Low Pressure Plasma Spray (LPPS-Thin Film®) at Sandia National Laboratories

SAND2010-4811P

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"Exceptional Service In the National Interest"

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Sandia National Laboratories

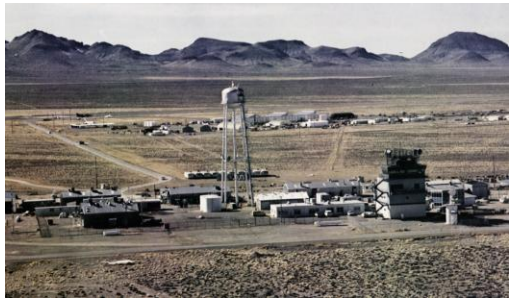
Sandia National Laboratories

Albuquerque, New Mexico

- More than 8,600 full-time employees
- More than 1,500 PhDs and 2,700 MS/MAs
- ~ 2,200 on-site contractors
- ~ $2.33 billion total budget

Yucca Mountain, Nevada

Kauai Test Facility, Hawaii



Tonopah Test Range, Nevada

WIPP, New Mexico

Pantex, Texas

Livermore, California

"Exceptional Service In the National Interest"

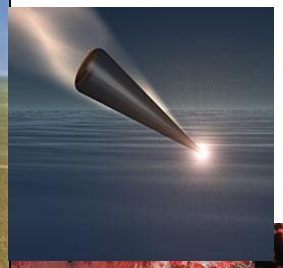
National Nuclear Security Administration

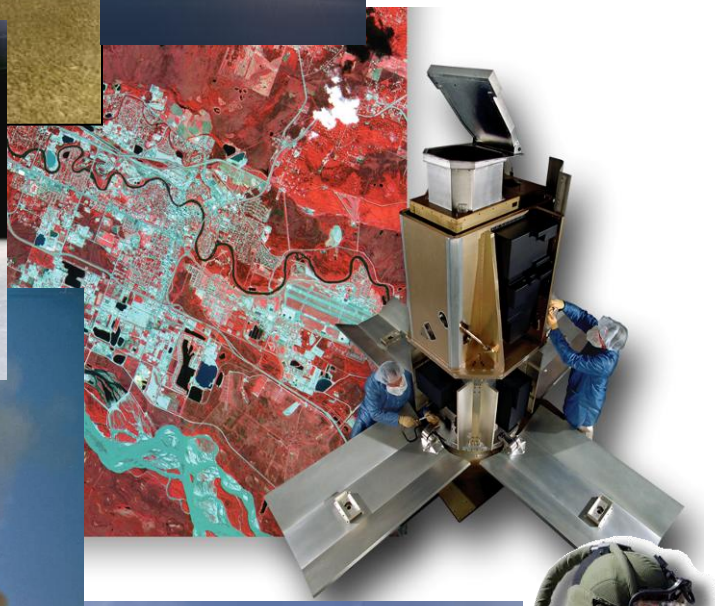
LOCKHEED MARTIN

Sandia National Laboratories

Sandia is a National Security Laboratory

We develop technologies to:





- **Sustain, modernize, and protect our nuclear arsenal**
- **Prevent the spread of weapons of mass destruction**
- **Provide new capabilities to our armed forces**
- **Protect our national infrastructures**
- **Ensure the stability of our nation's energy and water supplies.**
- **Defend our nation against terrorist threats**

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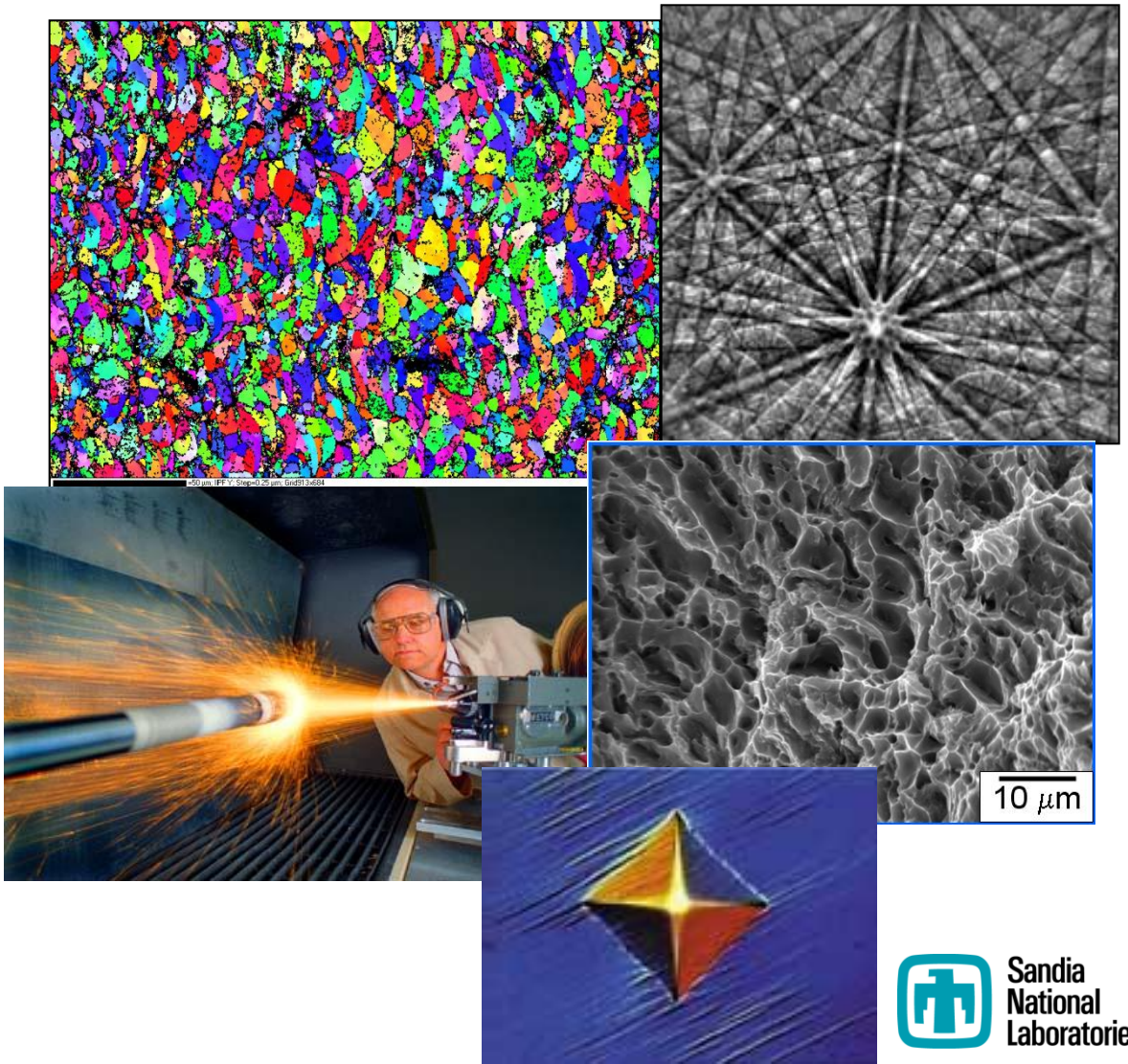
Sandia National Laboratories

The Materials Science & Engineering Center supports Sandia's missions.

- ~ 250 people
- ~ 100 Ph.D.'s
- Polymers
- Ceramics
- Metals
- Advanced analytical techniques
- Atomistic & mesoscale modeling
- Mechanical metallurgy
- Process diagnostics
- Tribology
- Materials aging



"Exceptional Service In the National Interest"

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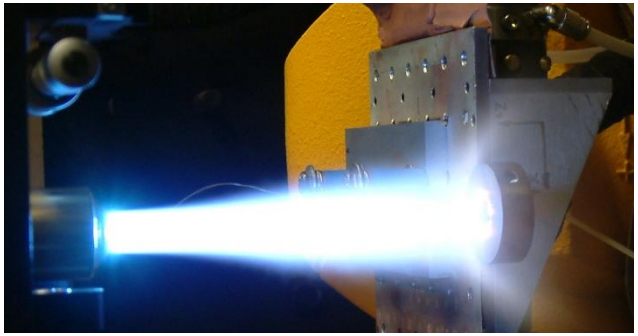
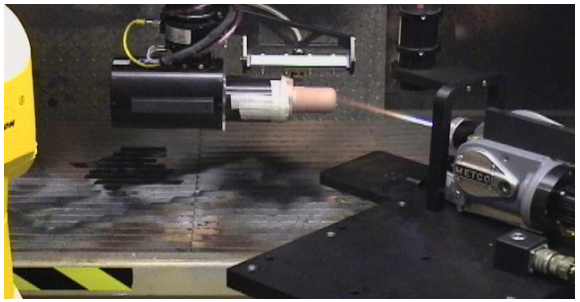
The TSRL operates a wide variety of thermal spray equipment.

Atmospheric Plasma Spray

Twin Wire Arc Spray



Powder Flame Spray



Wire Flame Spray

"Exceptional Service In the National Interest"

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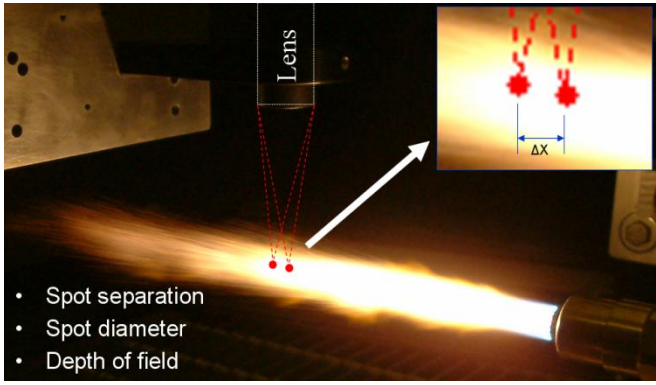
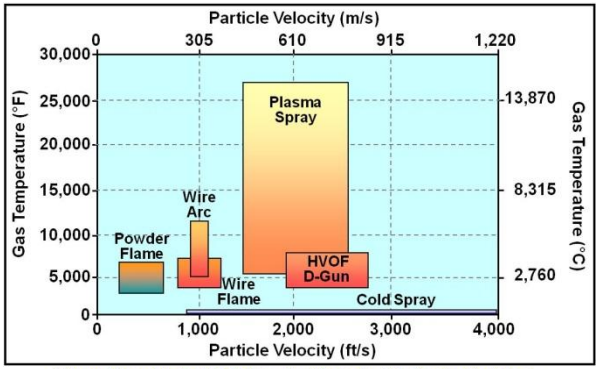
What makes the TSRL unique?

- Focus on Process Diagnostics for Thermal Spray
  - DPV-2000, Accura G3
  - L2F Laser Velocimeter
  - Control Vision
  - Spectroscopy
  - Xenon-Flash Thermal Diffusivity
  - Beckman Coulter Particle Size Analysis
- Access to wide range of T_p, V_p space
- Materials Science Expertise and Analysis Capability
- LPPS® Thin Film & Cold Spray
- Experience supporting spray processes across entire technical readiness scale

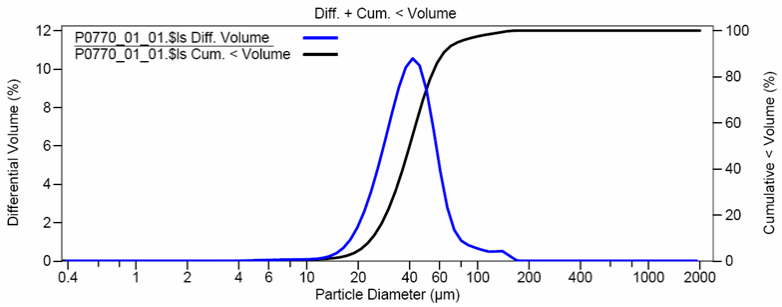
Sensor based particle diagnostics measure particle temperature (Tp) and velocity (Vp).



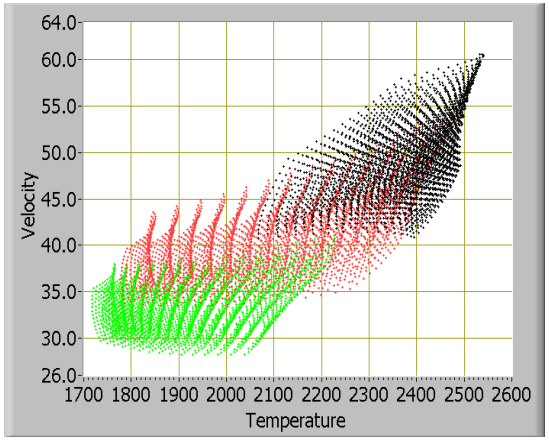
Tp, Vp space accessible at TSRL



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



Powder particle size distribution



Effect of Torch Hardware on Tp, Vp

Wire tip imaging

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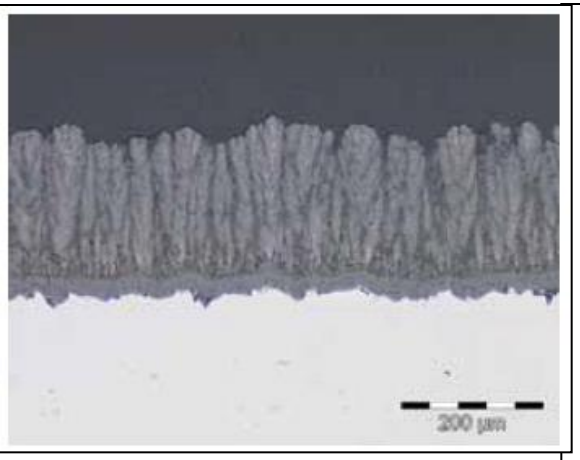
What is LPPS Thin Film®?

Low Pressure Plasma Spray

- Steady State Vacuum Plasma Spray at chamber pressures < 3 Torr (400 Pa)
- A Unique New Thermal Spray Process
  - Invented by Erich Muehlberger (Sulzer-Metco)
  - Capable of preparing dense coatings in the 5-50 micron thickness range.
  - Capable of coating large areas very quickly (1µm/m^2/minute)!
  - Capable of *Droplet, Mixed Mode, & Vapor Deposition*



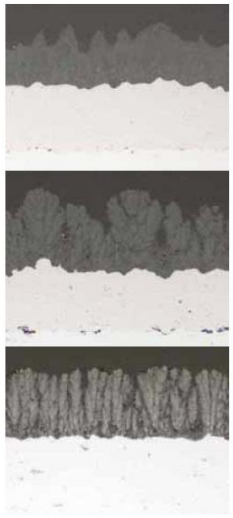


Fig. 5. LPPS-TF deposited TBC layers with increasing "degree of columnarity" and about 100 -150 µm thickness (low-, medium- and high columnarity).

** Figures from 2005 ITSC conference proceedings by: A. Refke, Wohlen/CH, D. Hawley, J. Doesburg and R.K. Schmid, Westbury/USA*

- ***"EB-PVD-like" Yitria-Stablized-Zirconia coatings were reported at the 2005 International Thermal Spray Conference in Basel, Switzerland!***

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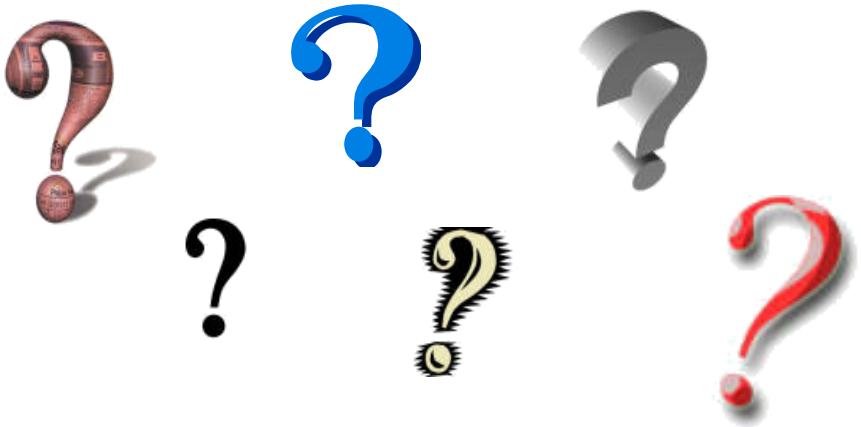
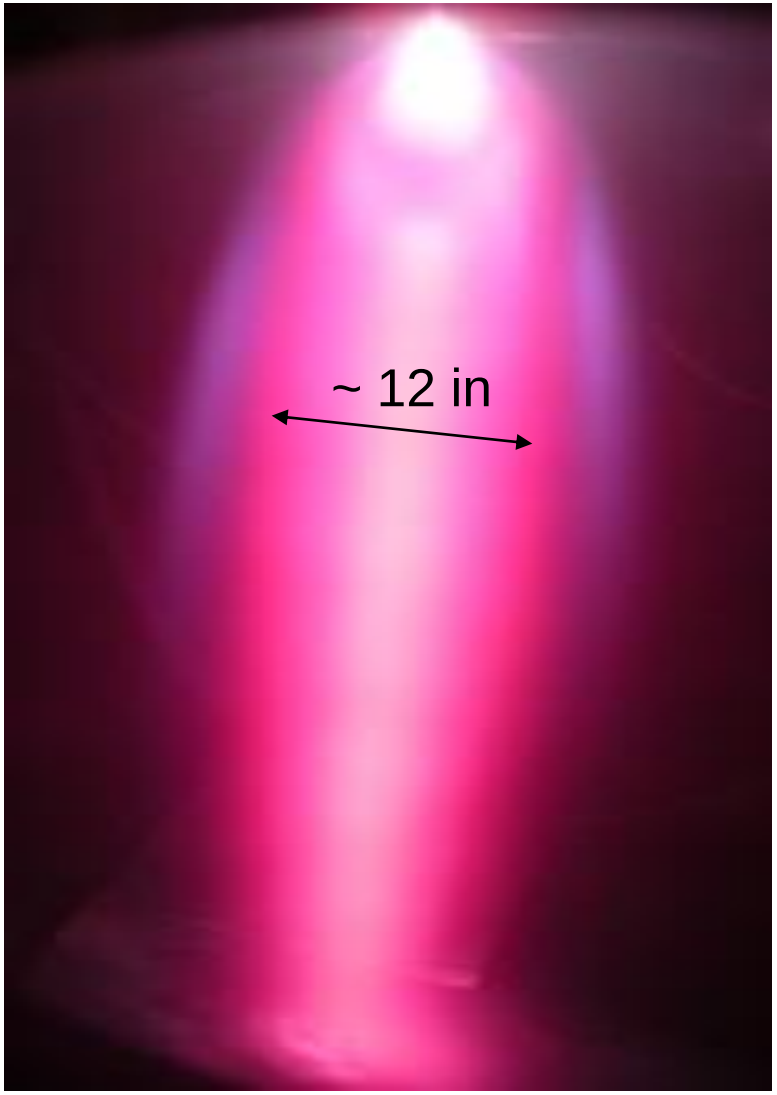
Why is SNL interested?

- Fundamentally new spray process
- Unique deposition mechanisms
- Unique microstructures
- Unique plasma – particle interactions
- Thinner coatings than any other thermal spray process
- Lots of potential for good science…





LPPS Plume at Sandia

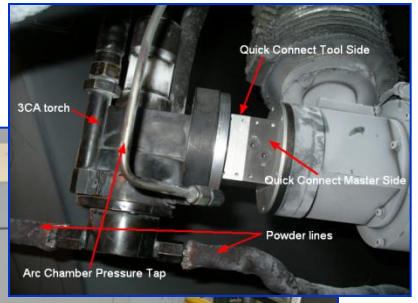
Sandia's LPPS System

LPPS Requires

- High Power (> 50 kw)
- Low Pressure (< 400Pa)
- Long Standoff Distance (> 1m)

~ 1000 L Vacuum Chamber

03CA Plasma torch



2000A, 50V Halmar Power Supply

Dual Vacuum Pump Systems w/ ~ 5000 m^3/hr total capacity

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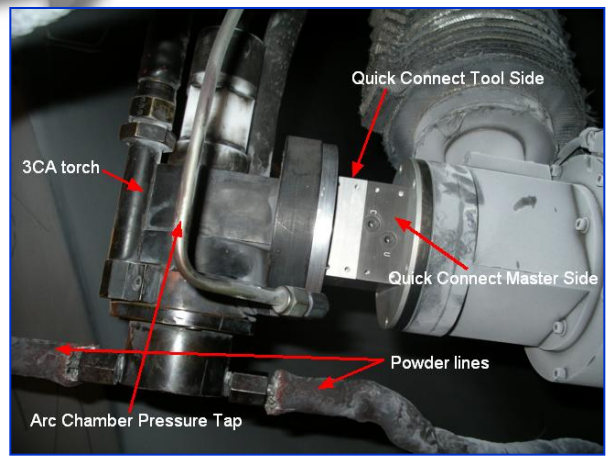
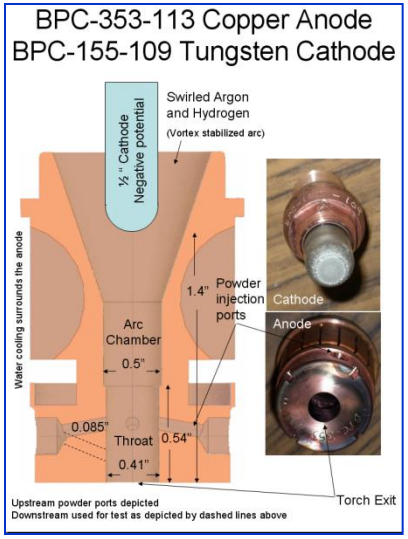
Anode Modification Solved Early Anode Wear Problems



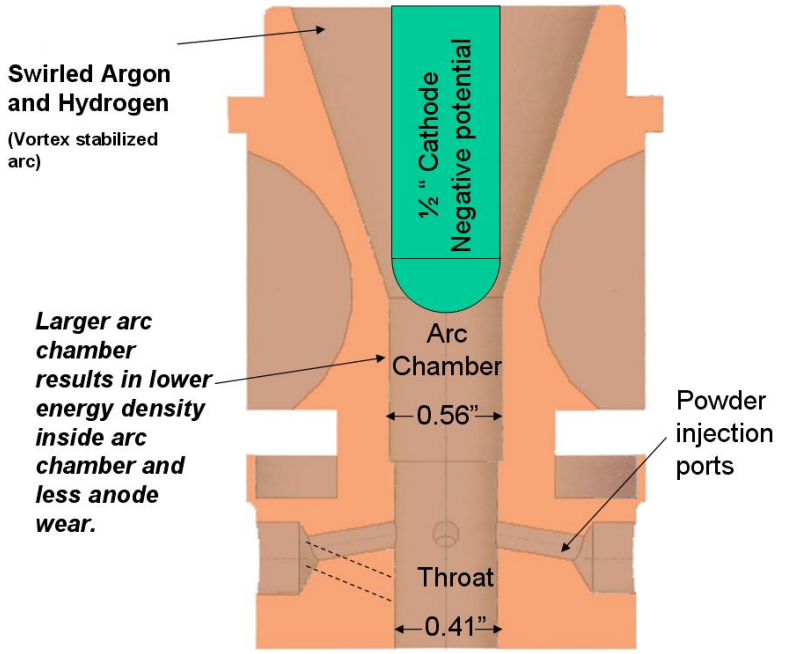


Increasing arc chamber diameter from 0.5" to 0.56" mitigated anode wear problem.

- Arc Current ~1800 amps, Ar/H Plasma
- Anode wear can contaminate coatings with copper.
- Attached arcs can burn through to the water jacket…

1st run 0.5" arc chamber, 0.41" throat: 23 minutes

2nd run 0.525" arc chamber, 0.41" throat: 38 minutes



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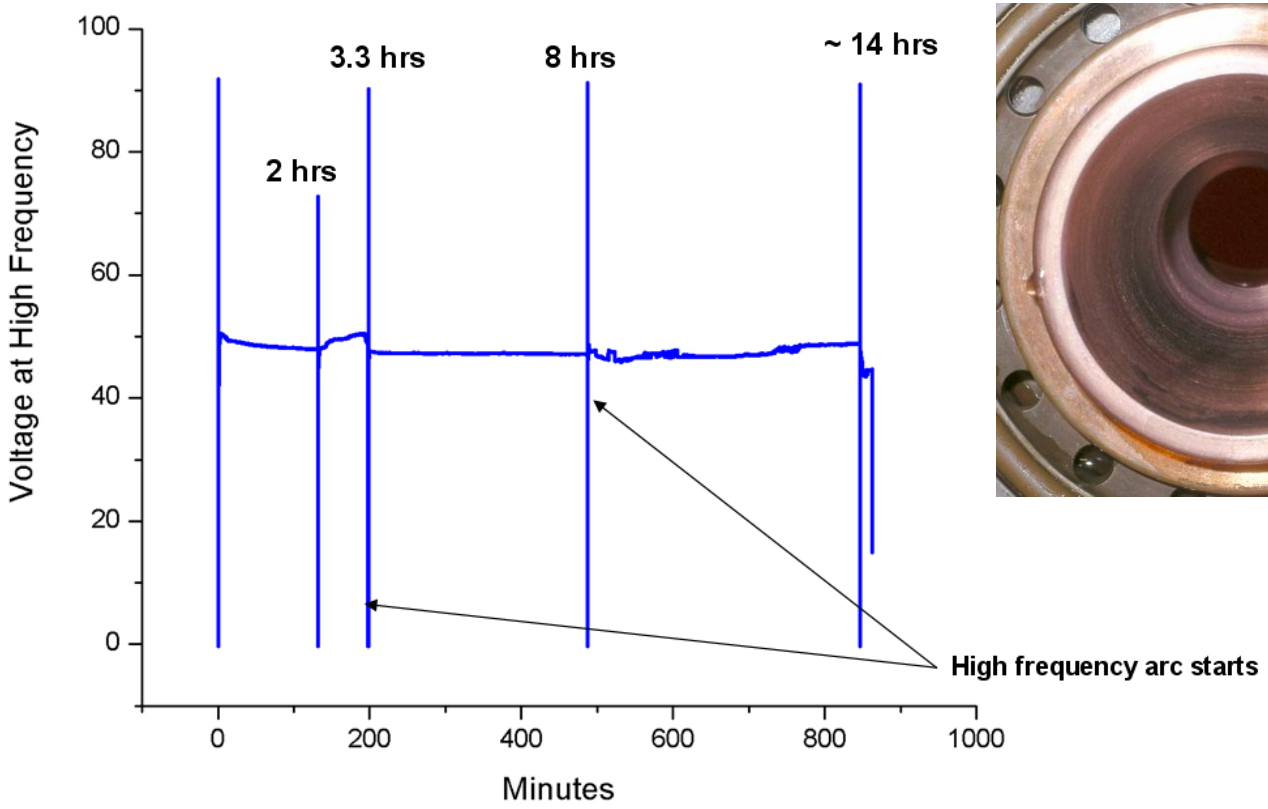
Stable arc voltage and low wear over an 8 hour period indicates a stable anode.



3.3 hours

The arc normally circulates about half way down the arc chamber

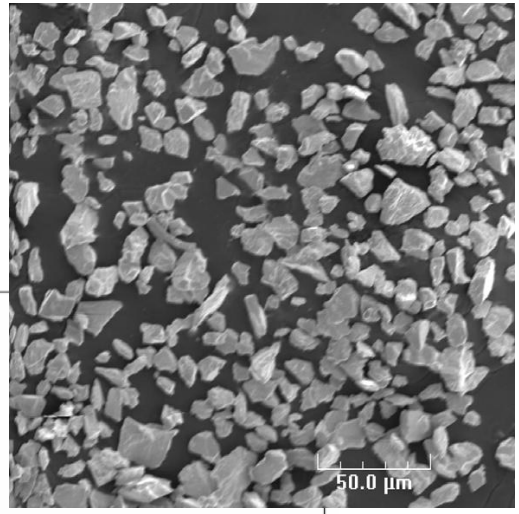
Visible damage from an event well above the arc chamber most likely an unstable arc during torch lighting.

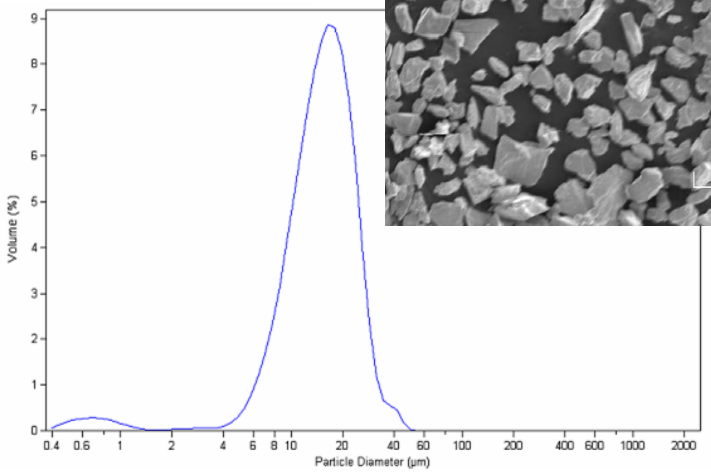
Arc chamber still intact and showing little wear

Fused and Crushed YSZ Feedstock was Used for All Experiments

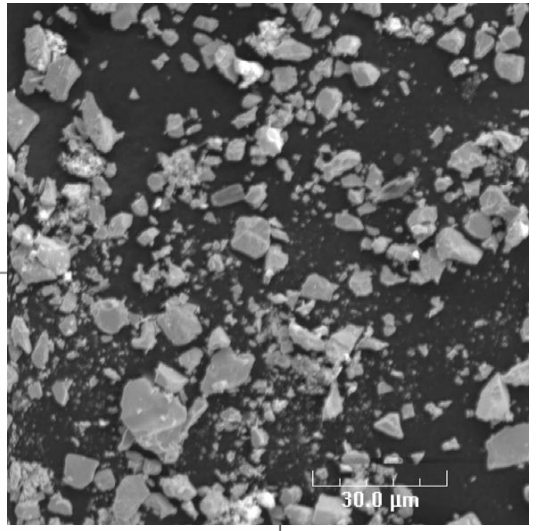
Powder	Mean (µm)	Standard Deviation (µm)	Morphology
Stock	15.98	6.954	Fused and Crushed
Ball Milled	14.21	10.86	Fused and Crushed

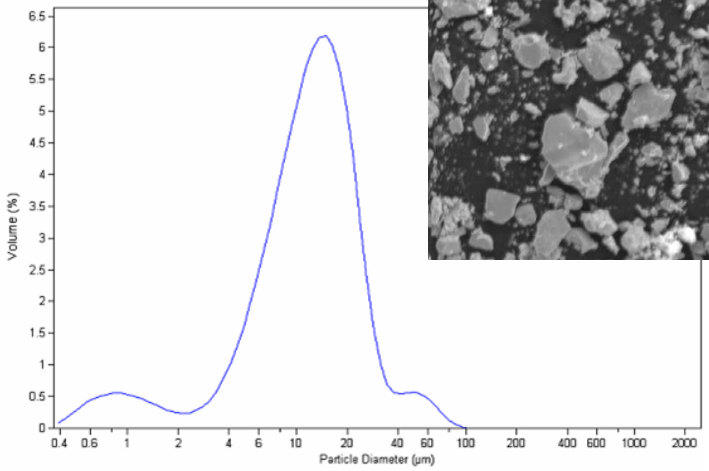
YSZ powder as-received (stock)





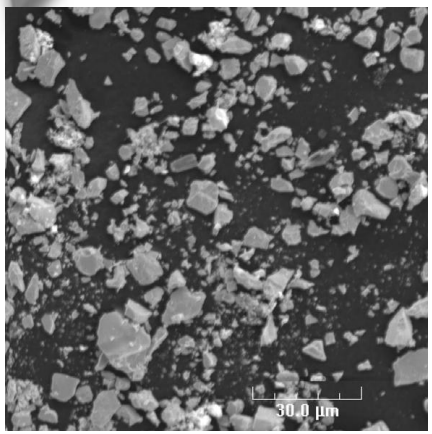
YSZ ball milled 24 hrs



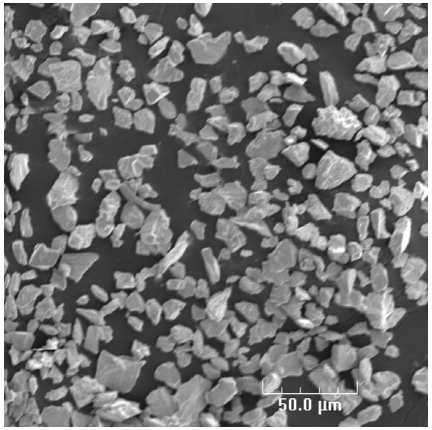


Amperit® 825 Zirconium Oxide – Yttrium Oxide 93-7 ($Zr0_2$-Y_2O_3) (H.C. Starck)

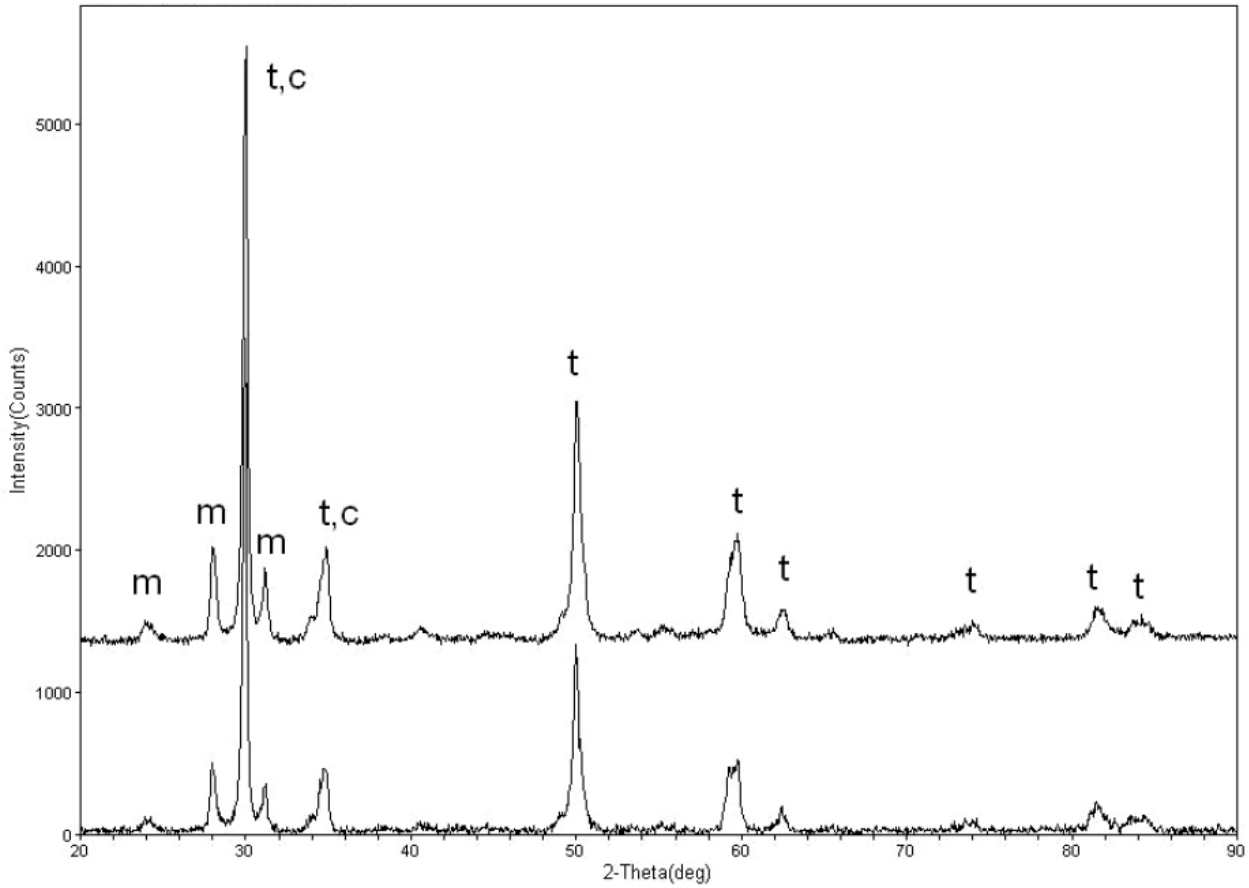
Feed Stock (Amperit 825 YSZ)



Ball Milled

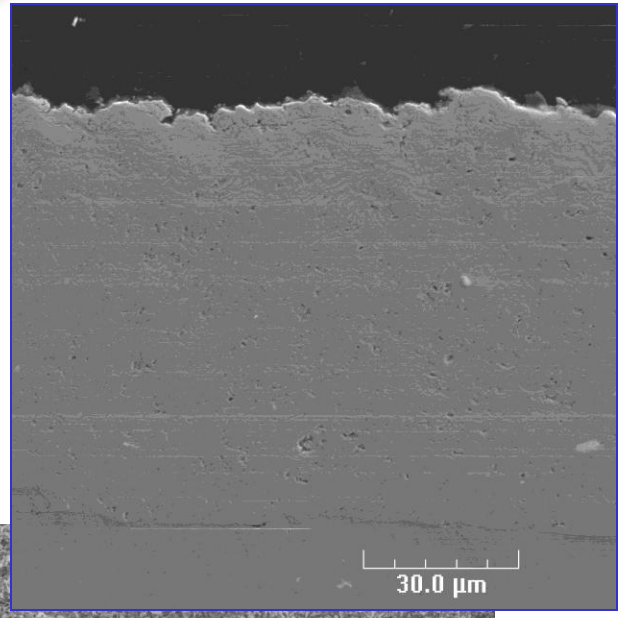


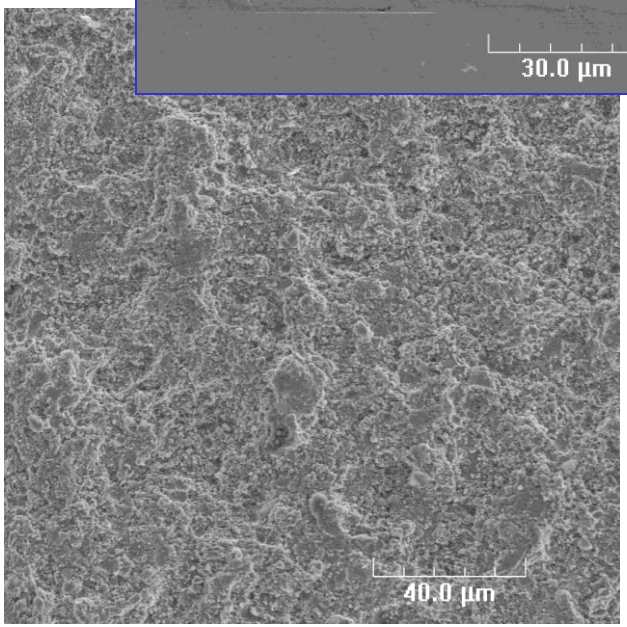
As-Received

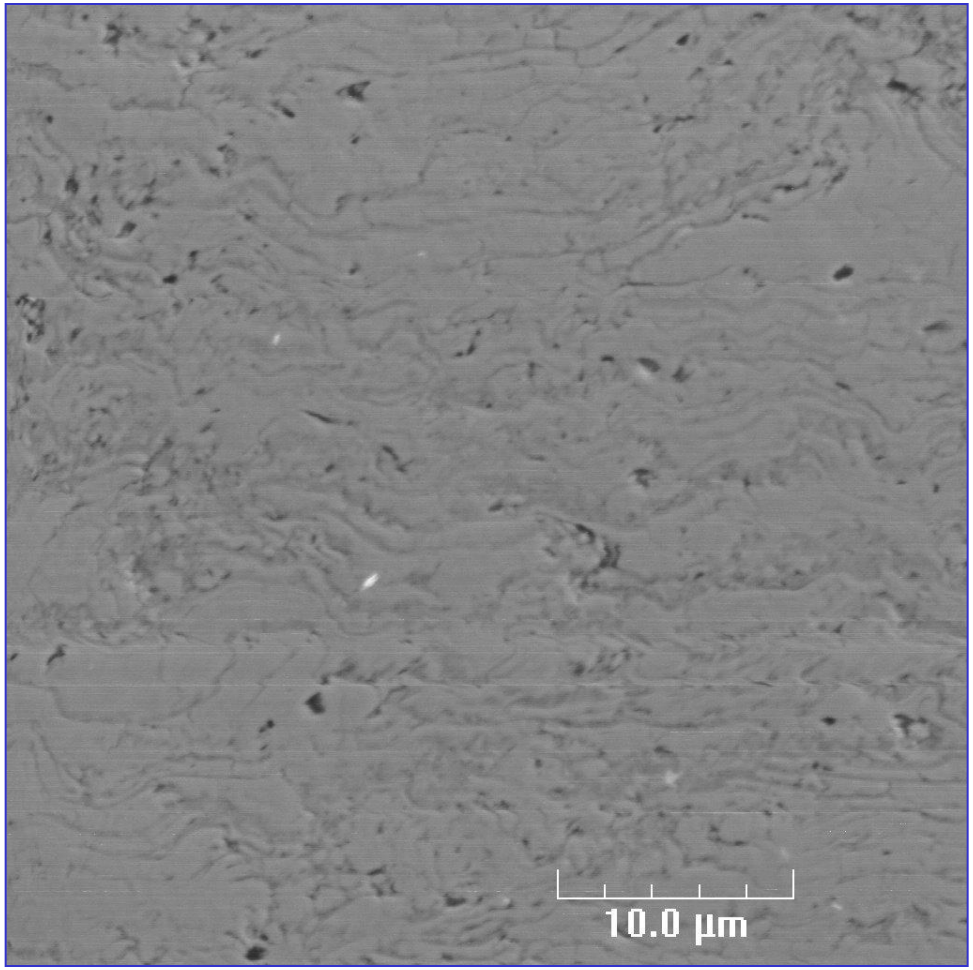


As-received (bottom) and ball milled (top) powder contained a mixture of monoclinic and tetragonal YSZ

Droplet Deposition (YSZ)

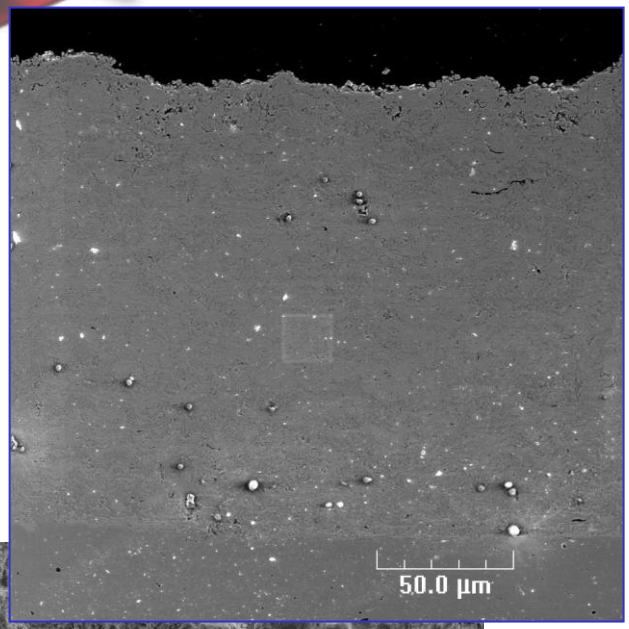


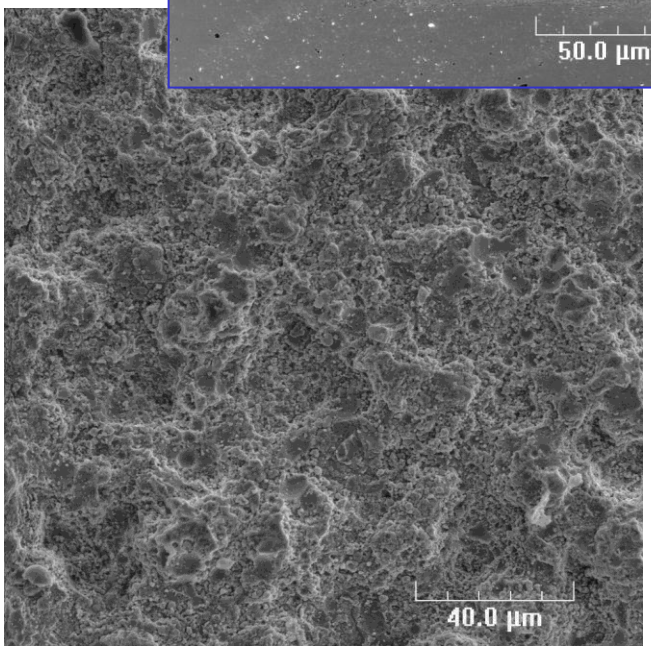


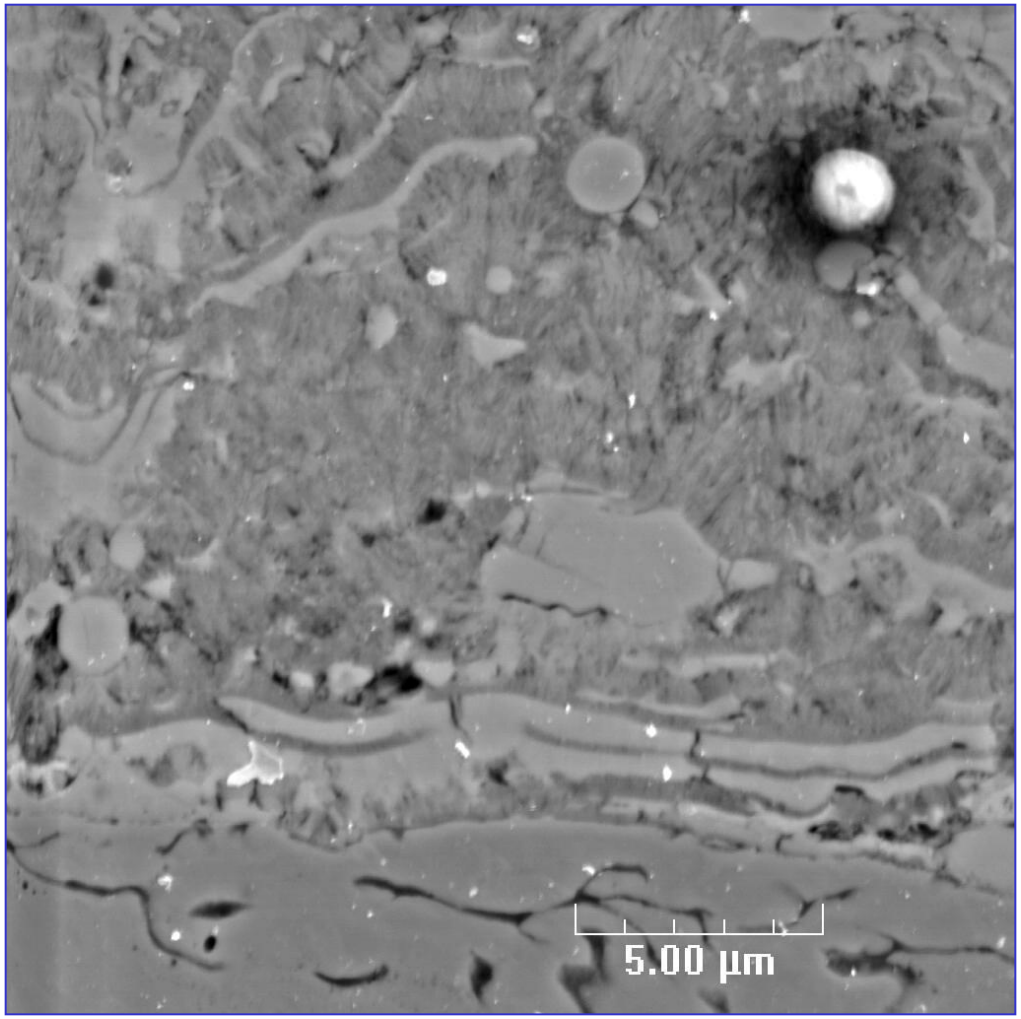


1700 A, 59Ar, 7H, 70He, 2.42 Torr, SD 45”

Mixed mode (vapor + liquid droplet) deposition (YSZ)

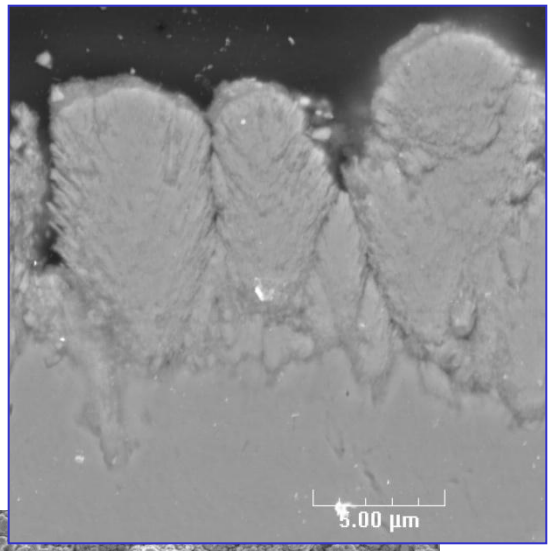


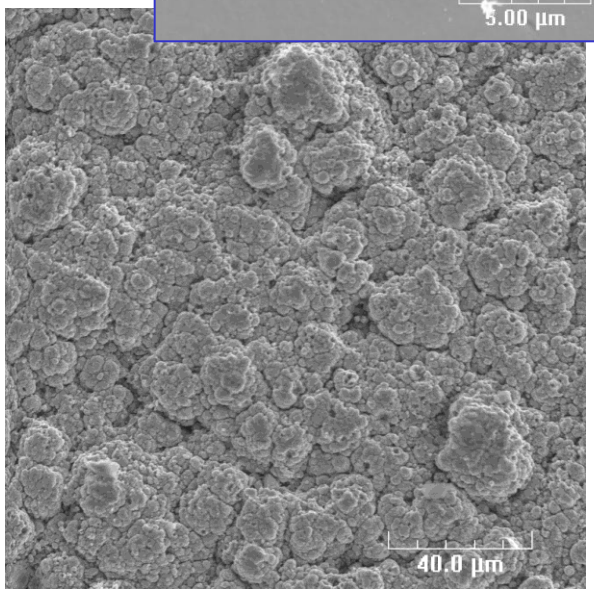


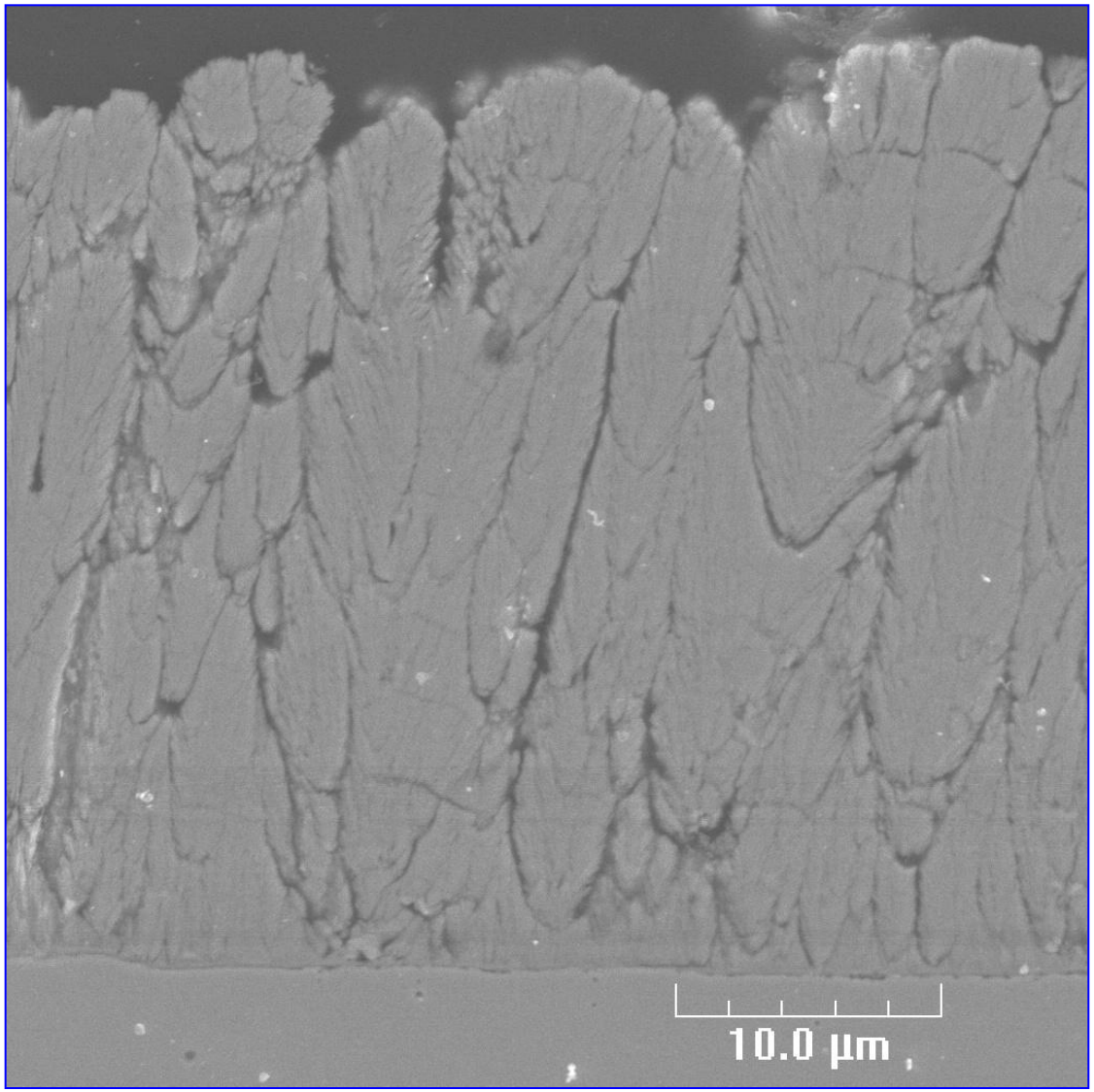


1700 A, 59Ar, 7H, 70He, 2.42 Torr, SD 45"

Vapor Growth (YSZ)

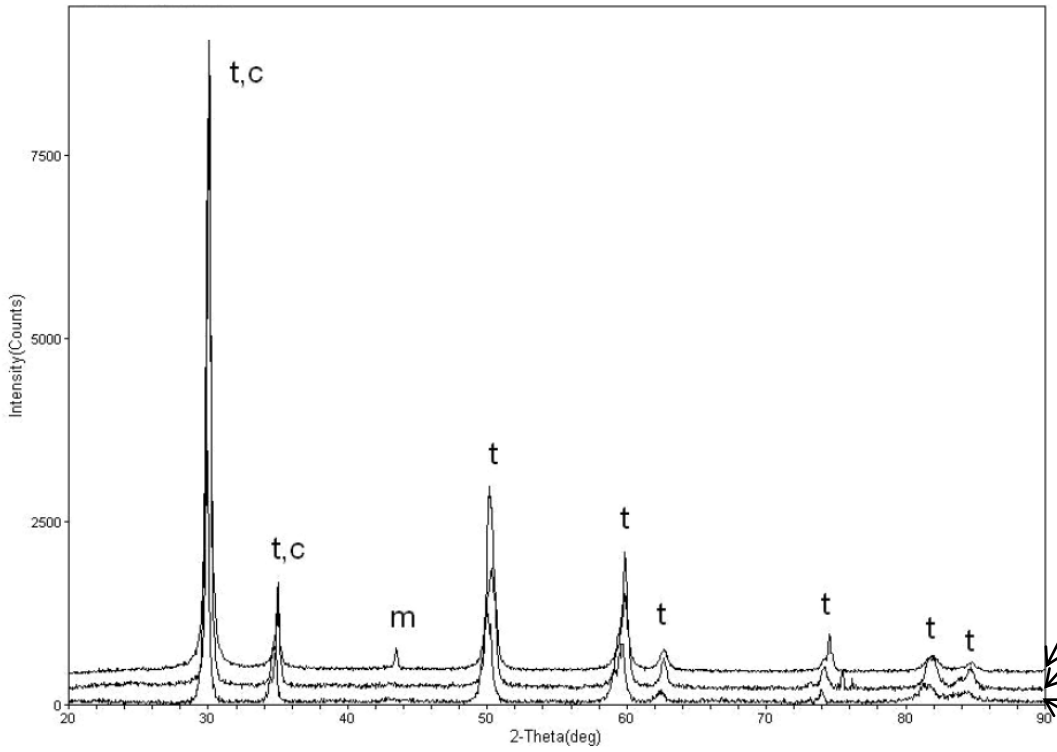


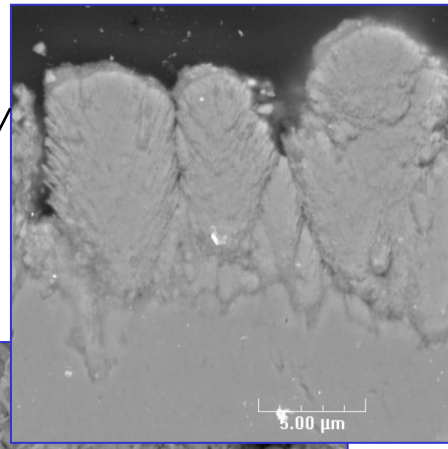


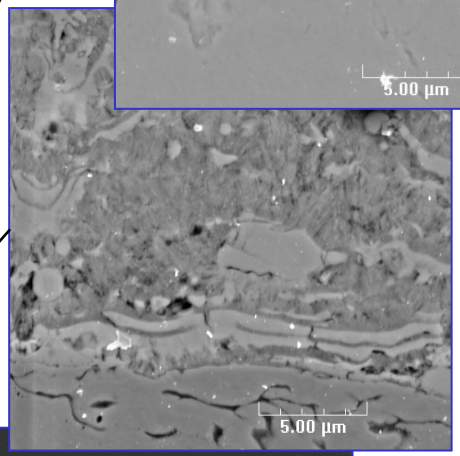


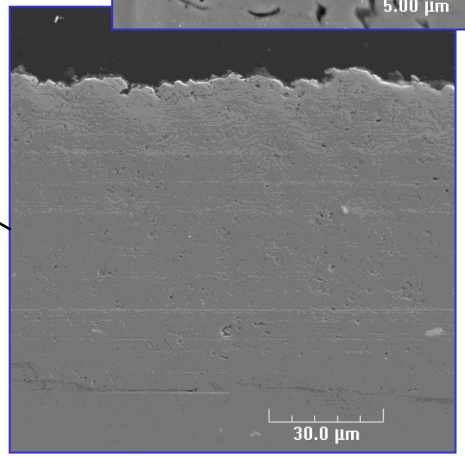
1800 A, 59Ar, 7H, 70He, 2.42 Torr, SD 45”, Shadowing

Similar crystallography observed in all coatings.









All three sprayed coatings have similar crystallography.
Almost all monoclinic YSZ transformed to tetragonal or cubic!
No evidence of amorphous phase found in any of the coatings!

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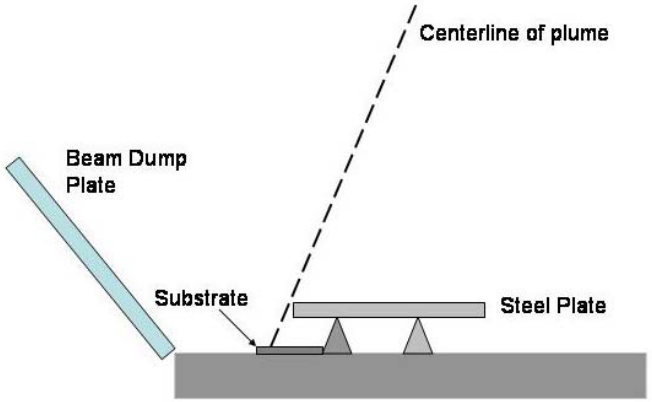
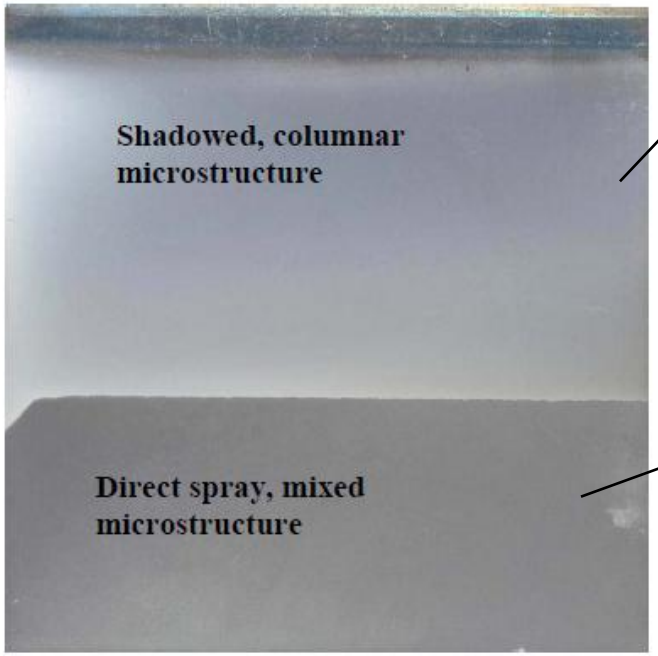
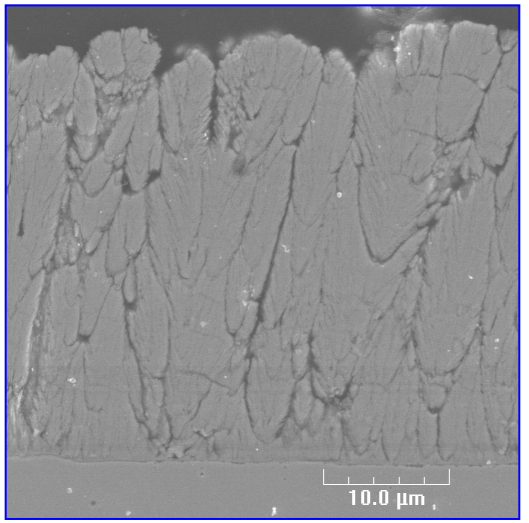
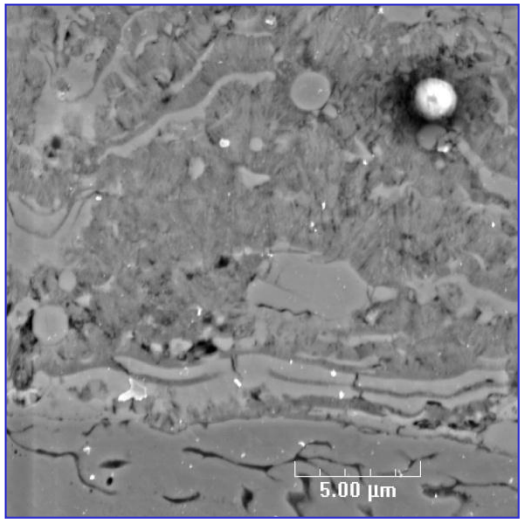
Simple Shadowing Experiment Proves a Multi-Phase Plume







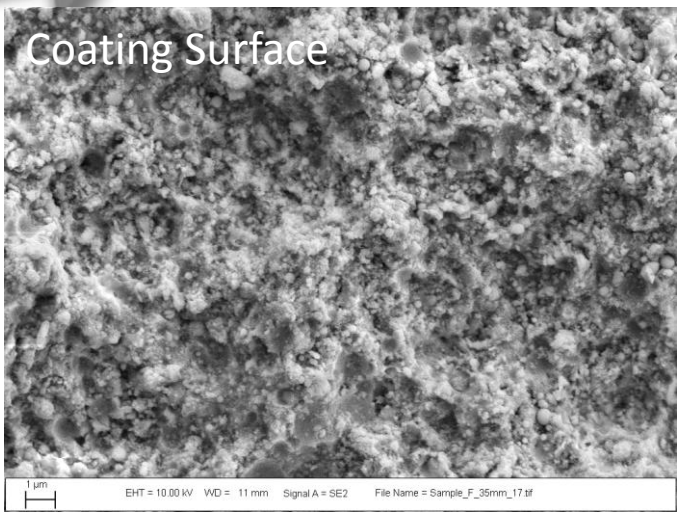


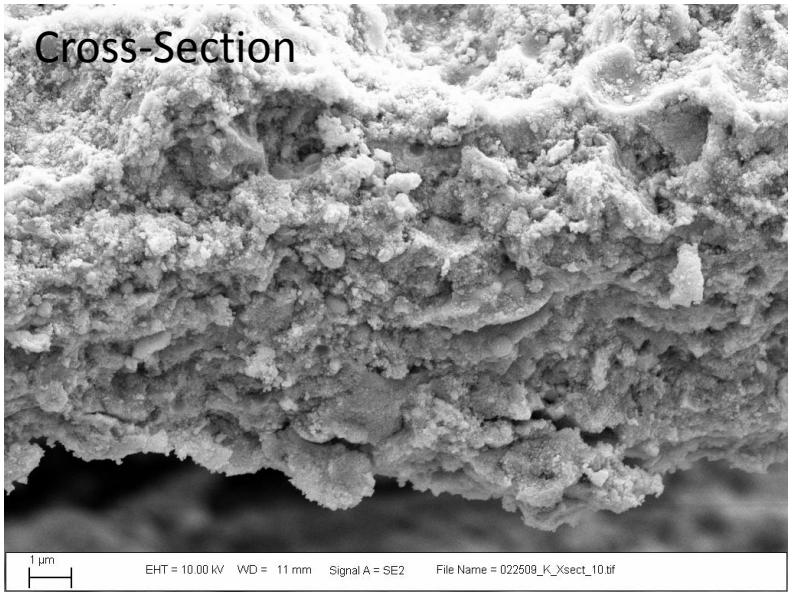
Suspension Feed System Used to Deliver Fine Powders to Sandia's LPPS System

- Low Pressure Plasma Spray Process allows for vapor deposition but small feedstock size is critical.
- SPS allows delivery of liquid feedstock suspensions containing sub-micron powders
- Smaller powders = better melting & higher vapor content plume

Suspension Feed System at TSRL →

Suspension Plasma Spray - LPPS YSZ



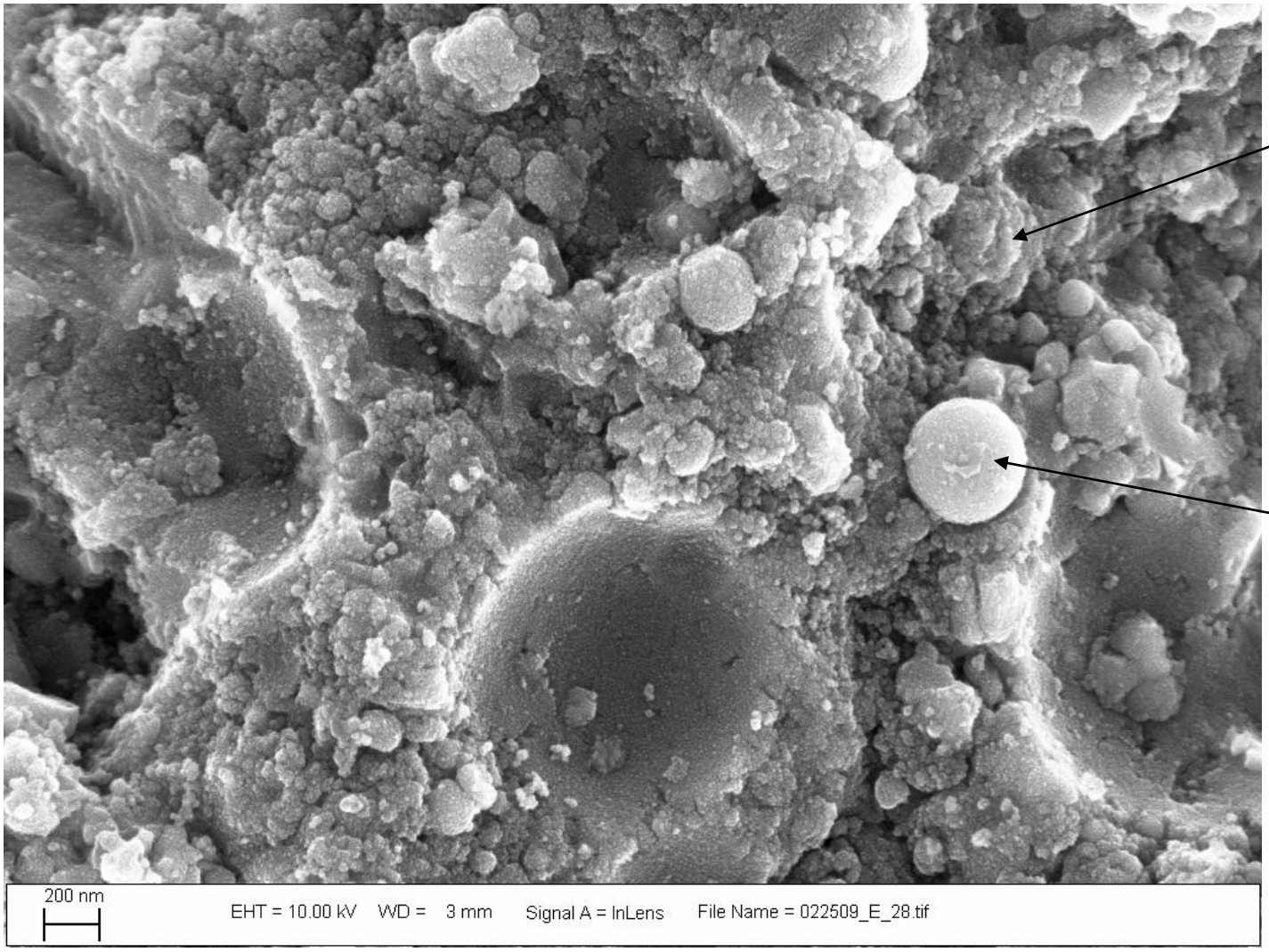


- 1 micron, Inframet powder, agglomerated, 4.5mol%Y in ZrO2
- Ethanol suspension: 1.0 v% YSZ, ~ 8wt% YSZ
- Ball mill for 4hrs min & sieve prior to loading in pressure pot
- 1800 amps, 59 Ar, 7 H, 70 He, 2.9 torr, SD 36-48”
- 230 micron sapphire orifice

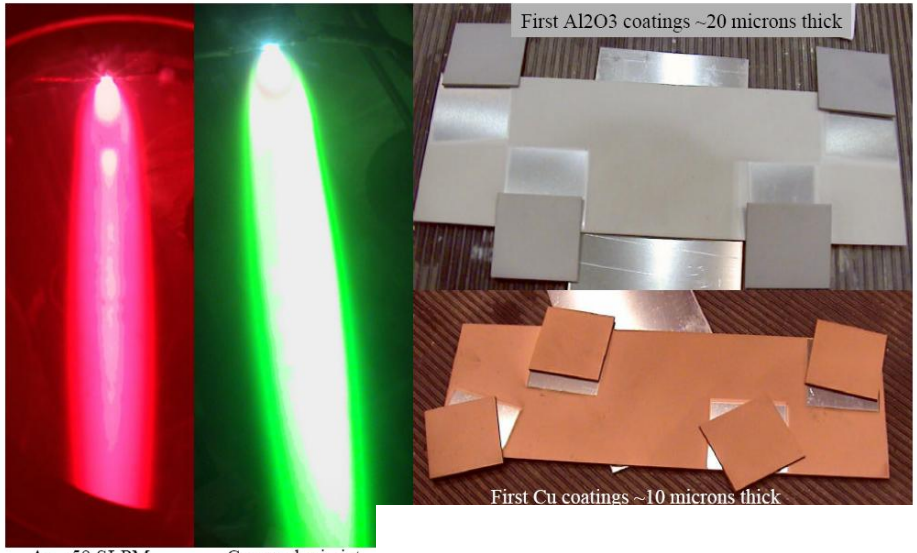
- Benefit:
  - SPS allows easy delivery of fine powders
- Cost
  - Ethanol in suspension quenches spray plume
  - Evaporating ethanol adds to gas load inside chamber (3.1 Torr)

Dense, Mixed Mode Coatings, > 30 microns thick!

Fine Structure in Suspension – LPPS YSZ fracture surface suggests growth from vapor

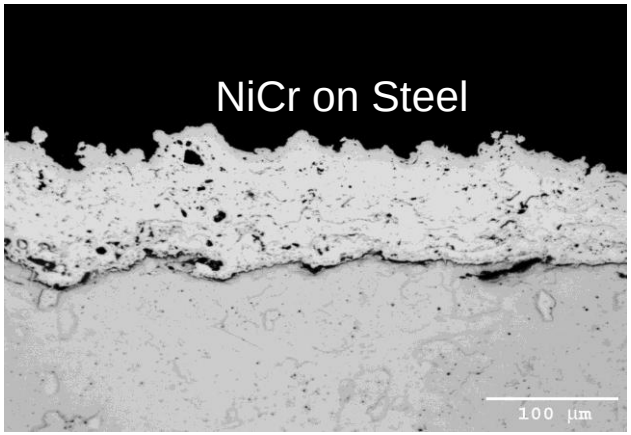


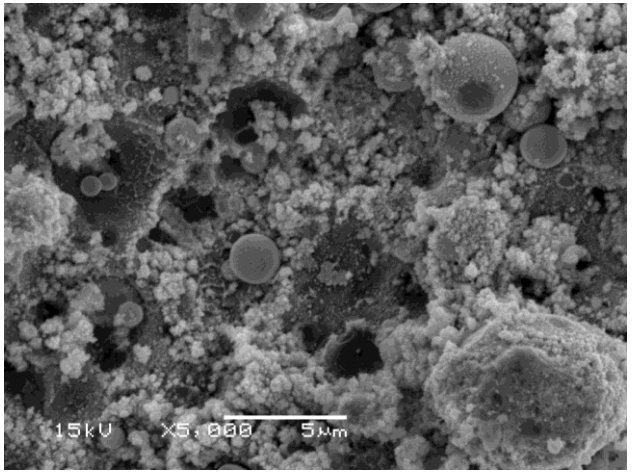
Materials other than YSZ have been sprayed using SNL's LPPS System.



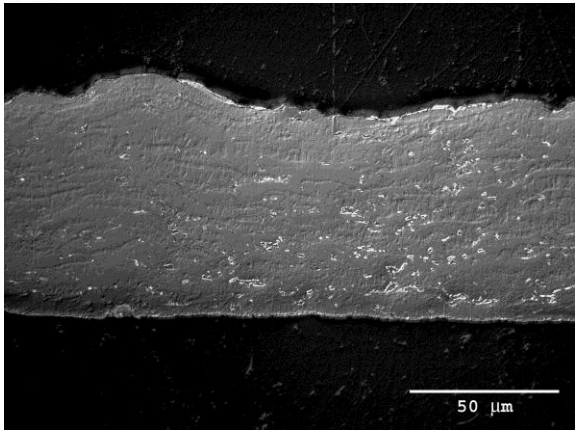
Ar – 59 SLPM
H2 – 7 SLPM
I – 1600A

Cu powder in jet

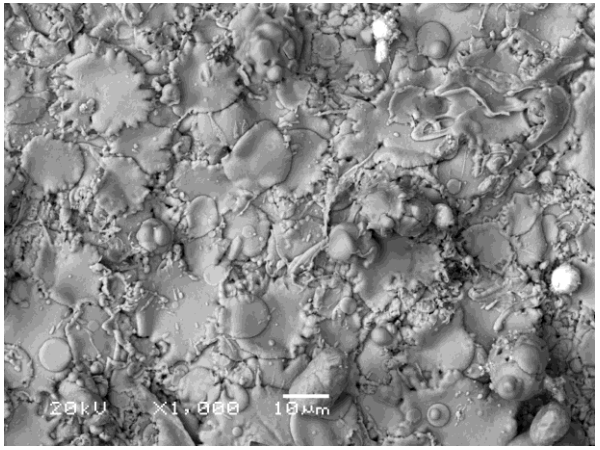




Tungsten (Mixed Mode Deposition)



Nickel (Droplet Deposition)



Copper (Droplet Deposition)

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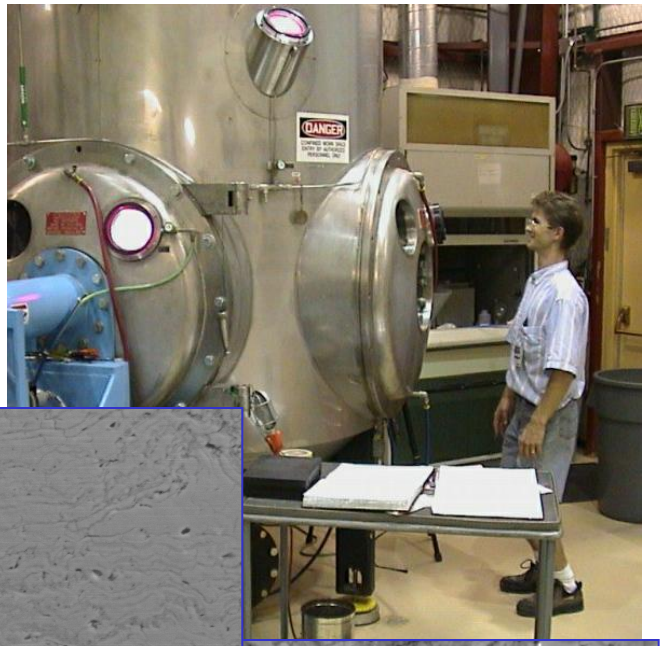
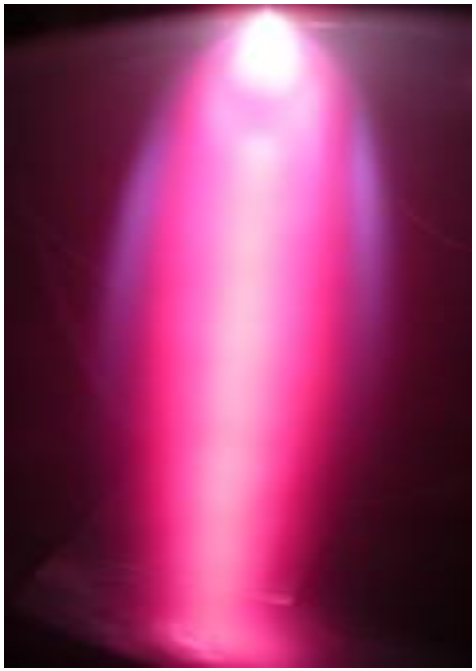
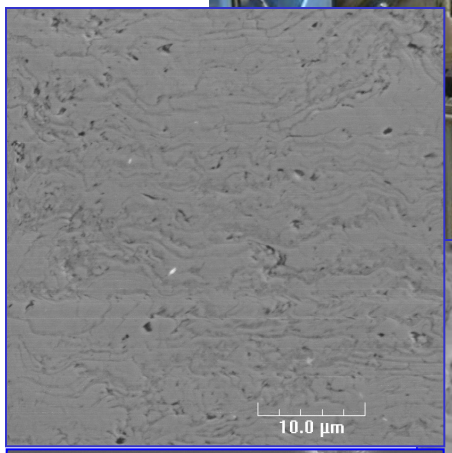
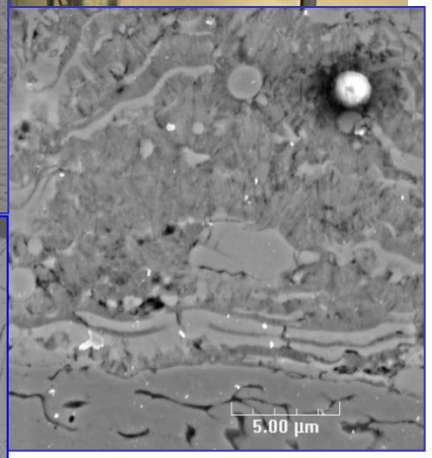
Summary

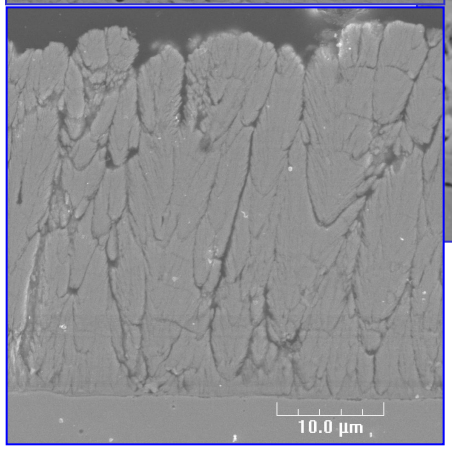
- Sandia's LPPS Thin Film system is fully functional and is being actively used to study YSZ coatings.
- Droplet, Mixed-Mode, and Vapor Deposition YSZ coatings have been demonstrated.
- Suspension Feedstock Injection has been used successfully to prepare YSZ coatings.











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Questions?

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This work was also sponsored by the United States Department of Energy under Contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy.

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