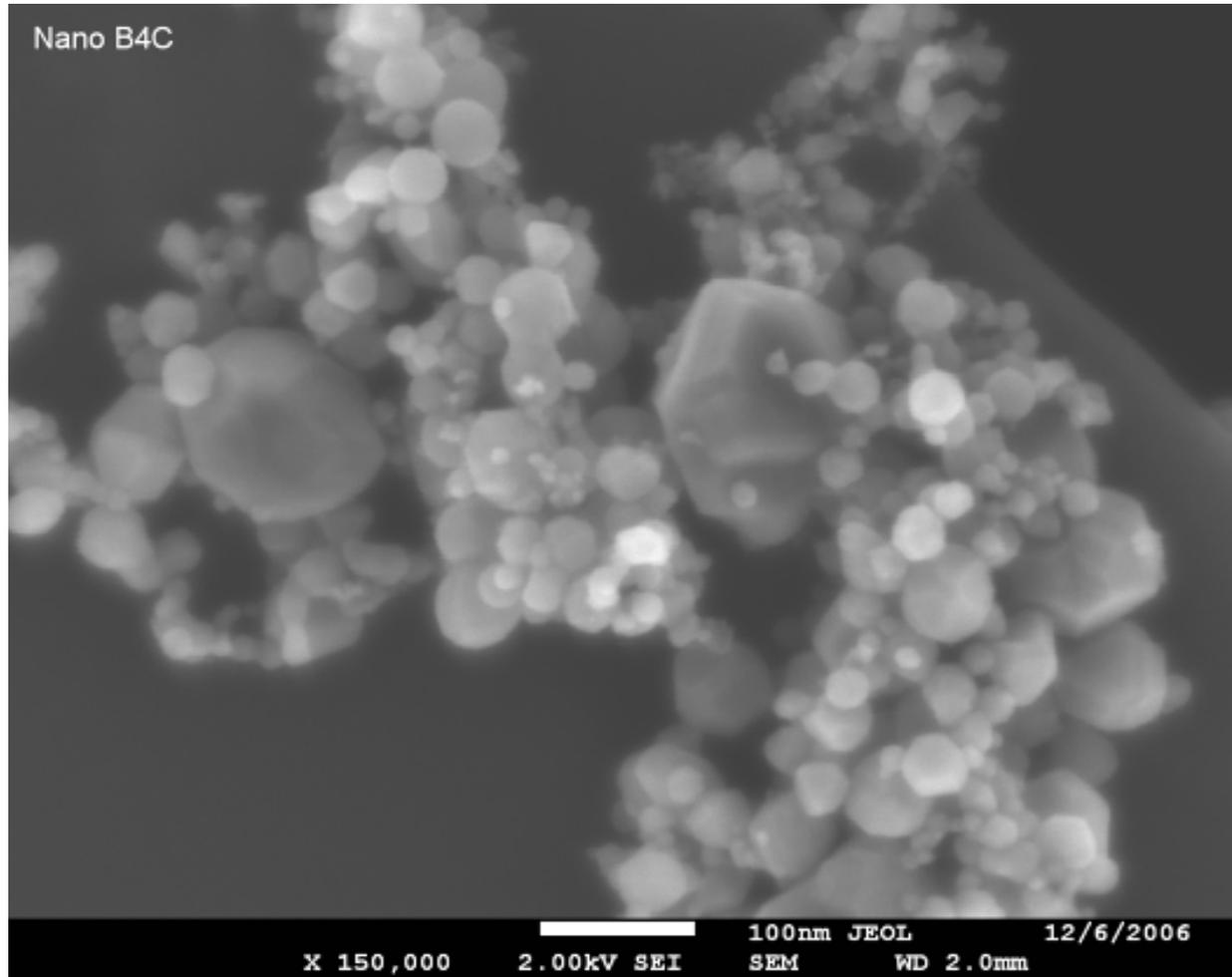


Nano Tungsten





Nano Boron Carbide





- High energy milling is a top down approach to fabricate nanostructured metals, alloys, ceramics, cermets, and reactive materials.





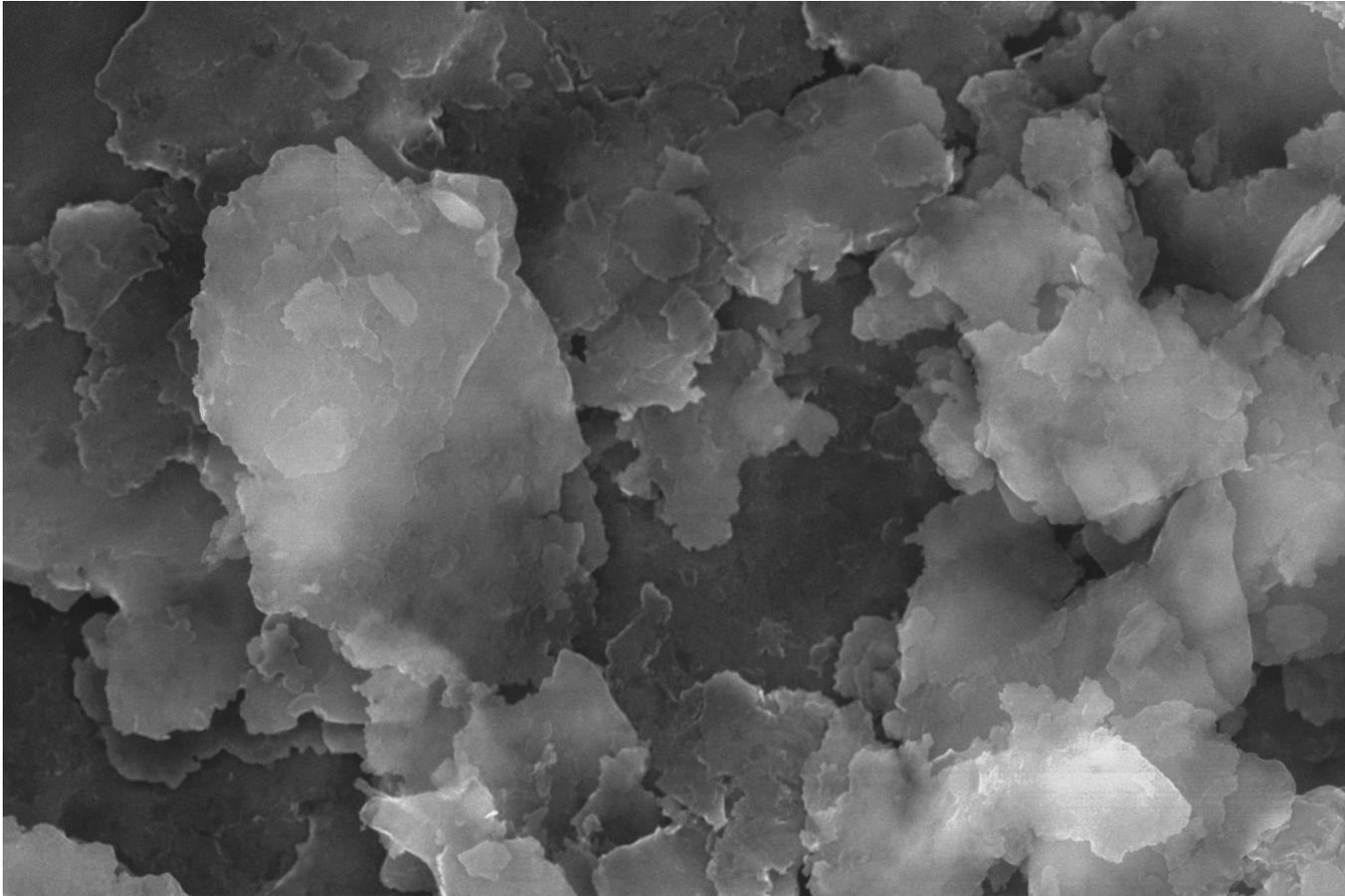
- Inert atmosphere for reactive materials
- Semi-continuous prototyping capability
- Experimental batch capability
- Tailor made compositions
- Three total units
 - Two (2) one liter machines
 - One (1) 8 liter machine



- **Able to impart nano sized grains into micron sized powders**



Nanostructured Aluminum Flakes



2 μ m



EHT = 15.00 kV

WD = 2 mm

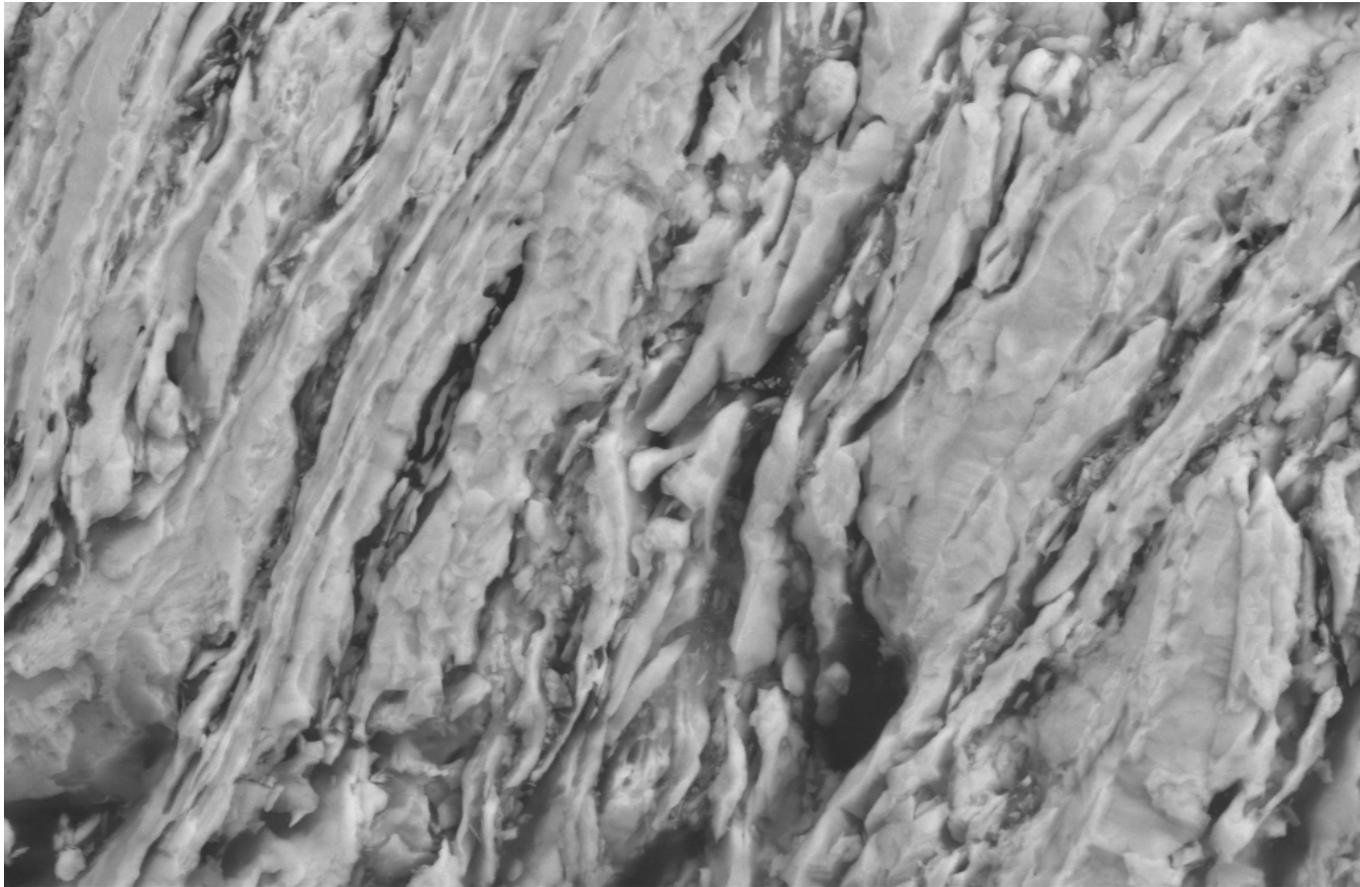
Signal A = InLens

Mag = 7.36 K X

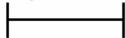
Date :12 Dec 2008

Reference Mag = Polaroid 545





1 μ m



EHT = 15.00 kV

WD = 6 mm

Signal A = BSD

Mag = 9.74 K X

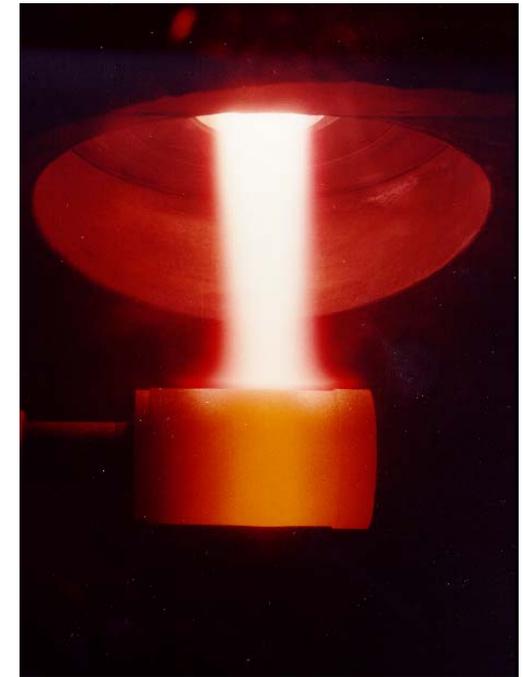
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Reference Mag = Polaroid 545





- Coatings can be applied via liquid or vapor deposition
- VPSD (Vacuum Plasma Spray Deposition)
- Can be used to net shape bulk products or apply coating layers



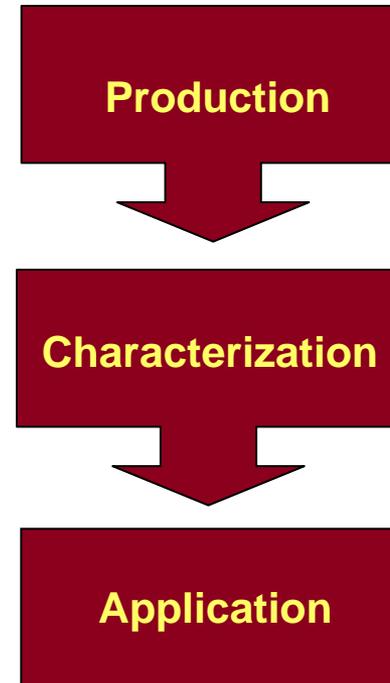


- Current equipment:
 - Field Emission Scanning Electron Microscope
 - X-ray Fluorescence
 - X-ray Diffractometer
 - Small angle X-ray scattering & Ultra small angle X-ray scattering
 - Thermal analysis equipment





- Particle size and distribution
- Composition
- Morphology
- Surface Area
- Thermal Properties
- Passivation layer thickness
- Crystallite size
- Phase identification



- Picatinny's facilities allow for rapid characterization of materials as soon as they are made.



- Zeiss Gemini Ultra Variable Pressure FE-SEM with EDX
 - Capable of low voltage
 - VP mode
 - STEM attachment
 - 1nm resolution @ 20kV
 - 12-1000000x magnification
 - High efficiency in-lens
 - Secondary and Backscatter
 - Used to determine:
 - Particle size
 - Distribution
 - Morphology
 - Passivation layer
 - Composition





- Rigaku ZSX Primus II X-ray Fluorescence
 - Capable of holding up to 64 samples
 - Quantitative analysis results
 - Composition determination
 - Solid, liquid, or powder samples





XRD Unit



SAXS



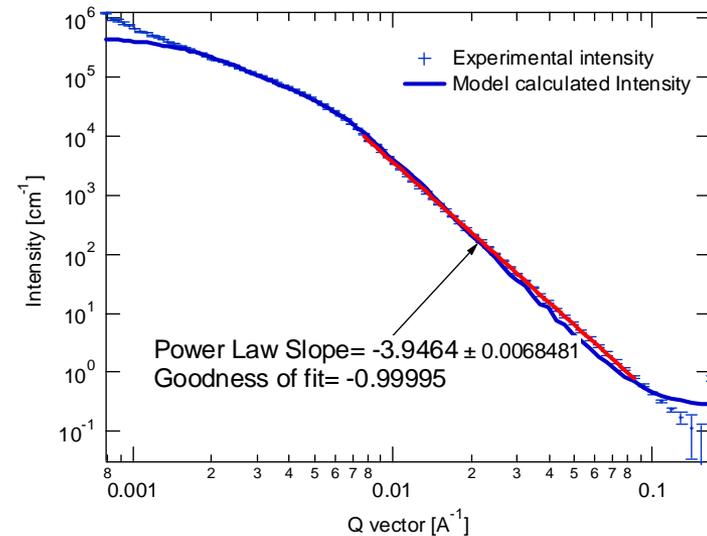
USAXS

- Rigaku Ultima XRD
 - Determines phase and crystallite size
 - Bulk sample holder
 - Hot stage
 - 6 ring powder sample holder





- Combining Small Angle and Ultra-small Angle X-ray Scattering allows *simultaneously* characterizing
 - Primary, secondary particle size & particle morphology
 - User friendly software developed
 - Total length scale, 1 nm – 2 μm



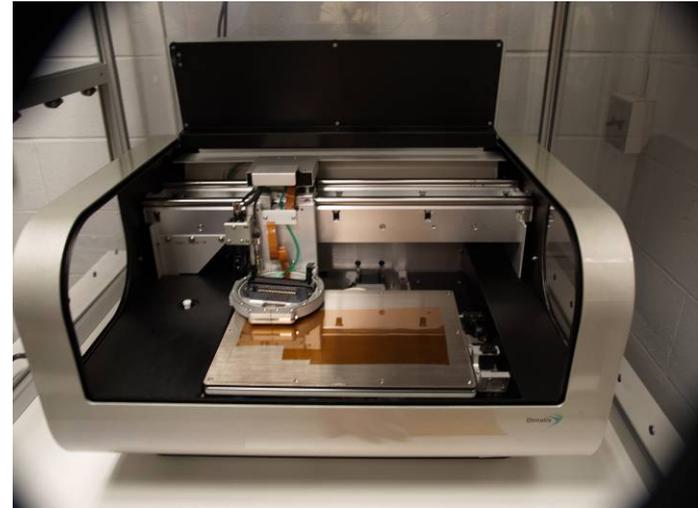


- Netzsch STA 449 C Jupiter – QMS 403 Aeolos Integrated TG-DSC / Mass Spec
 - Thermal properties
 - Oxidation
 - Exotherms
 - Endotherms
 - Melting Point
 - Moisture content
 - Organics content
 - Aging characteristics
 - Passivation layer thickness





- Nano ink printer
- Low voltage electron microscope
- Optical Microscopes
- Polishing and grinding units
- Spray dryer
- BET
 - Surface area
- Glow Discharge
 - Composition
- Oxygen analyzer





- **Powder Production Capability**

- Able to produce nanostructured materials with enhanced properties using high energy milling.

- Able to produce almost any nano scale powder at a rate up to 1kg/hr for potential applications involving anti-corrosion technologies.

- **Materials Characterization Capability**

- Collaboration also available for corrosion mechanism identification

- Able to obtain vast amounts of information about bulk materials or nanomaterials in a short time.





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