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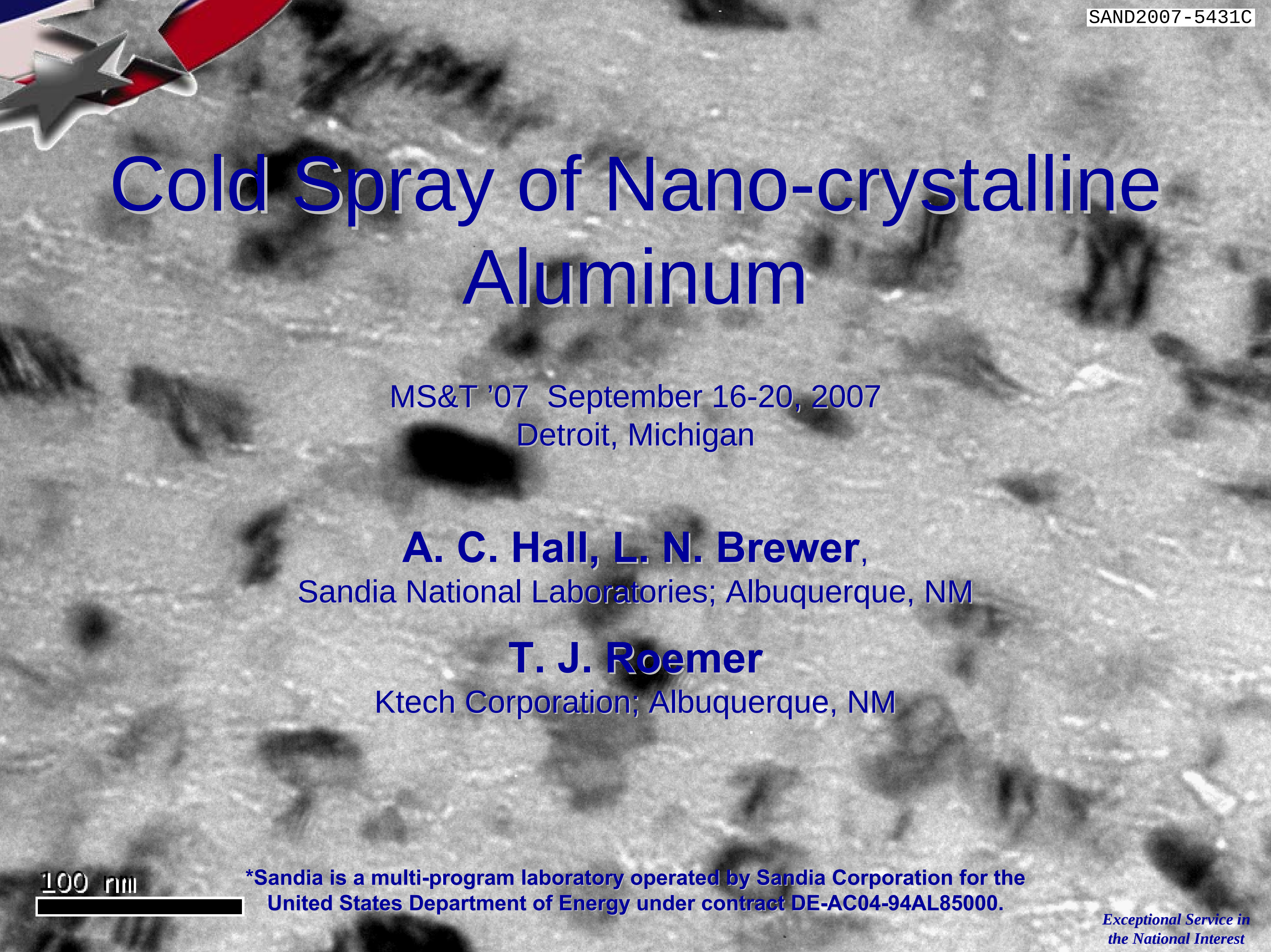

Cold Spray of Nano-crystalline Aluminum

MS&T '07 September 16-20, 2007
Detroit, Michigan

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Sandia National Laboratories; Albuquerque, NM

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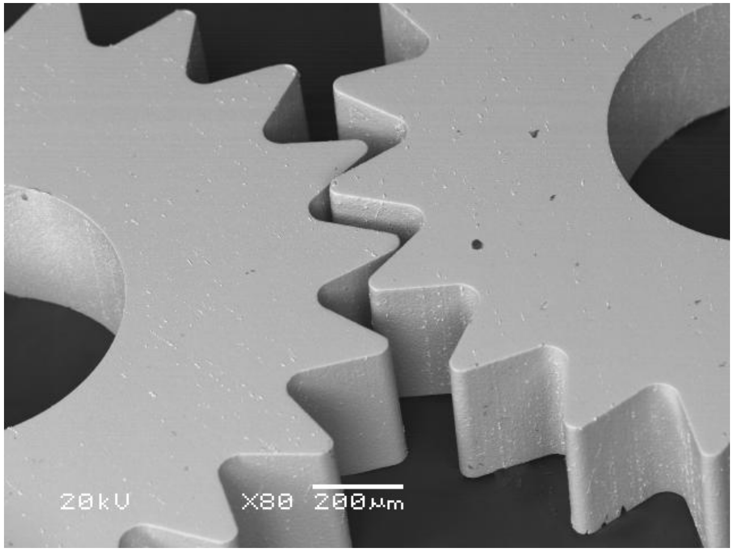
Ktech Corporation; Albuquerque, NM

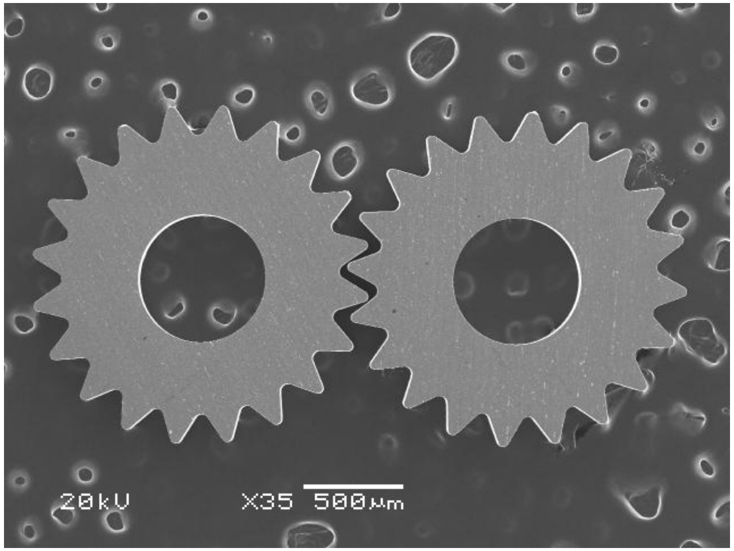
***Sandia is a multi-program laboratory operated by Sandia Corporation for the United States Department of Energy under contract DE-AC04-94AL85000.**

Exceptional Service in the National Interest

Sandia wants nanocrystalline material for fabrication of mesoscale parts.

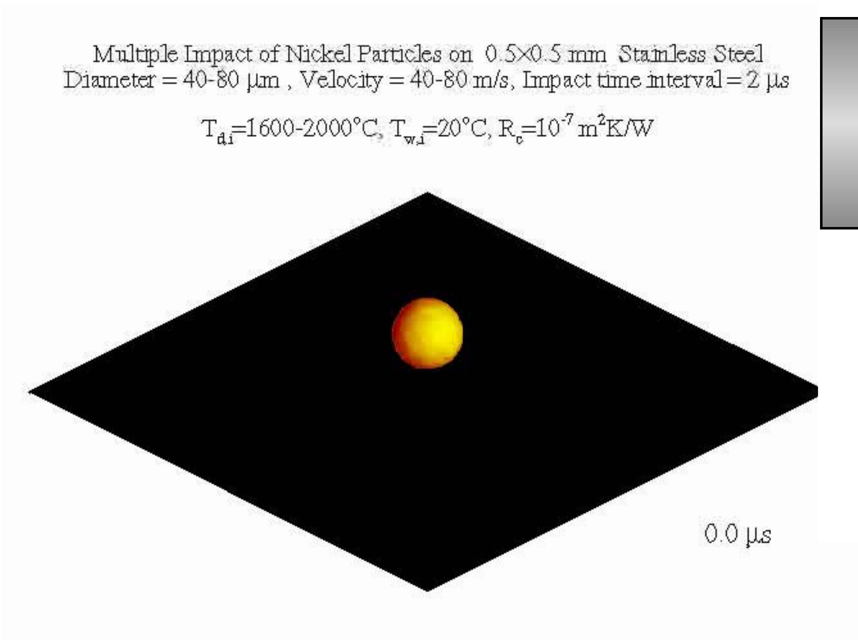
- Consolidation of nanocrystalline powders is challenging but necessary
- Sandia National Laboratories is interested in consolidation of nanocrystal materials for fabrication of meso-scale machinery
- Small components dimensions require nanocrystalline materials

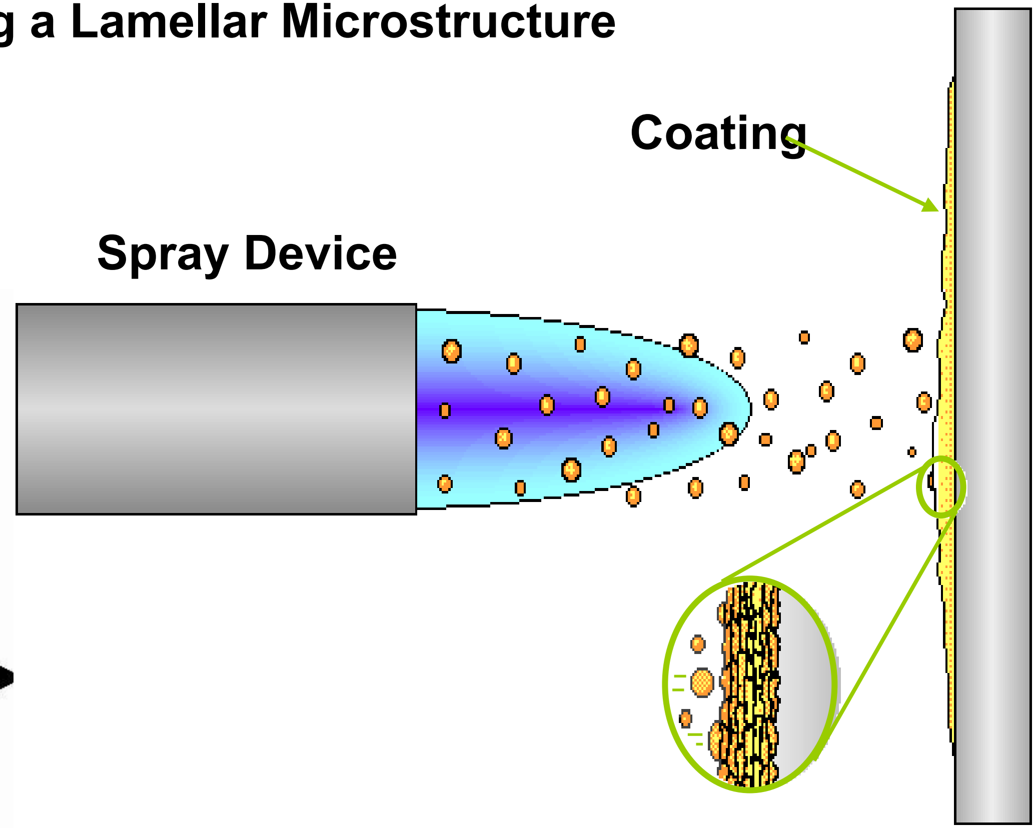




What is Thermal Spray?

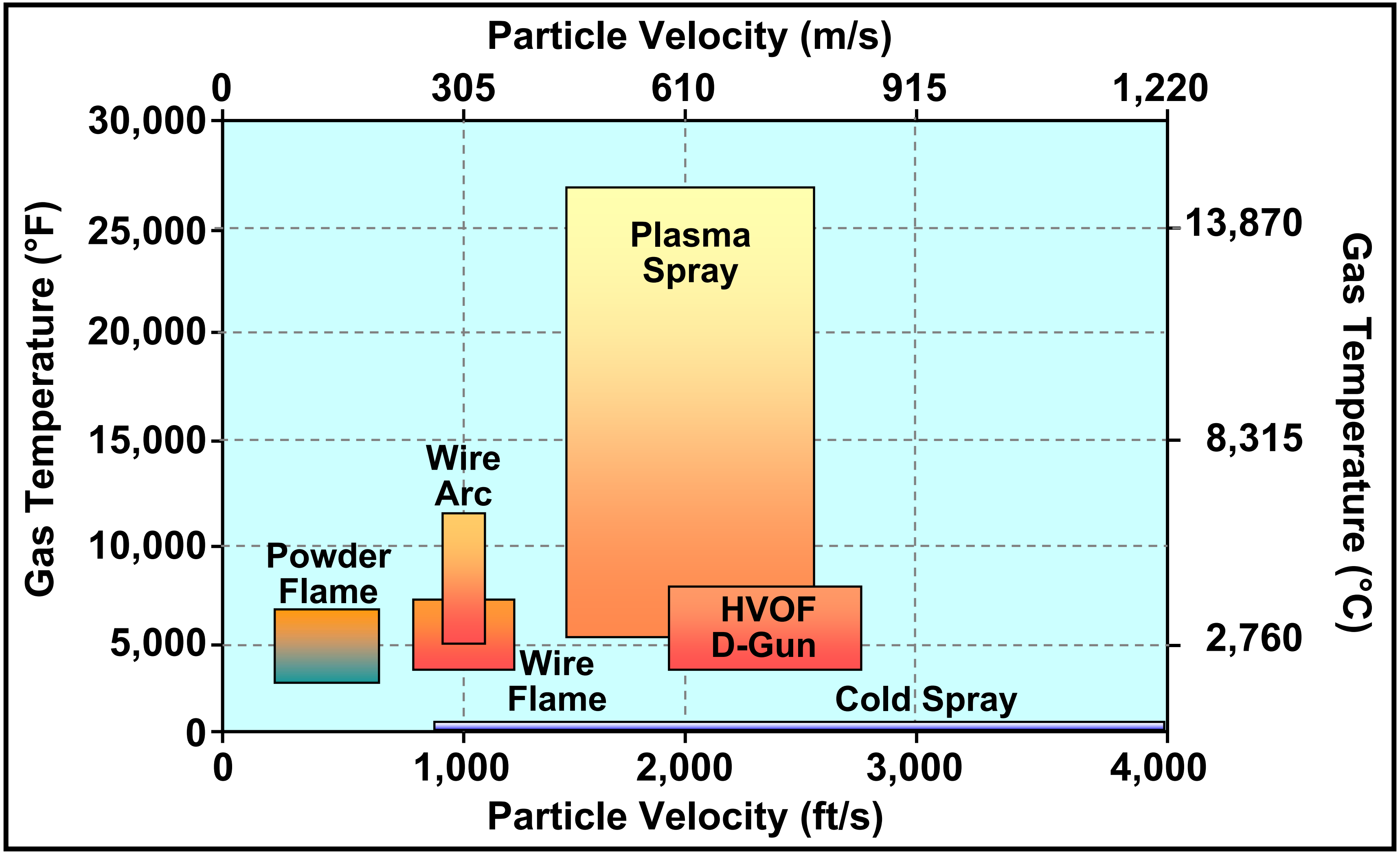
~ 10 - 100 µm Molten or Semi-Molten Droplets are Sprayed onto a Target Surface Where they “Splat” Cool at Rates up to 10^4 -10^8 K/sec Forming a Lamellar Microstructure





*Droplet Impact Simulation by Prof. J. Mostaghimi, et al, Univ. of Toronto, 1998.

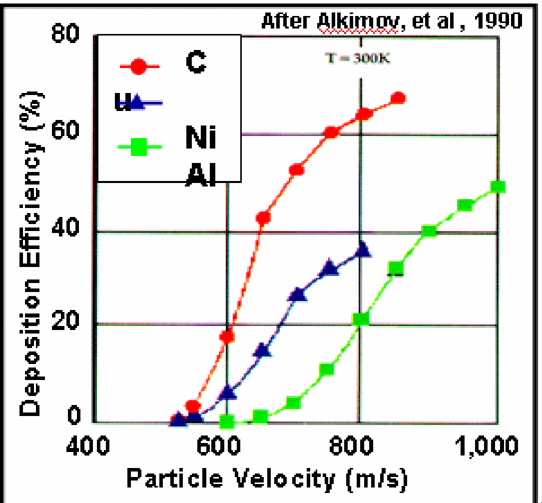
Spray Process Comparison*

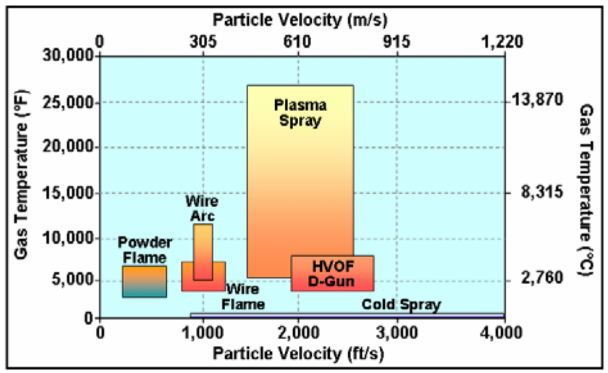


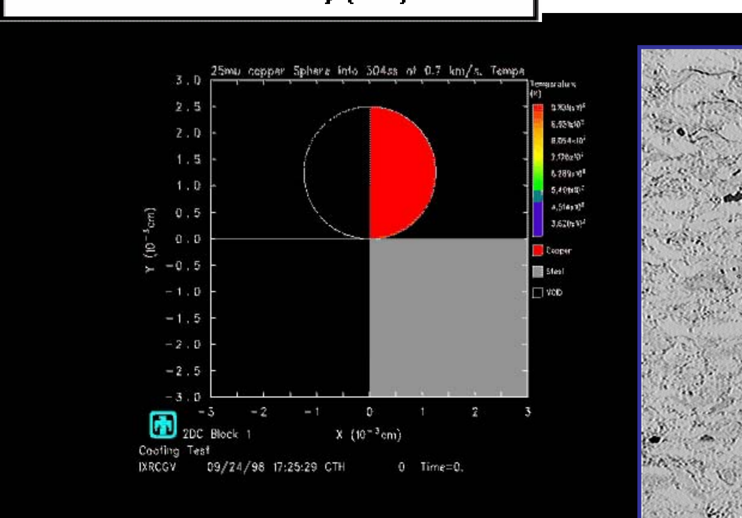
*Adapted from plots by R.C. McCune, Ford Motor Co. & A. Papyrin, Ktech Corp.

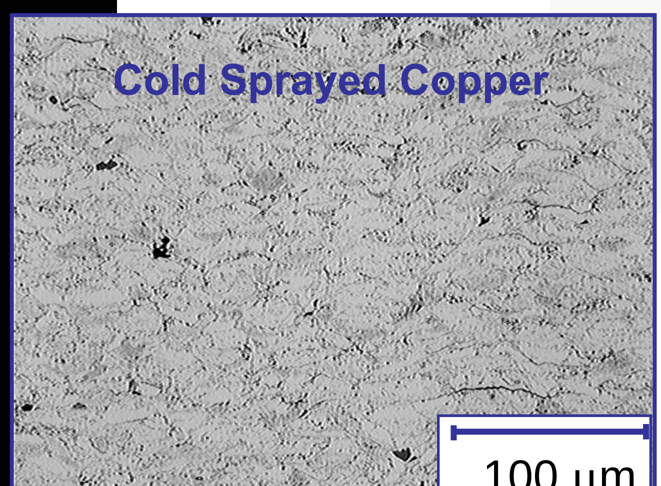
Cold Spray: A "Cold" Process from Siberia

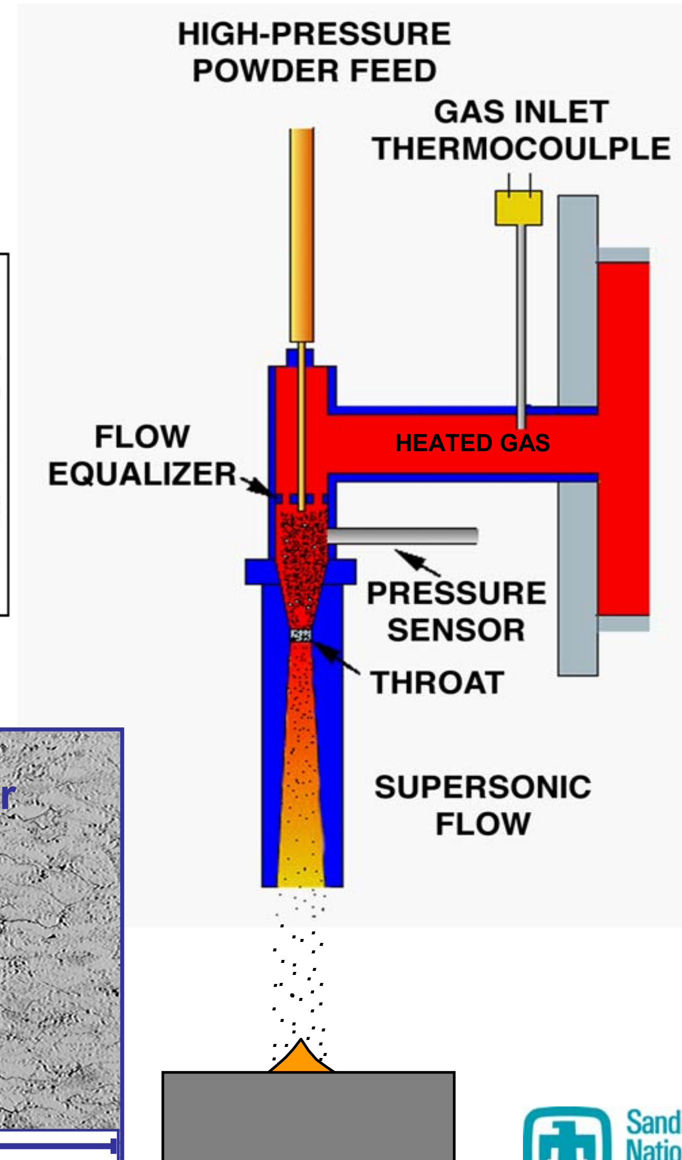
- High density metal coatings
- Low oxide content
- Compressive residual stress











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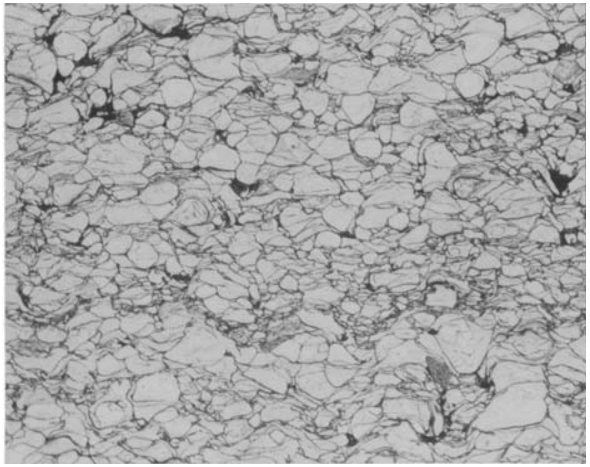
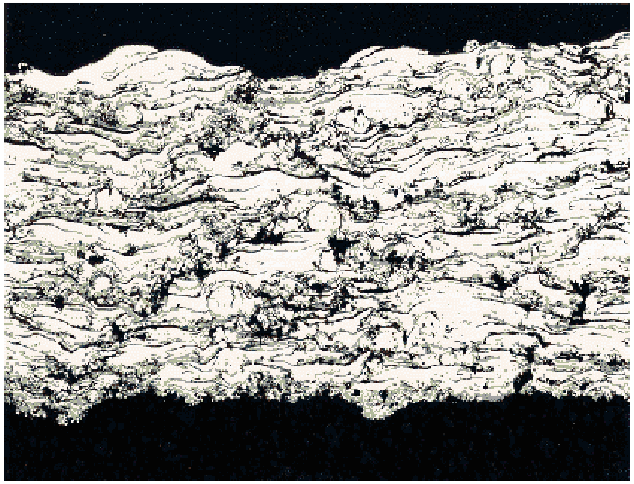
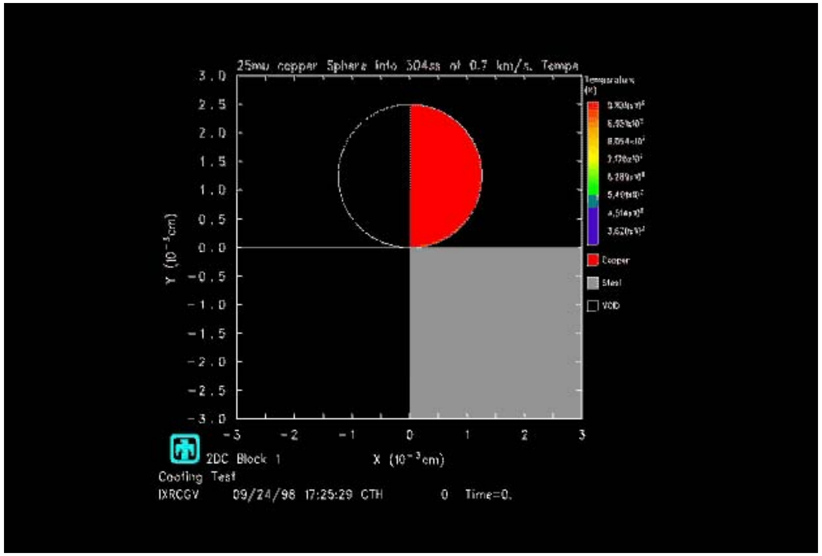
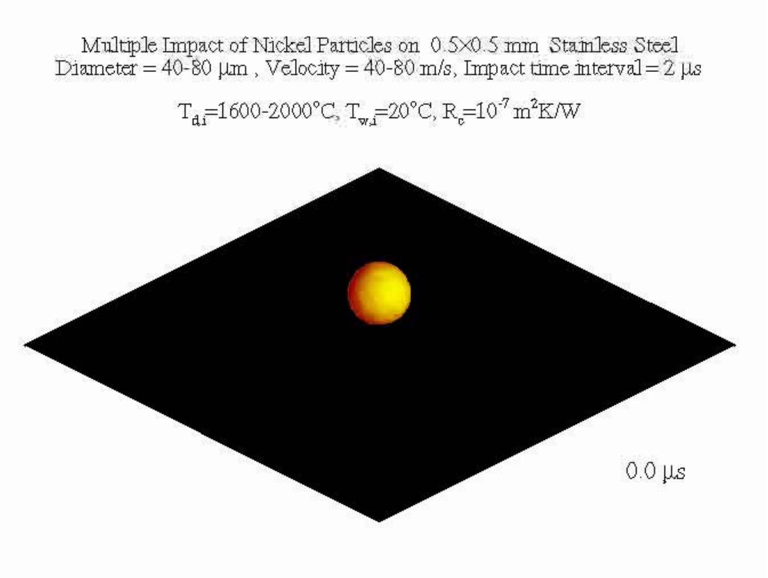
Cold Spray ≠ Thermal Spray









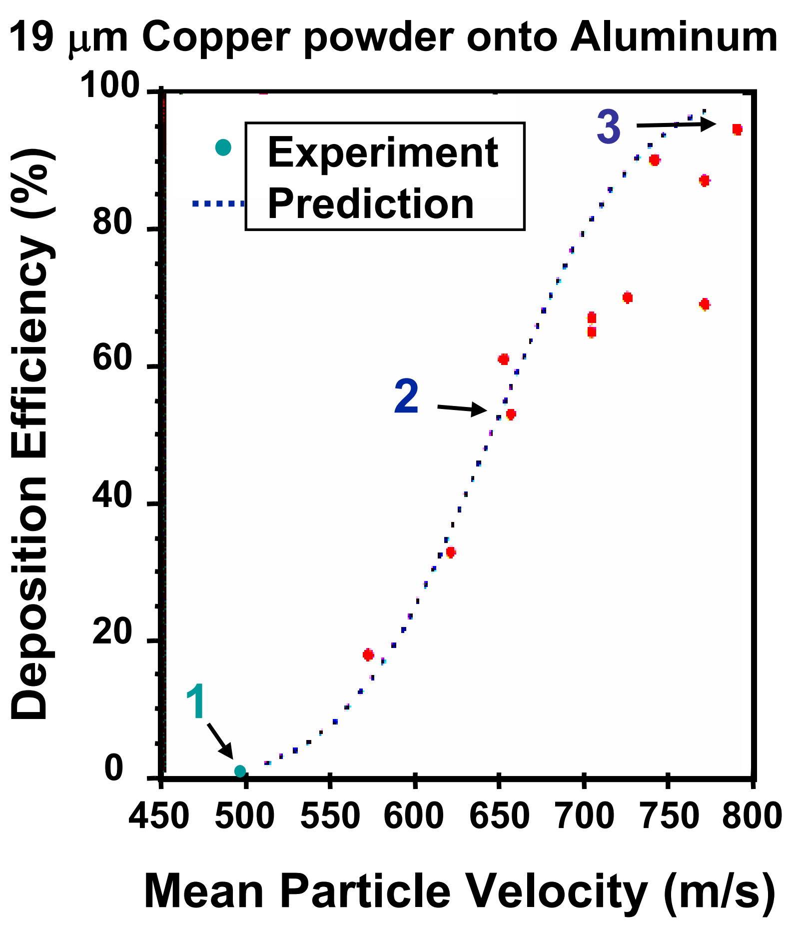
Solid particle impact

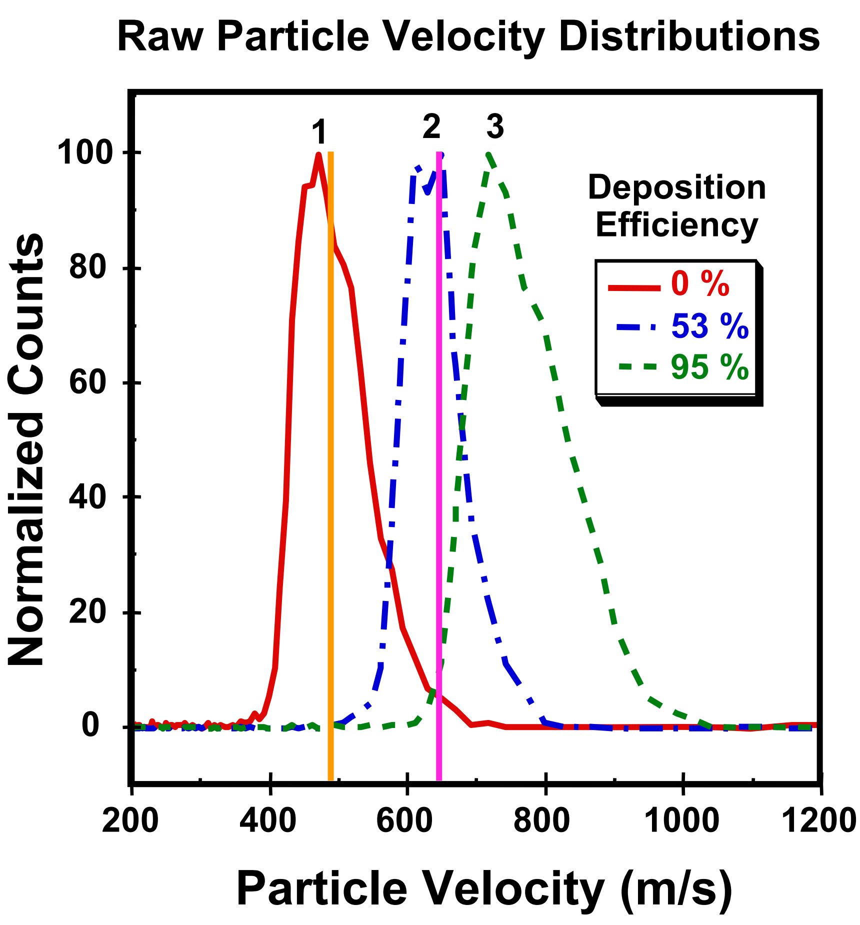
Liquid droplet impact

*Droplet Impact Simulation by Prof. J. Mostaghimi, et al, Univ. of Toronto, 1998.

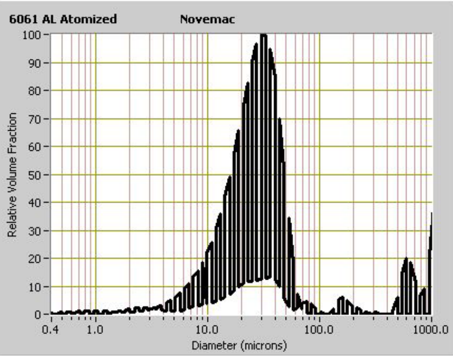
Understanding the cold spray process.

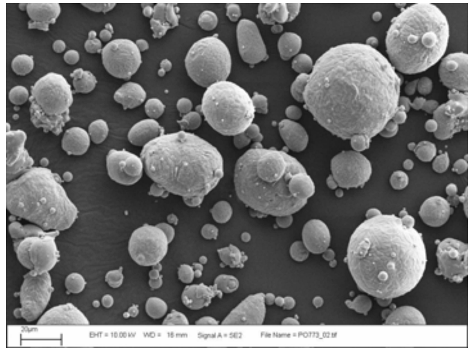
There exists a minimum critical velocity, V_{crit}, above which particles will adhere to the substrate and form a deposit.

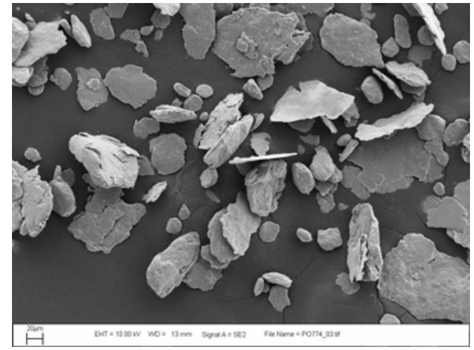


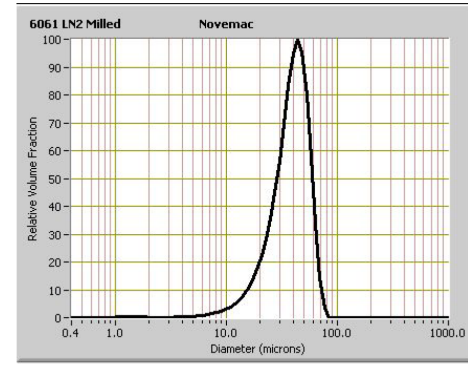


Spherical gas atomized 6061 & 5083 aluminum powders were LN_2 ball milled to create a feedstock with a nanocrystalline microstructure

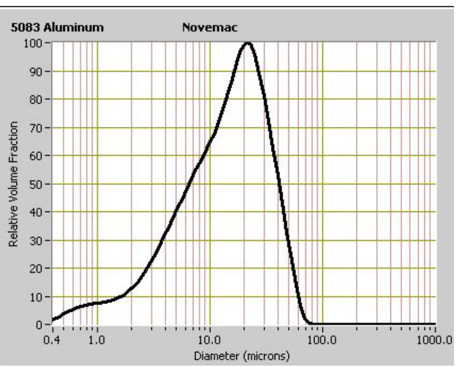


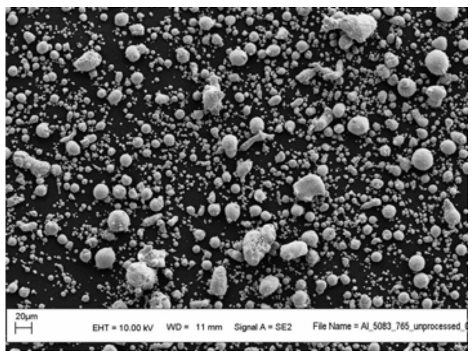


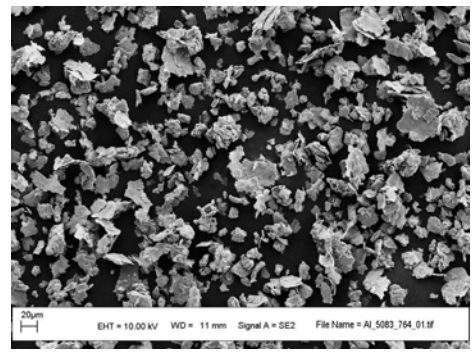


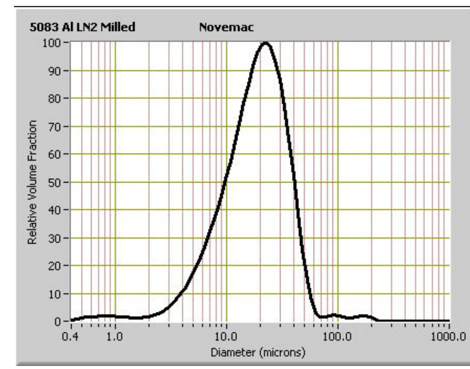


Gas Atomized (As-received) ⟵ ⟶ LN_2 Ball Milled



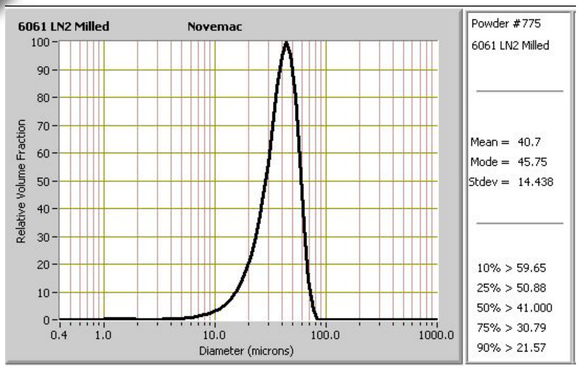


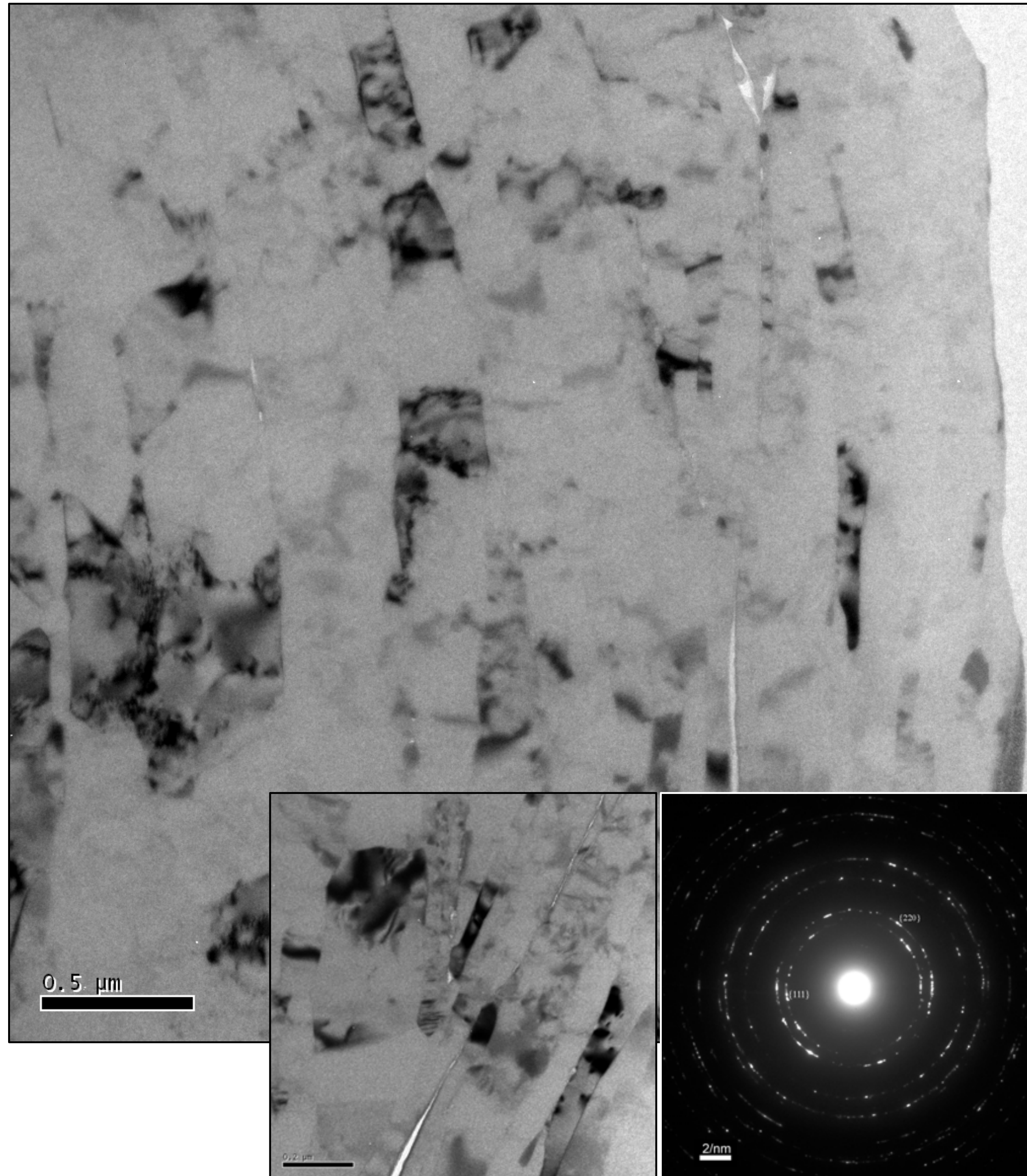




Powder / Condition	Mean Size	Standard Deviation	Morphology
6061 Al / Gas Atomized (As-received)	27.8	13.67	Spherical
6061 Al / Gas Atomized & LN2 Ball Milled	40.7	14.44	Platelet
5083 Al / Gas Atomized (As-received)	18.2	13.6	Spherical
5083 Al / Gas Atomized & LN2 Ball Milled	22.6	17.8	Platelet

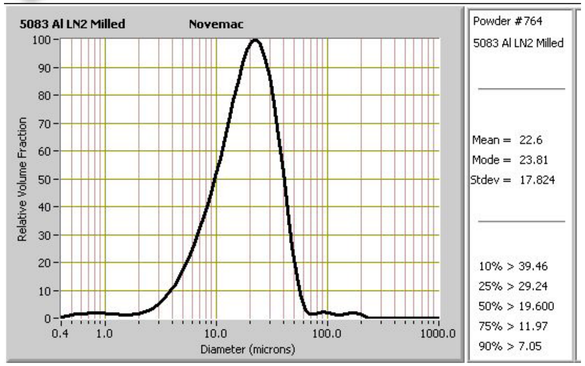
6061 Al powder contains 250-400 nm grains after LN_2 ball milling

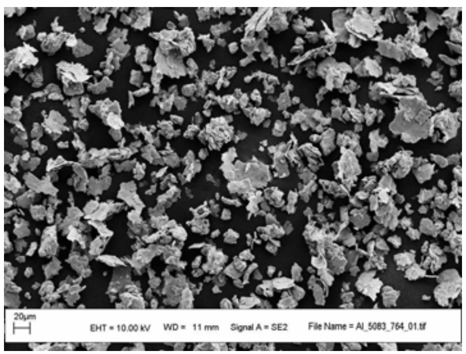




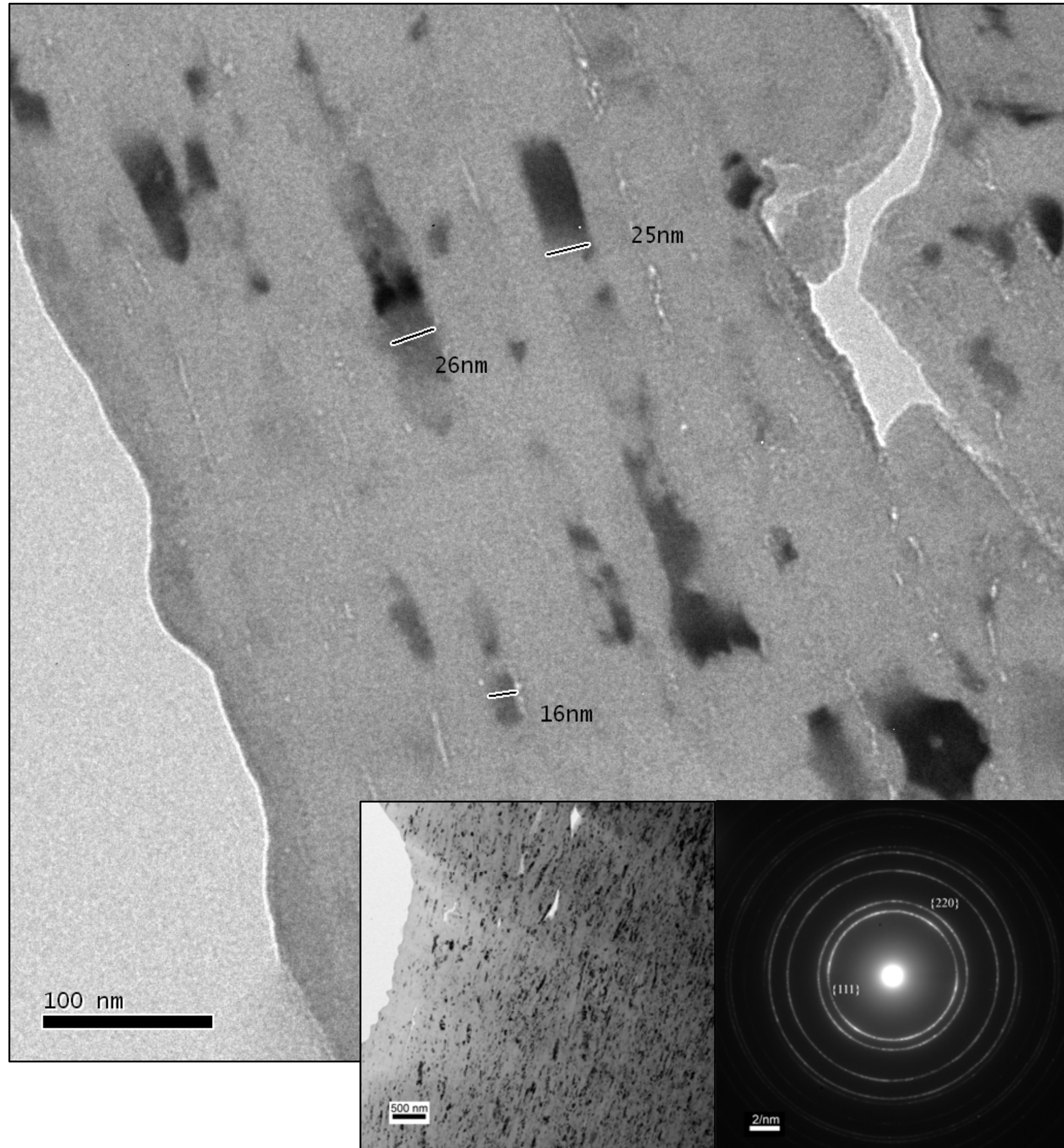
- 40 micron average particle size
- 14 micron standard deviation
- 250-400nm grains within particles (ultra-fine grain material)
- Lath-like grain structure w/ inter grain porosity,
- No strong crystallographic texture evident in SADP

5083 LN_2 ball milled Al microstructure similar to, but much finer than LN_2 milled 6061 Al

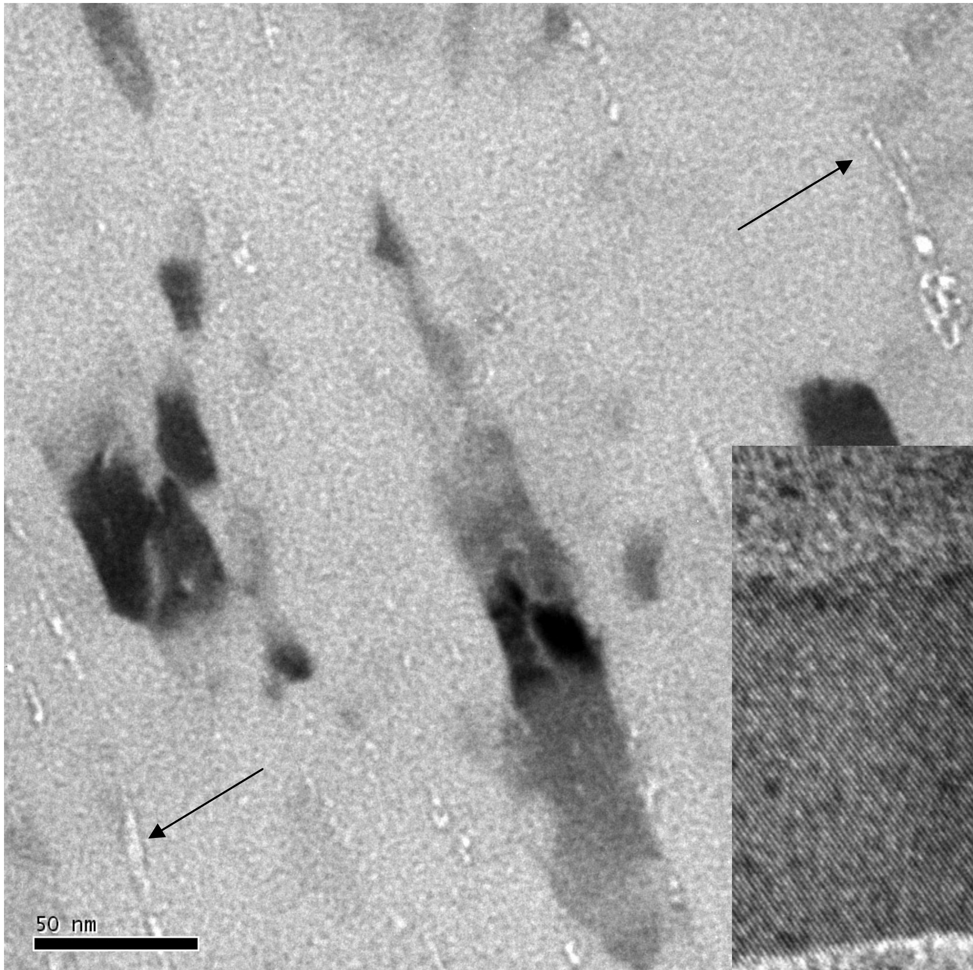




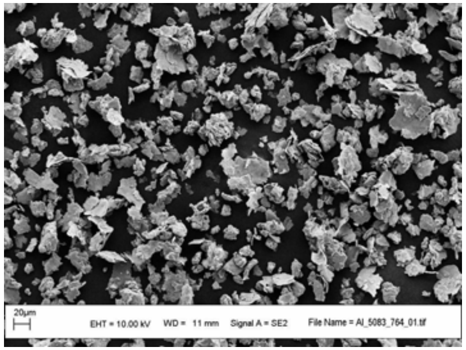
- 22 micron average particle size
- 17 micron standard deviation
- Microstructure comprised of elongated grains, parallel to surface of particle, with thicknesses <100nm
- Porosity visible, mostly at GB's
- SAD pattern shows ring pattern with very little texture (perhaps a slight amount for the {111} reflection)



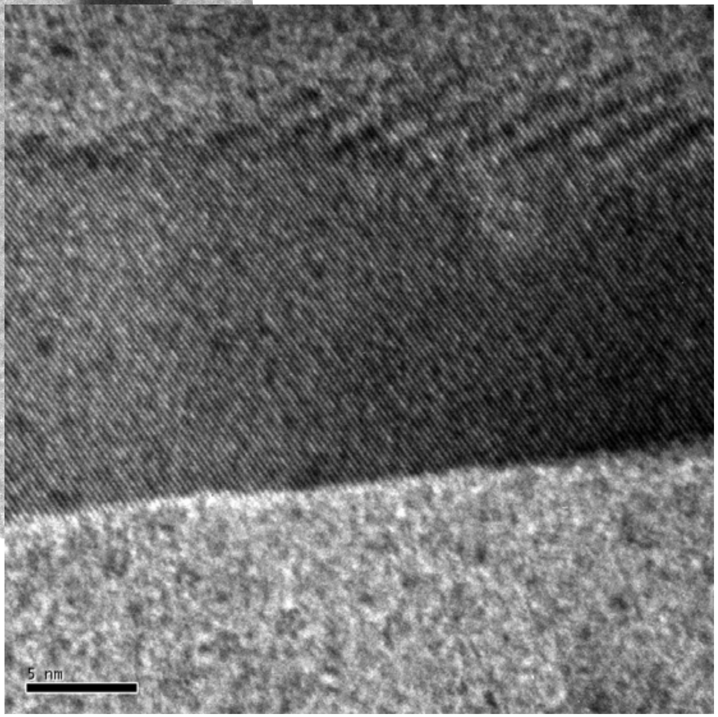
Under-Over focus (Fresnel contrast) series confirms GB porosity in LN_2 milled 5083 Al



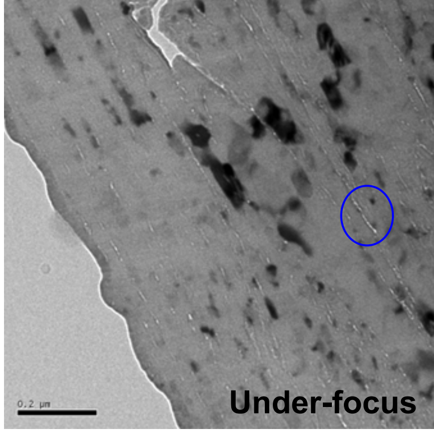
Bright field image of individual grains showing porosity at grain boundaries

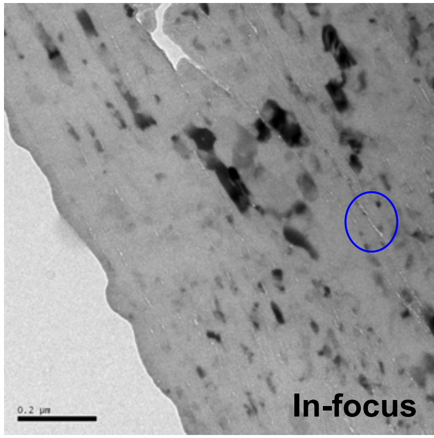


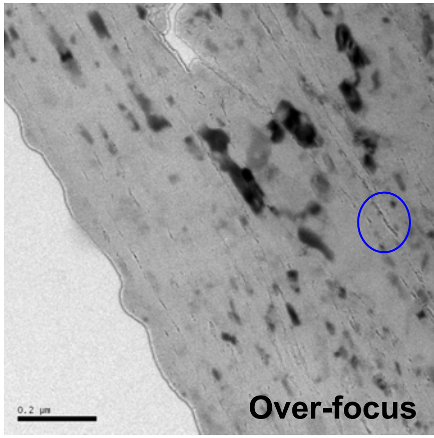
LN_2 Ball milled powder



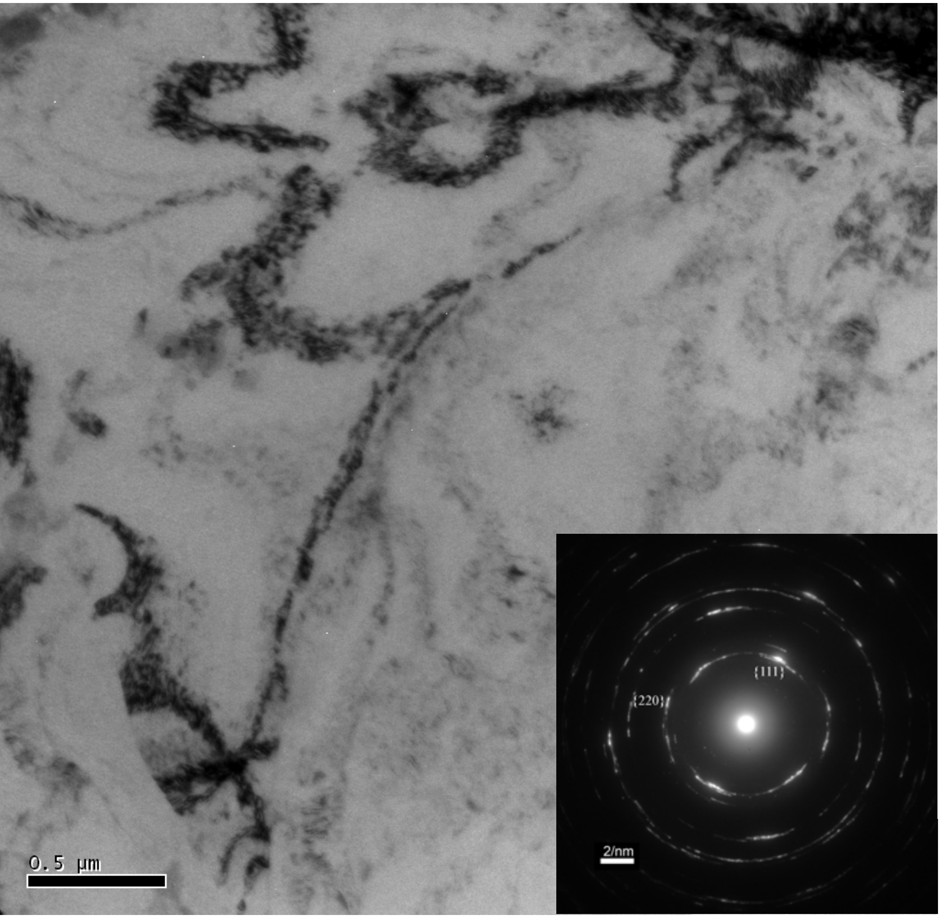
Lattice image of single grain



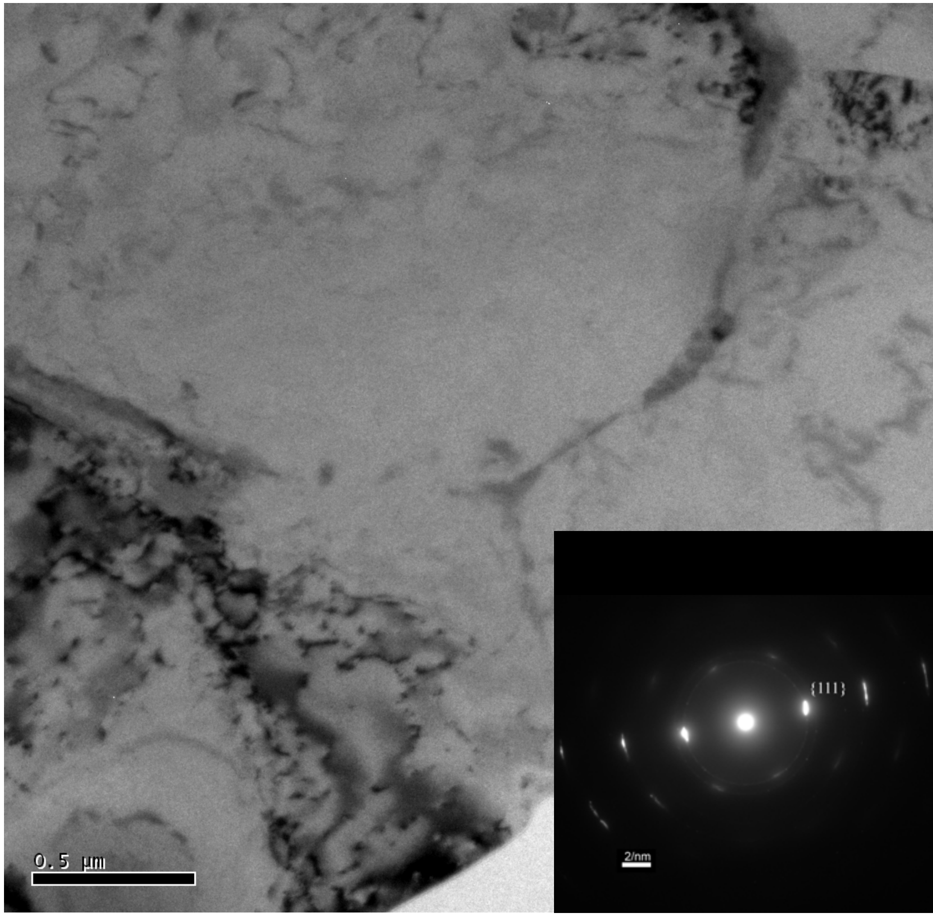




Coatings prepared using as-received powders show "normal" sized grains



5083 as-received



6061 as-received

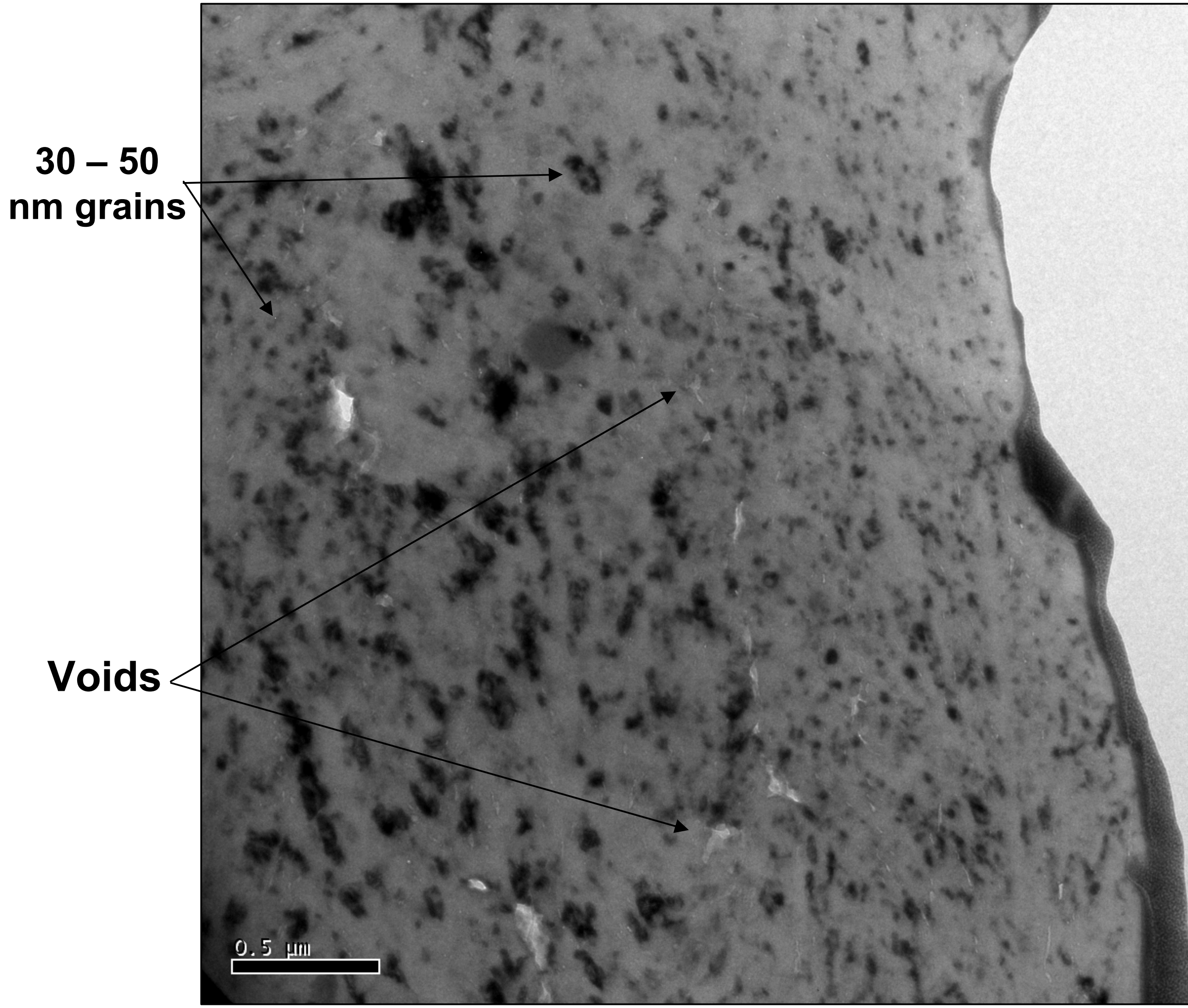
- **Large (>500nm) grains with heavy deformation**
- **SADP show smeared spots from deformed grains.**

- **Large grains (> 1μm) observed throughout 6061 as-received coatings**
- **SADP from single grain**

Exceptional Service in the National Interest

Sandia National Laboratories

6061 LN_2 ball milled powder results in a truly nanocrystalline coating



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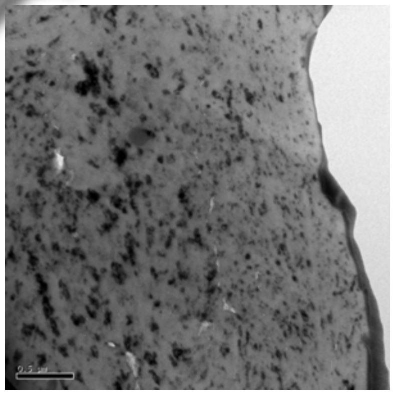
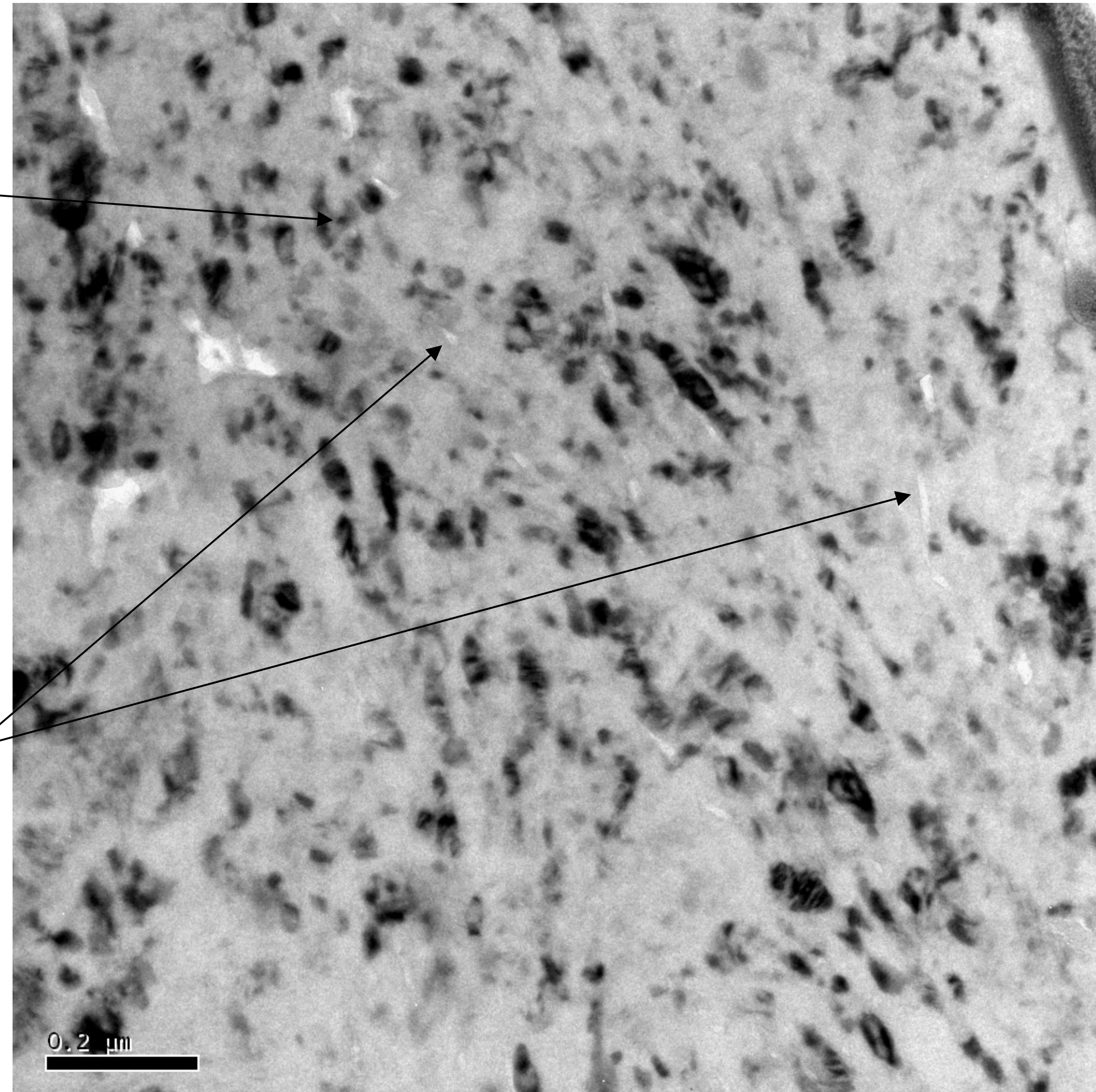


30 – 50 nm grains

Voids



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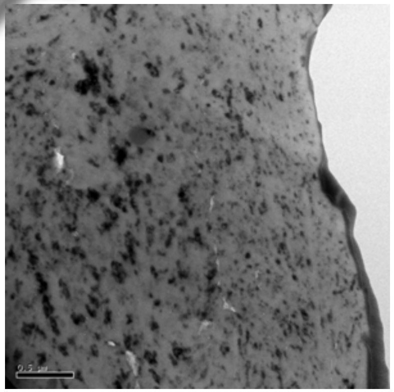
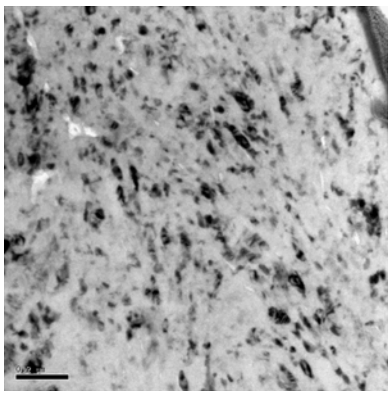
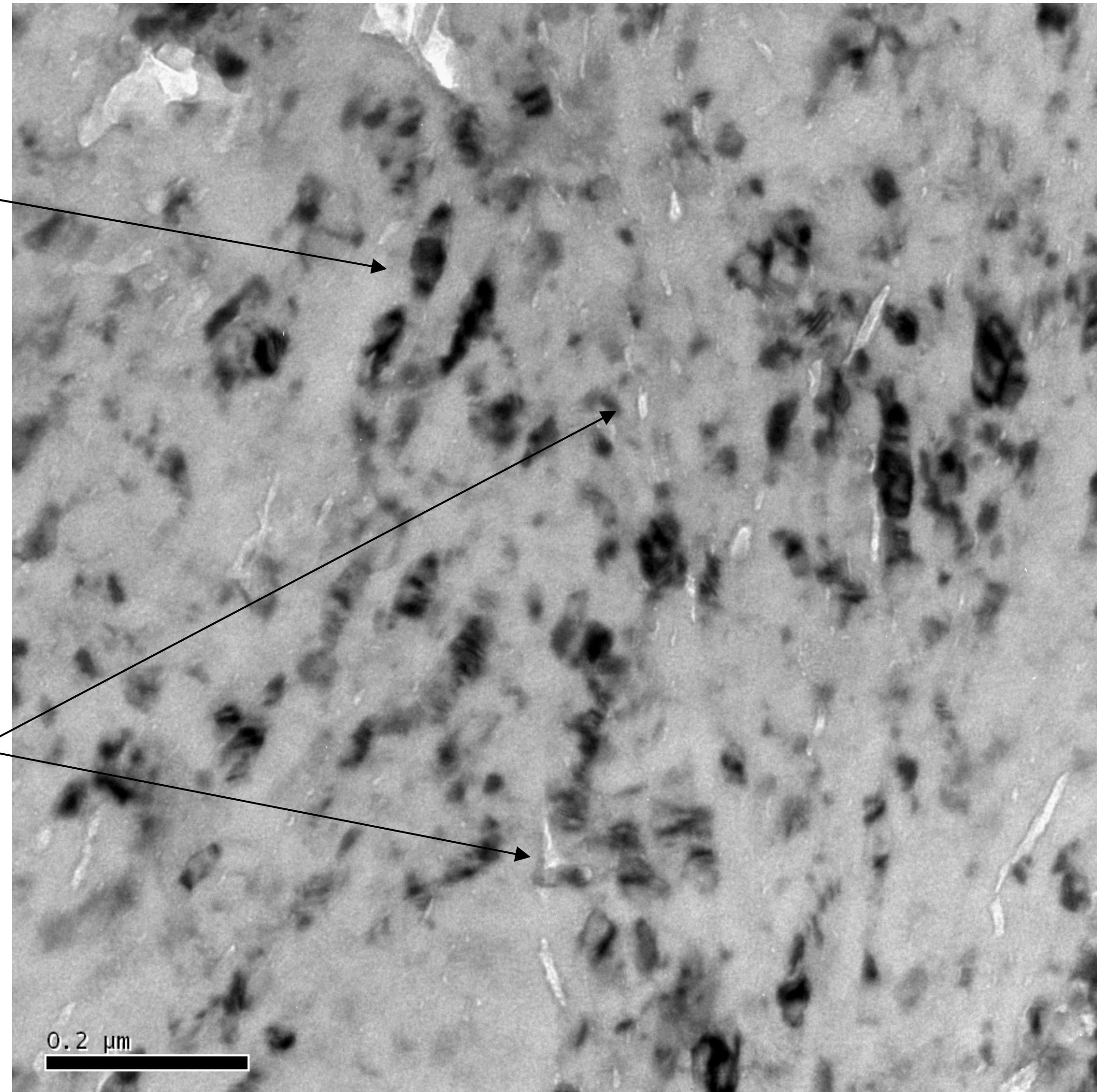




30 – 50 nm grains

Voids



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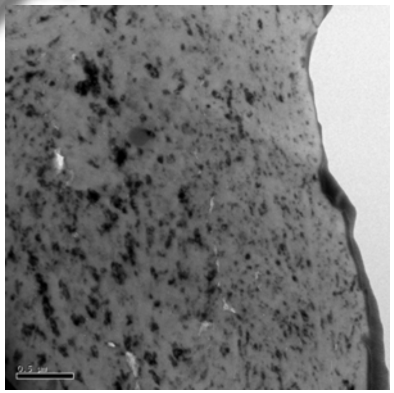
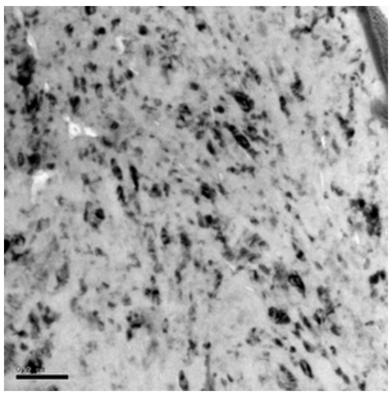
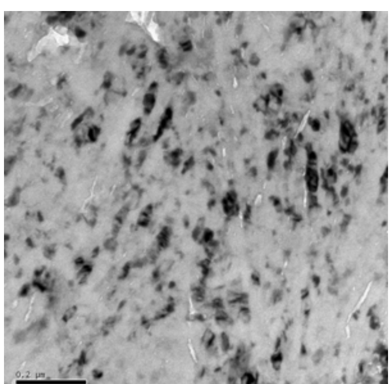
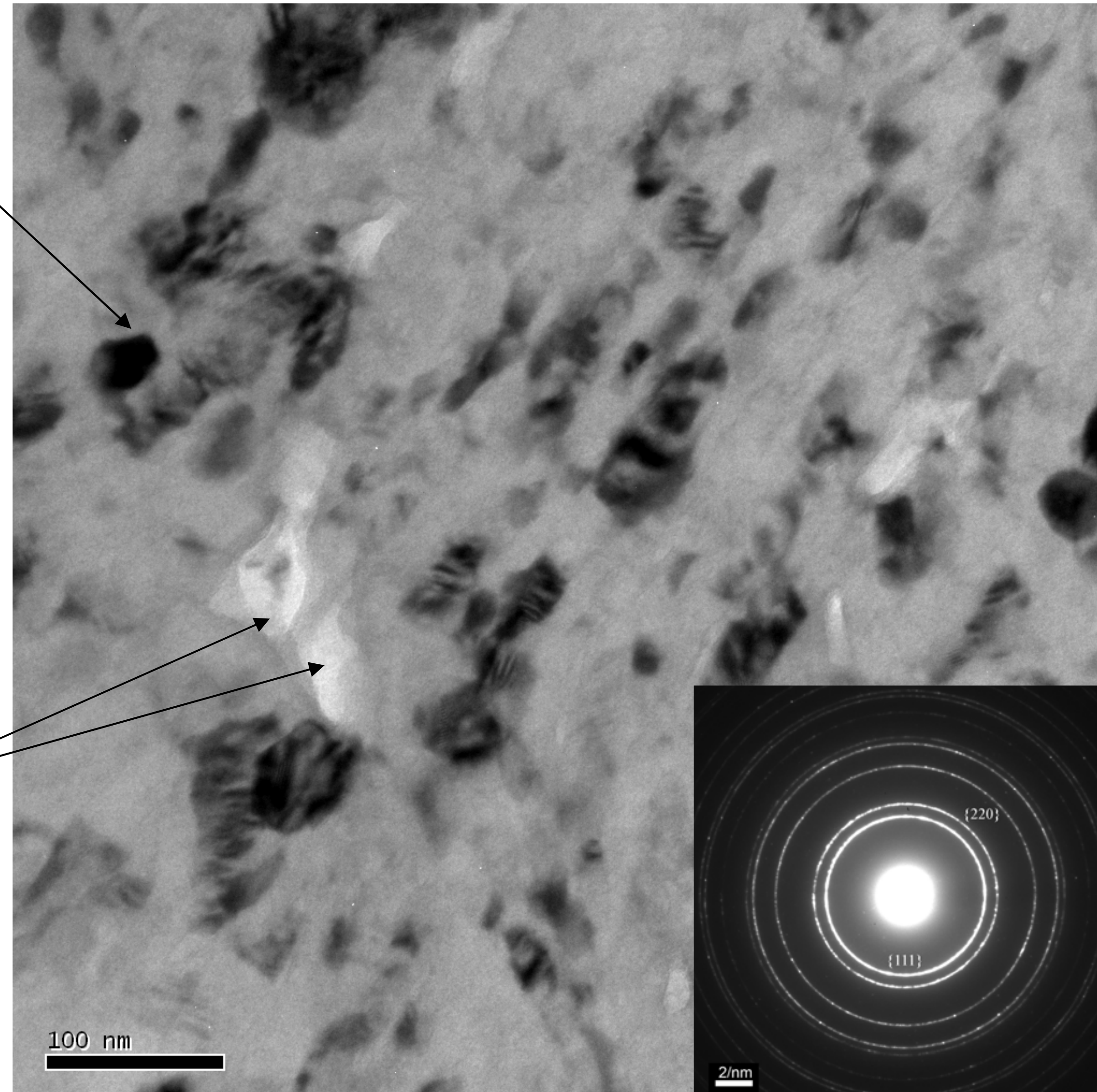






30 – 50 nm grains

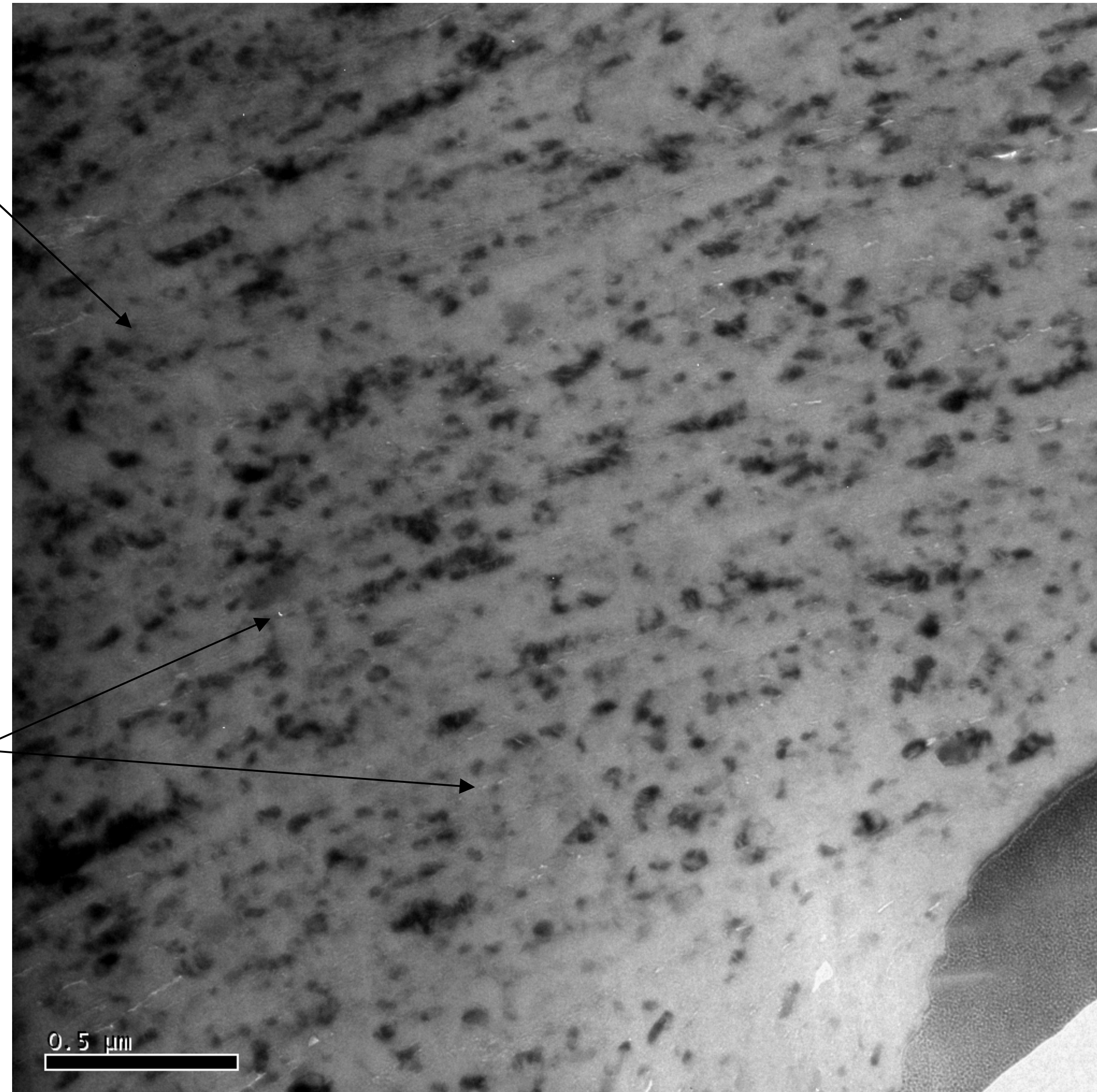
Voids



No discernable texture!

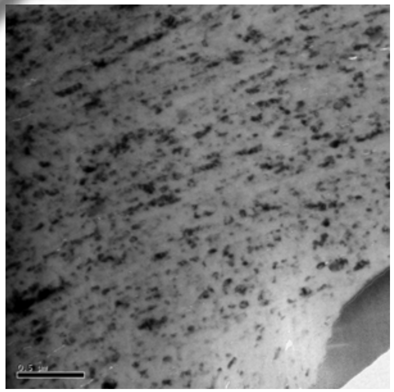
5083 LN_2 ball milled powder also results in a truly nanocrystalline coating

20 – 50
nm grains

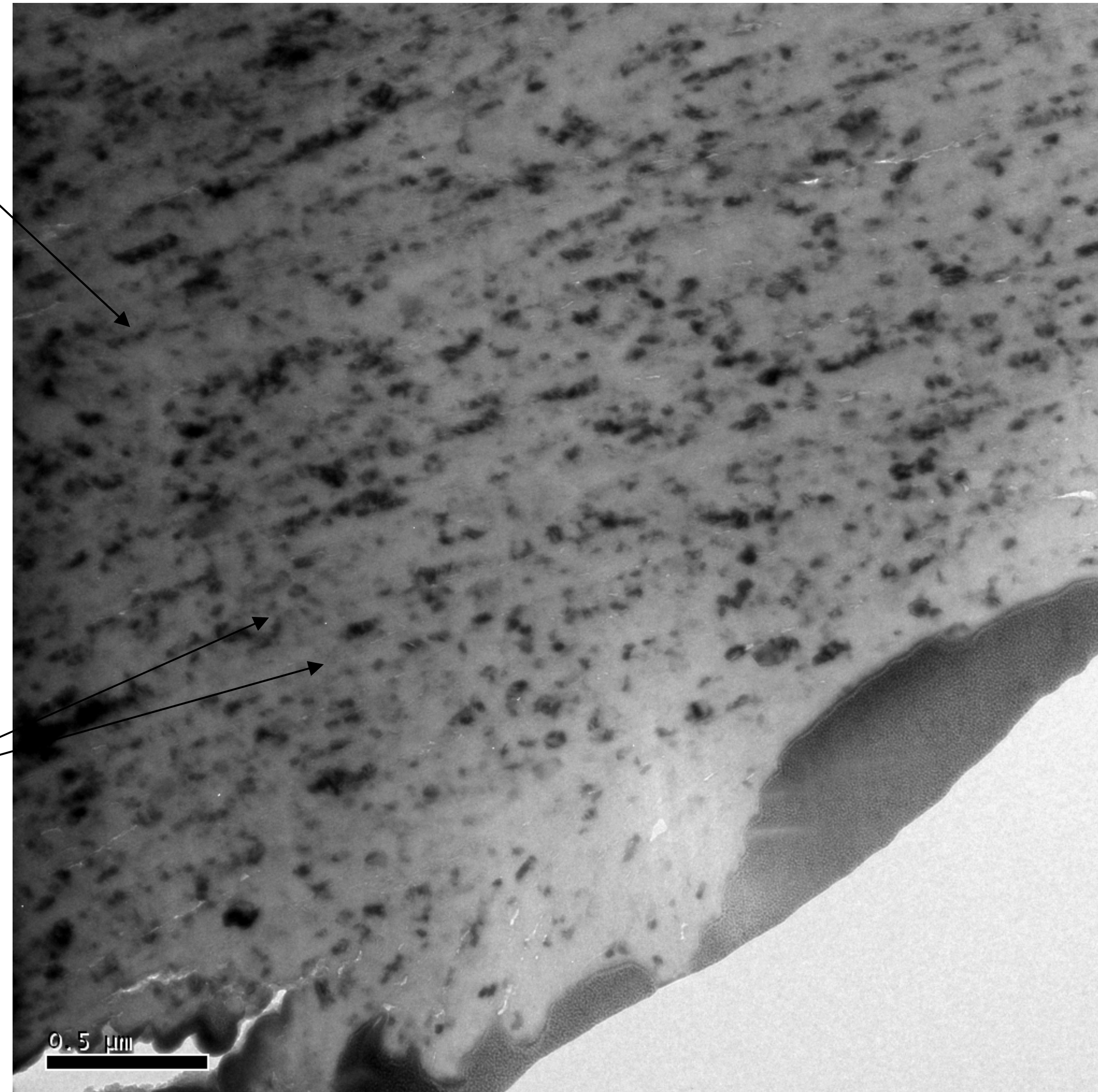


Voids

5083 LN_2 ball milled powder also results in a truly nanocrystalline coating

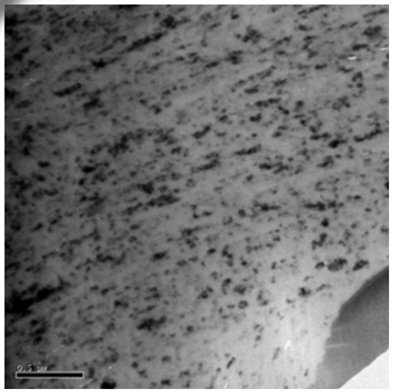


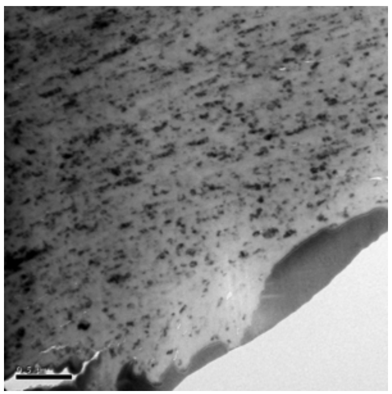
20 – 50 nm grains

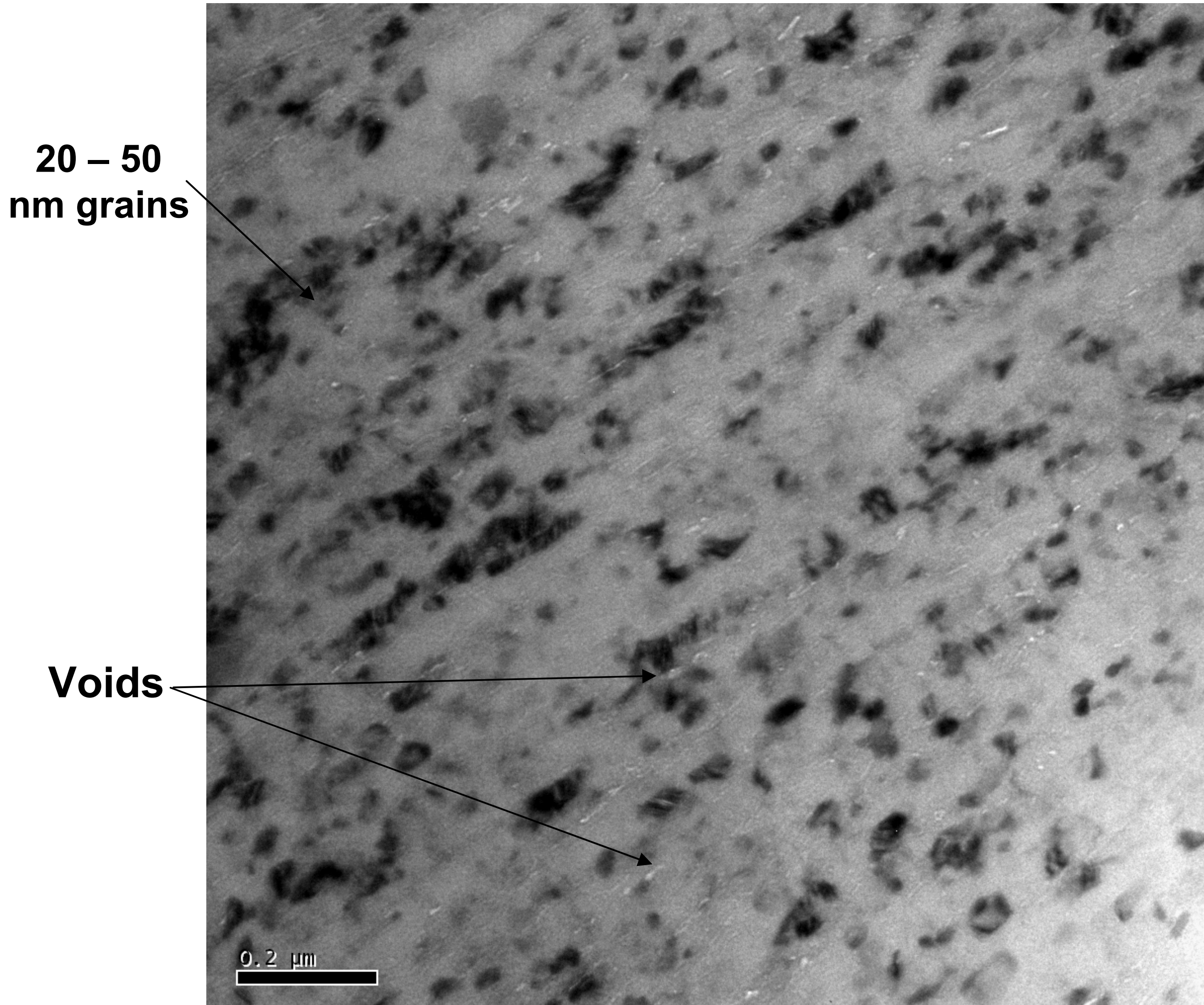


Voids

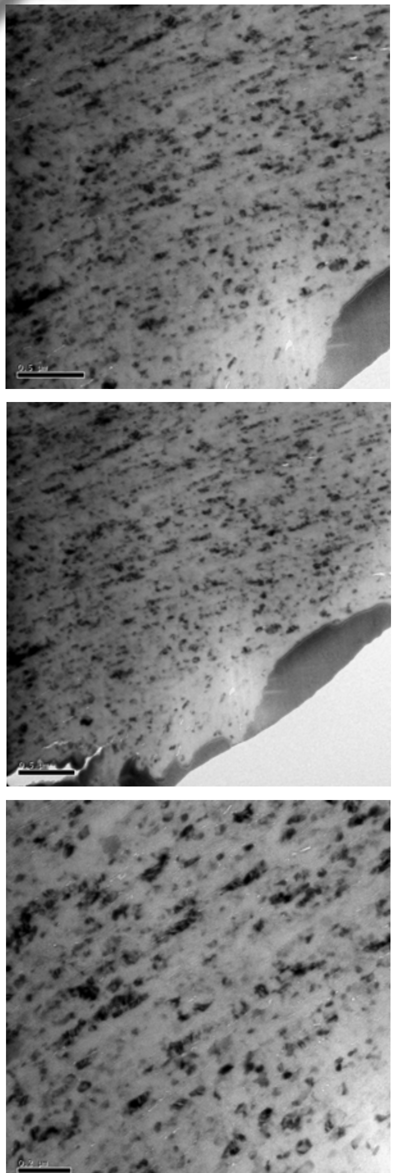
5083 LN_2 ball milled powder also results in a truly nanocrystalline coating





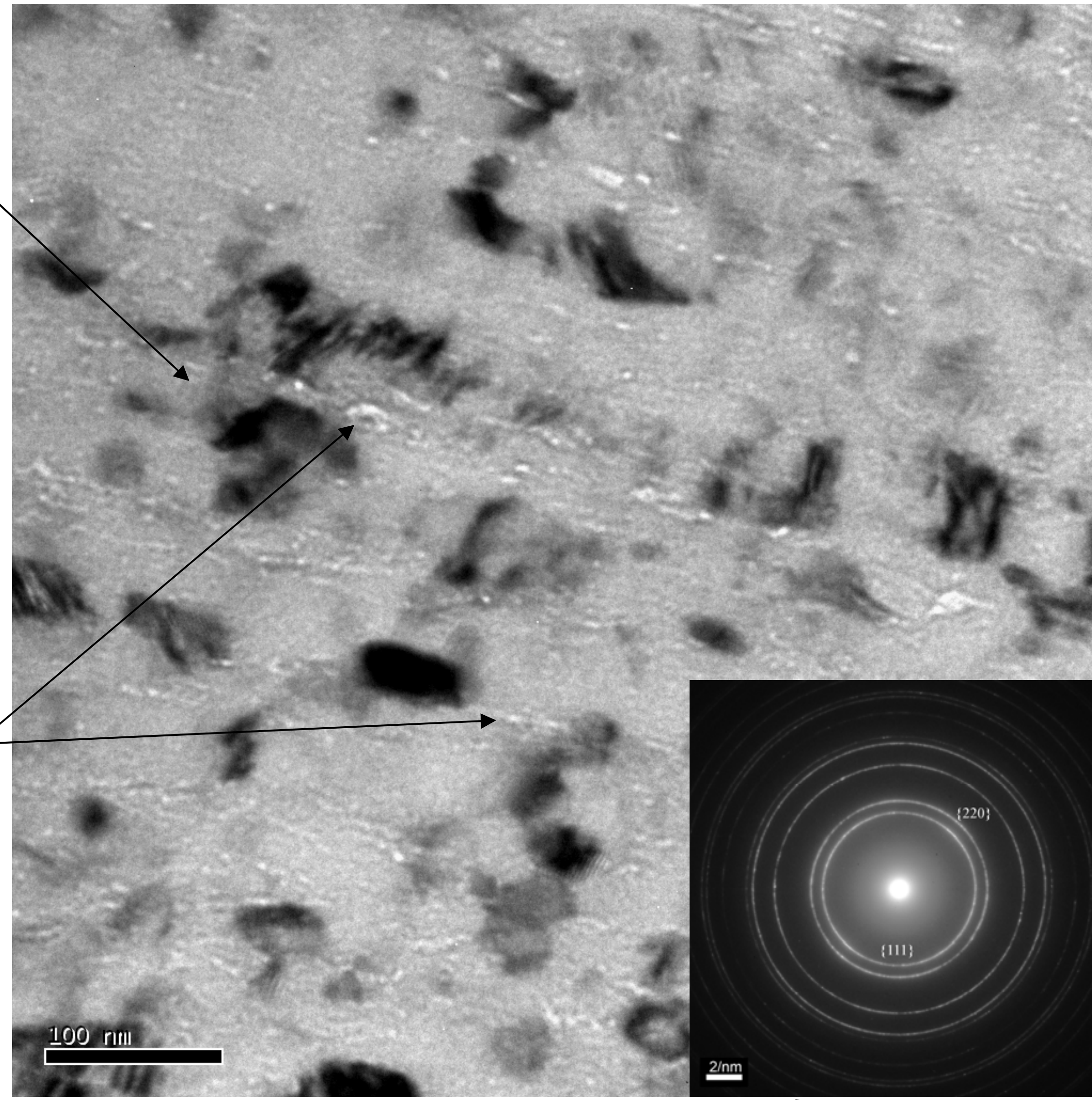


5083 LN_2 ball milled powder also results in a truly nanocrystalline coating



20 – 50 nm grains

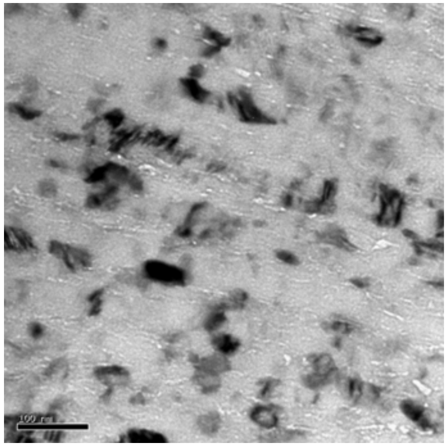
Voids



No significant texture

These microstructures are approaching the minimum grain size for aluminum

- Minimum grain size for Aluminum is ~ 20 nm
- Dislocations are unstable in Aluminum grains smaller than 18nm
- Plastic deformation is responsible for grain refinement in LN_2 ball milled and cold sprayed aluminum
- Both cold sprayed samples show grain sizes between 20 and 50 nm.
- Grain refinement through plastic deformation will not create Al grains less than ~ 20 nm in size.



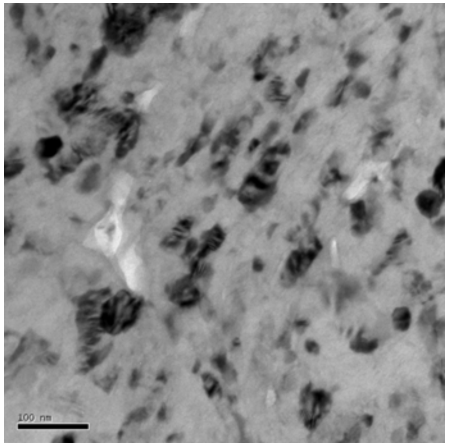


TABLE 1
Critical length of dislocation stability for metal nanocrystals with slipping interfaces

	Cu	Al	Ni	α-Fe
Elastic modulus G (GPa)	33	28	95	85
Lattice parameter b (nm)	0.256	0.286	0.249	0.248
Peierls stress σ_p (10^{-2} GPa	1.67	6.56	8.7	45.5
Λ (nm), sphere	38	18	16	3
Λ (nm), cylinder	24	11	10	2

A. E. Romanov, Continuum Theory of Defects in Nanoscaled Materials, *NanoStructured Materials*, 1995, 6(1-4) p 125-134

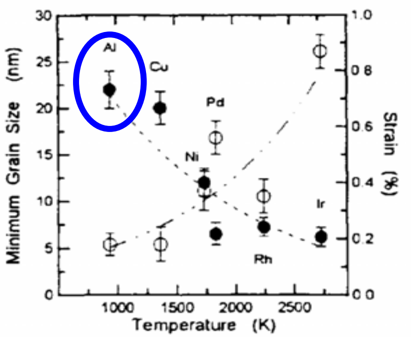
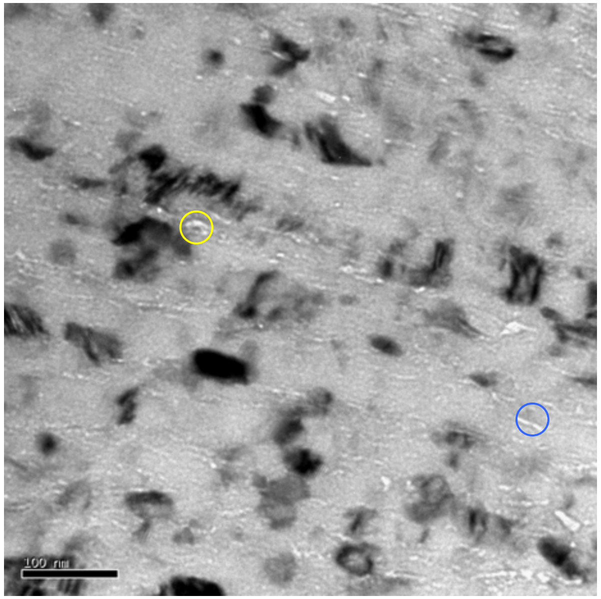


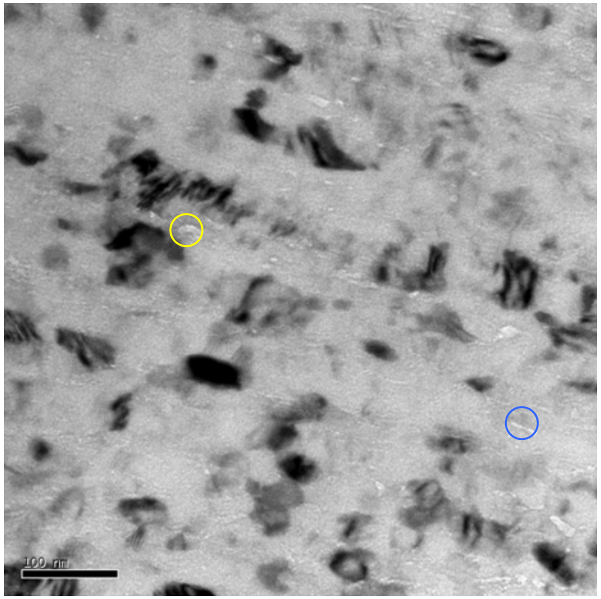
Figure 1. Minimum average grain size (filled symbols) and atomic-level strain (open symbols) for ball-milled fcc metals versus melting temperature.

J. Eckert, Relationships Governing the Grain Size of Nanocrystalline Metals and Alloys, *NanoStructured Materials*, 1995, 6(1-4), p 431-416

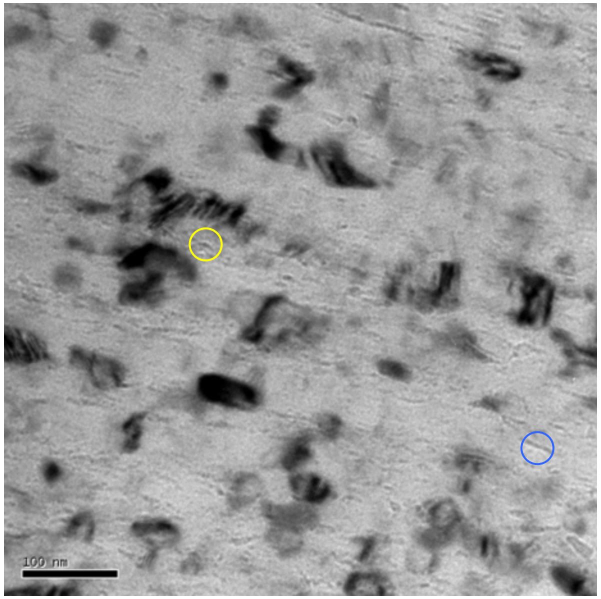
Under-focus / over-focus TEM image pairs prove that the features in the micrographs are voids.



Under-focus



In-focus

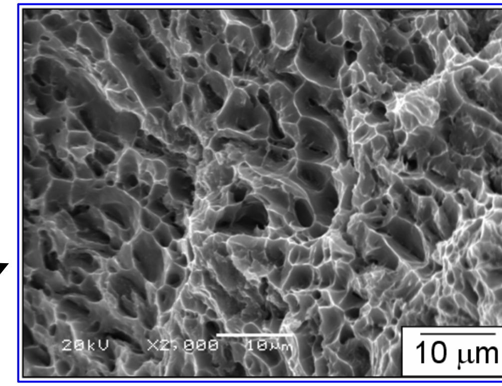


Over-focus

- Voids are:
  - Visible throughout sample
  - Aligned parallel to layers of nano-grains
- Under-focus/Over-focus pairs demonstrate Fresnel contrast, indicative of voids.

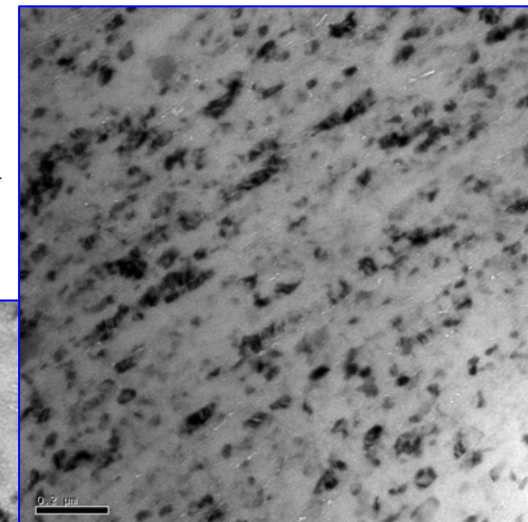
Void formation is likely due to large amounts of plastic deformation from ball milling and cold spray.

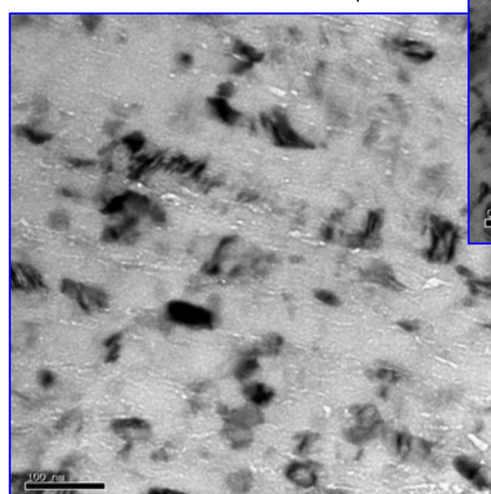
- Void formation is commonly associated with ductile fracture
- Homogenous void is nucleation possible*
- Heterogeneous void nucleation is more likely*
  - Within precipitates
  - At interfaces
  - At grain boundary triple points
- Both 5083 & 6061 contain precipitate forming elements
- Aluminum nitrates are known to form in LN_2 ball milled aluminum†.



Ductile fracture surface in cold sprayed aluminum

Cold sprayed nanocrystalline aluminum showing voids at grain boundaries





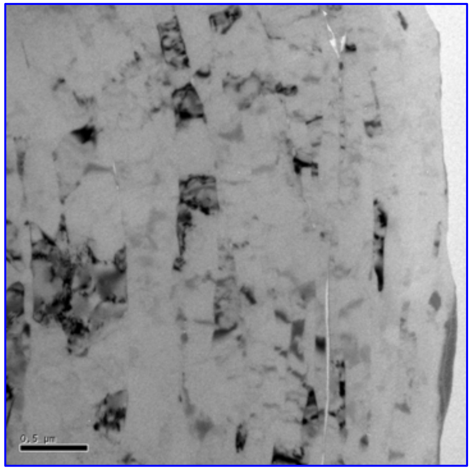
*S. H. Goods and L. M. Brown, The Nucleation of Cavities By Plastic Deformation, *Acta Metallurgica*, 1979, 27, p 1-15

†L. Ajdelsztajn, B. Jodoin, G. E. Kim, J. M. Schoenung, J. Mondoux, Cold Spray Deposition of nanocrystalline Aluminum Alloys, *Metallurgical and Materials --Transactions A*, 2005, 36A, p 657-666

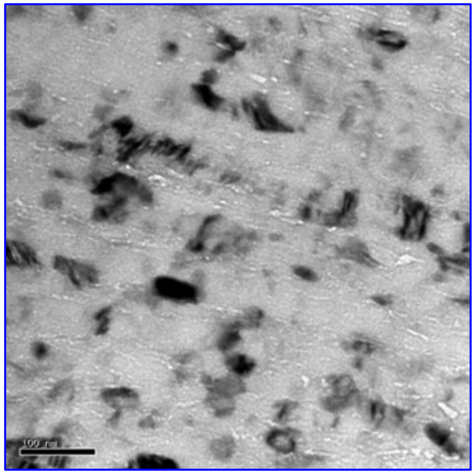
Summary

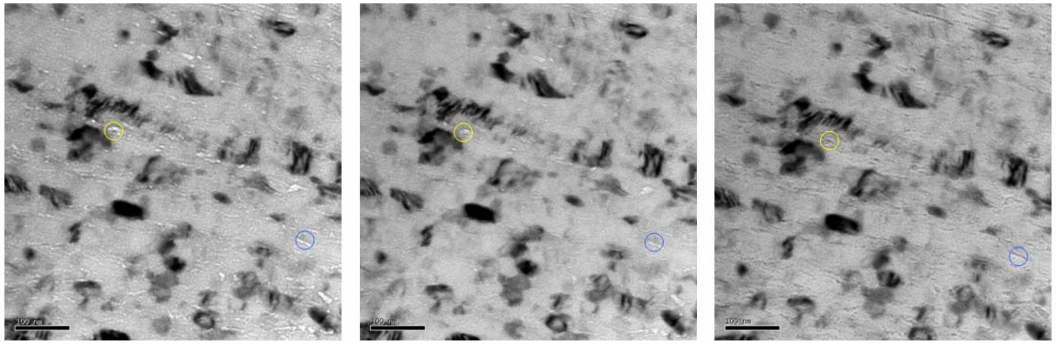
- Cold Spraying LN_2 ball milled aluminum is an effective method for preparing nanocrystalline aluminum
- Significant grain refinement can occur during the cold spray process
- Voids are observed in nanocrystalline cold sprayed aluminum.

LN_2 ball milled powder containing 250 - 400 nm grains



Cold Spray coating containing 20 - 50 nm grains





Under-focus In-focus Over-focus