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

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Dr. Aaron C. Hall, Dr. Rodney Williamson,
James Mccloskey, David Urrea,
Sandia National Laboratories

Andrew Mayer, Timothy Roemer, David Beatty
Ktech Corporation

Dr. Deidre Hirschfeld, Nicholas Spinhirne
New Mexico Institute of Mining and Technology

Erich Muehlberger
Sulzer-Metco

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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.

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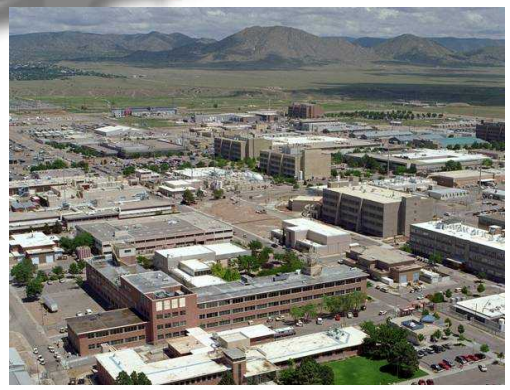

Albuquerque, New Mexico

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

Yucca Mountain, Nevada

Kauai Test Facility, Hawaii

Tonopah Test Range, Nevada

WIPP, New Mexico

Pantex, Texas

Livermore, California

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National Nuclear Security Administration

LOCKHEED MARTIN

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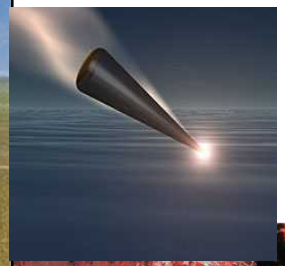
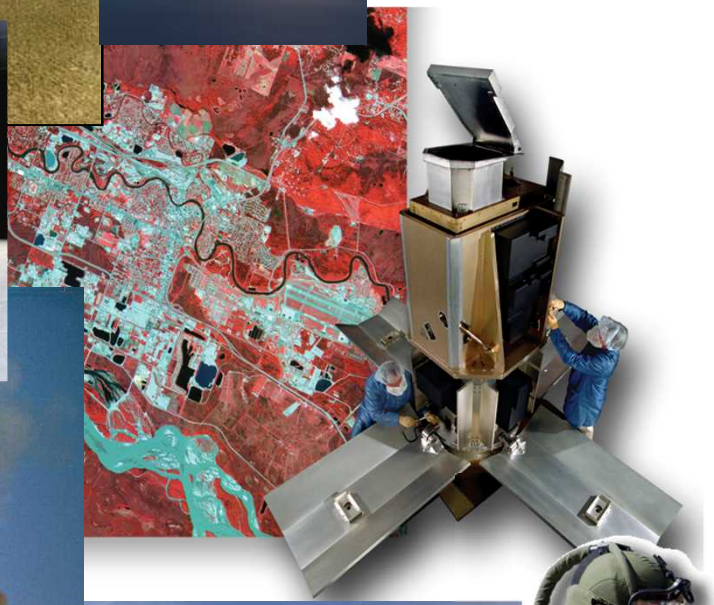
Sandia is a U.S. National Security Laboratory

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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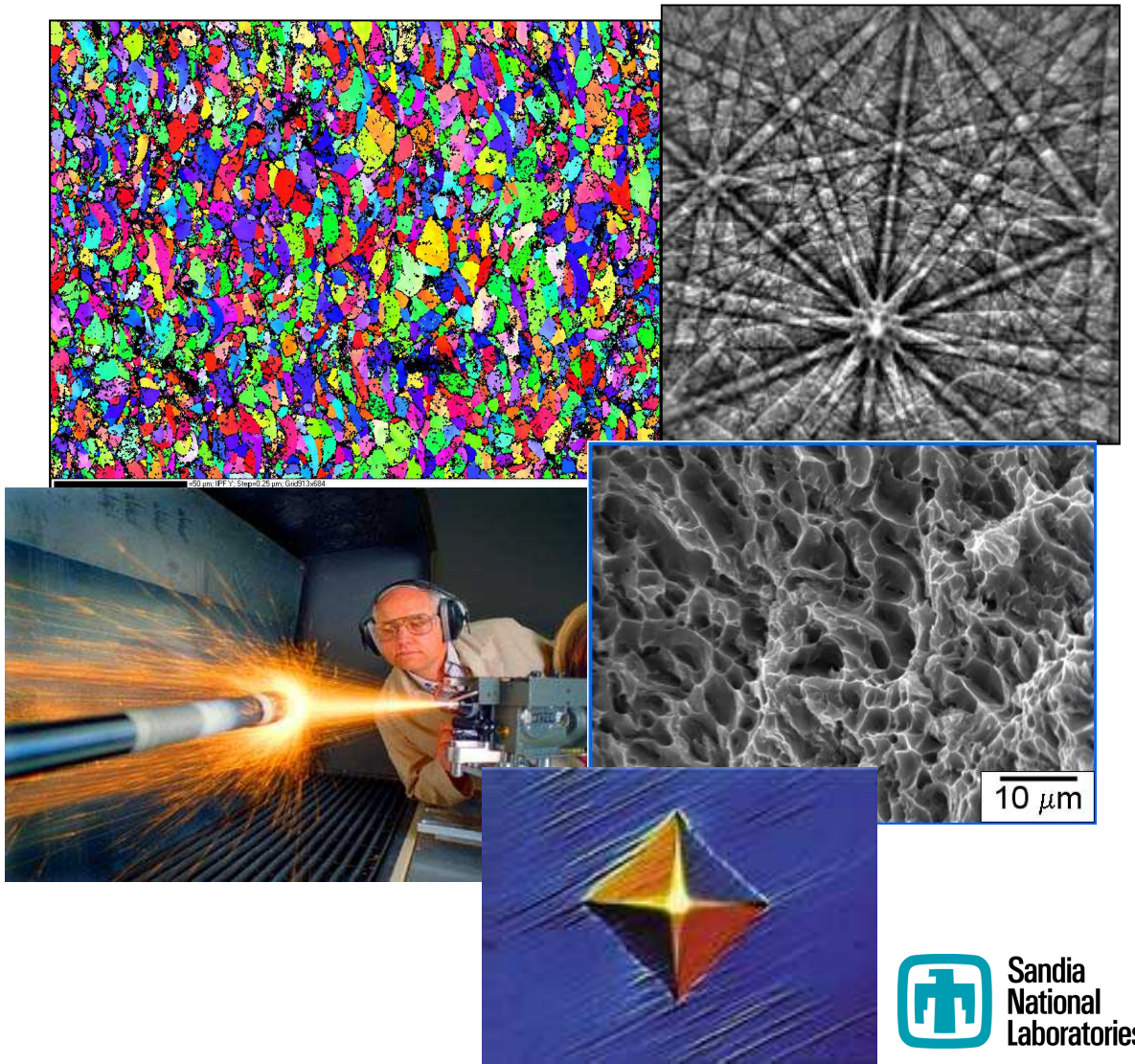
Sandia National Laboratories

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



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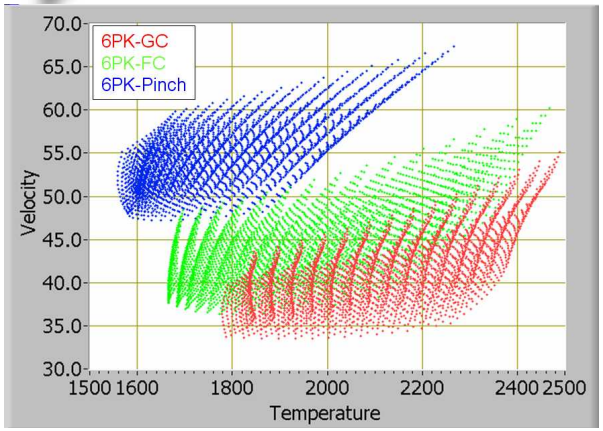
Sandia National Laboratories

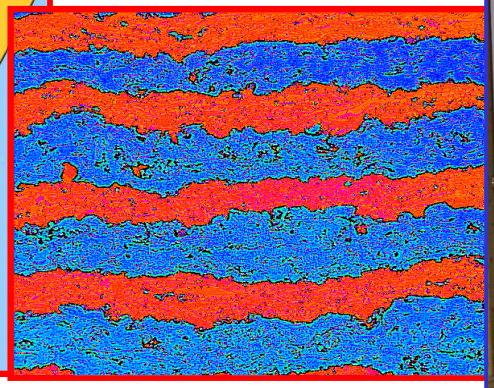
What is Sandia's Thermal Spray Research Laboratory (TSRL)?



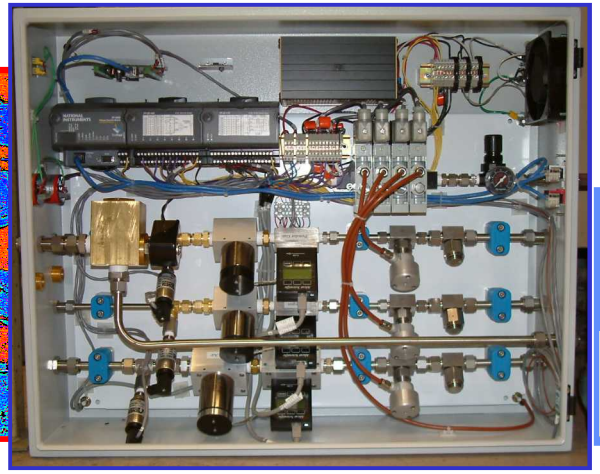
We focus on process diagnostics for thermal spray.

- A team of scientists & engineers dedicated to understanding *process-microstructure-property* relationships in thermal sprayed coatings
- Part of SNL's Materials Science and Engineering Center
- Primary resource for thermal spray coatings within the U.S. Department of Energy

We team to model spray processes

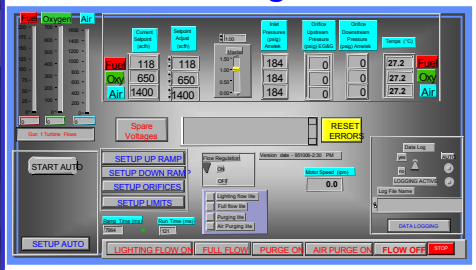


We design coatings & conduct materials R&D



We develop custom control technology for spray applications

We support Sandia production when it involves thermal spray coatings



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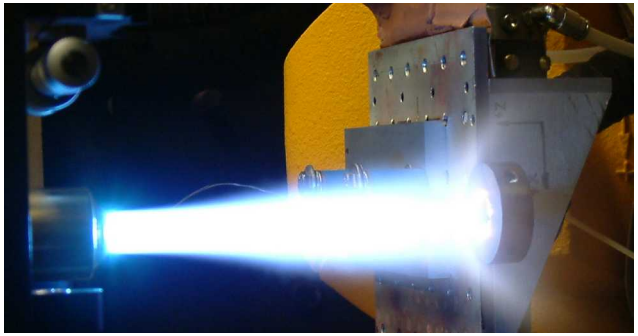
The TSRL operates at least one version of every major spray process.

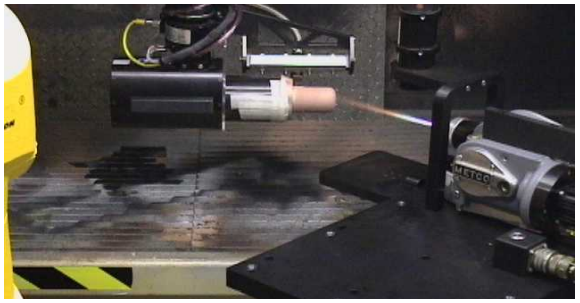
Atmospheric Plasma Spray

Twin Wire Arc Spray

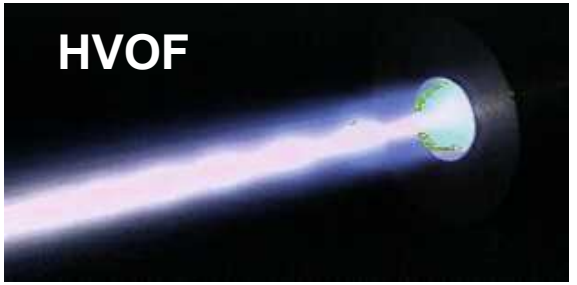


Powder Flame Spray

Vacuum, controlled atmosphere, and low pressure plasma spray



Wire Flame Spray



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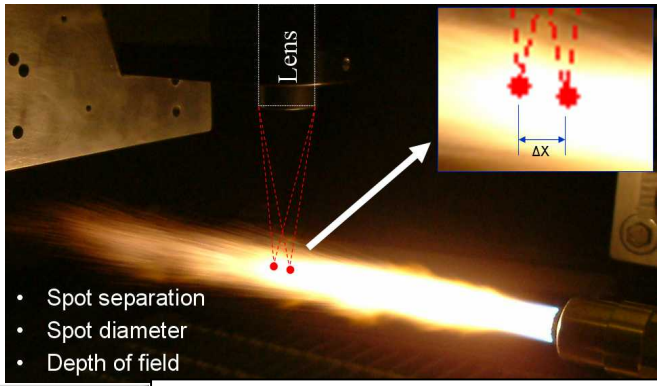
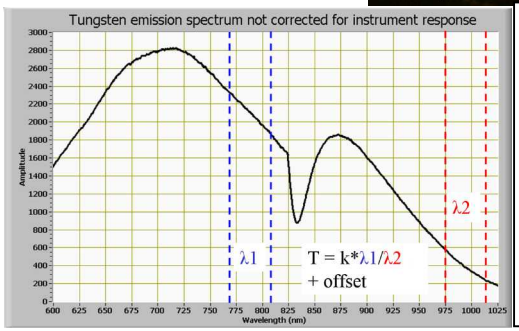
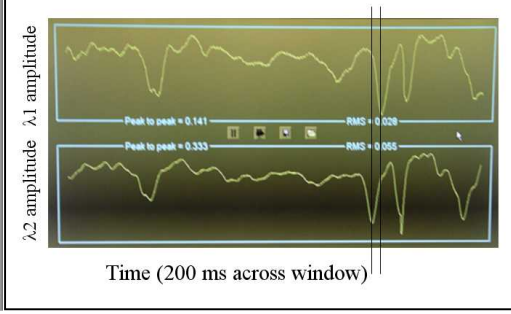
What makes the TSRL unique?

- **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
- Materials Science Expertise and Analysis Capability
- Strong Ties to Materials & Process Modeling at SNL
- LPPS® Thin Film & Cold Spray

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

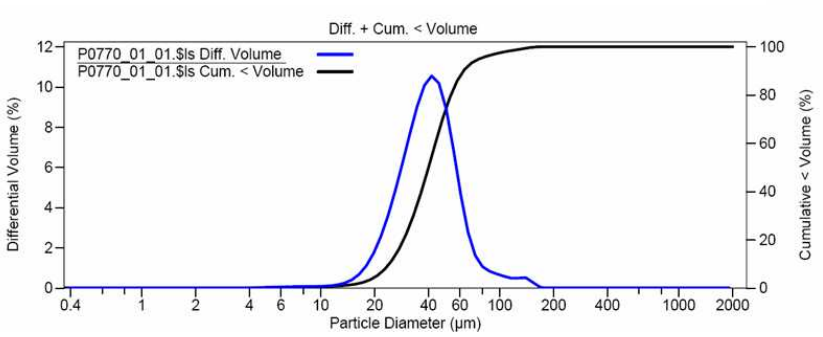




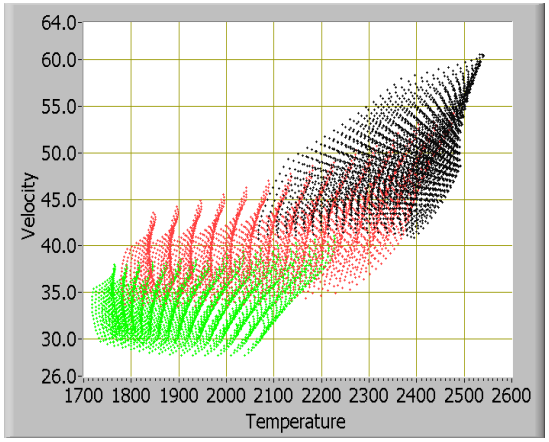


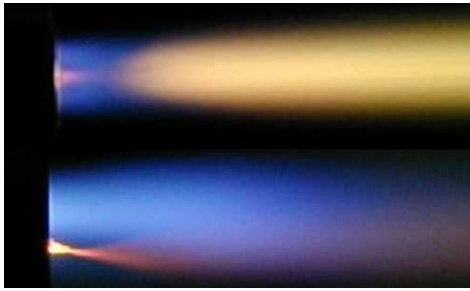
Tp ∝ λ1/ λ2

Vp = Δx /Δt



Powder particle size distribution





Wire tip imaging

Accessible Tp, Vp space with different torch hardware

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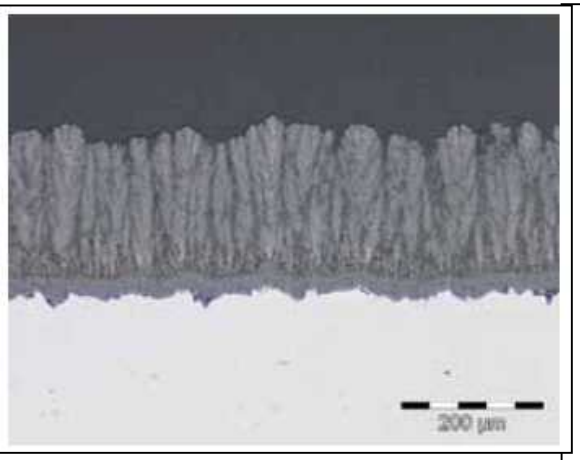
What is LPPS Thin Film®?

Low Pressure Plasma Spray

- Steady State Vacuum Plasma Spray at chamber pressures < 20 Torr
- 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*



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

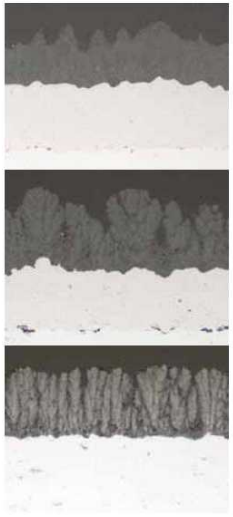


Fig. 5. LPPS-TF deposited TBC layers with increasing "degree of columnarity" and about 100 -150 µm thickness (low-, medium- and high columnarity).

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

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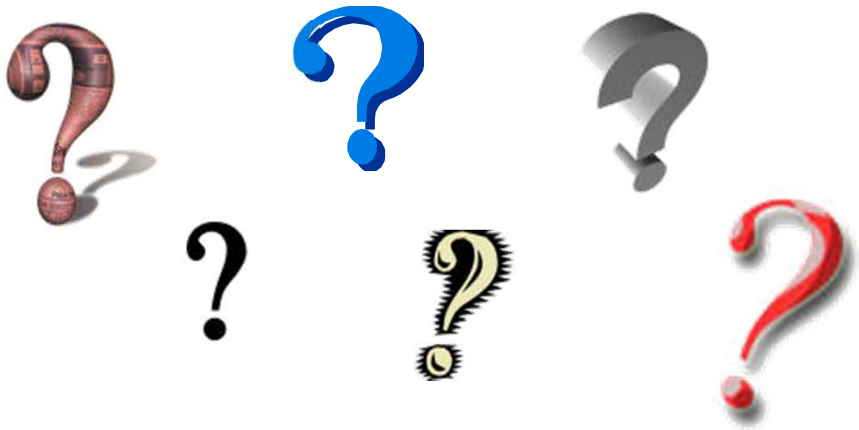
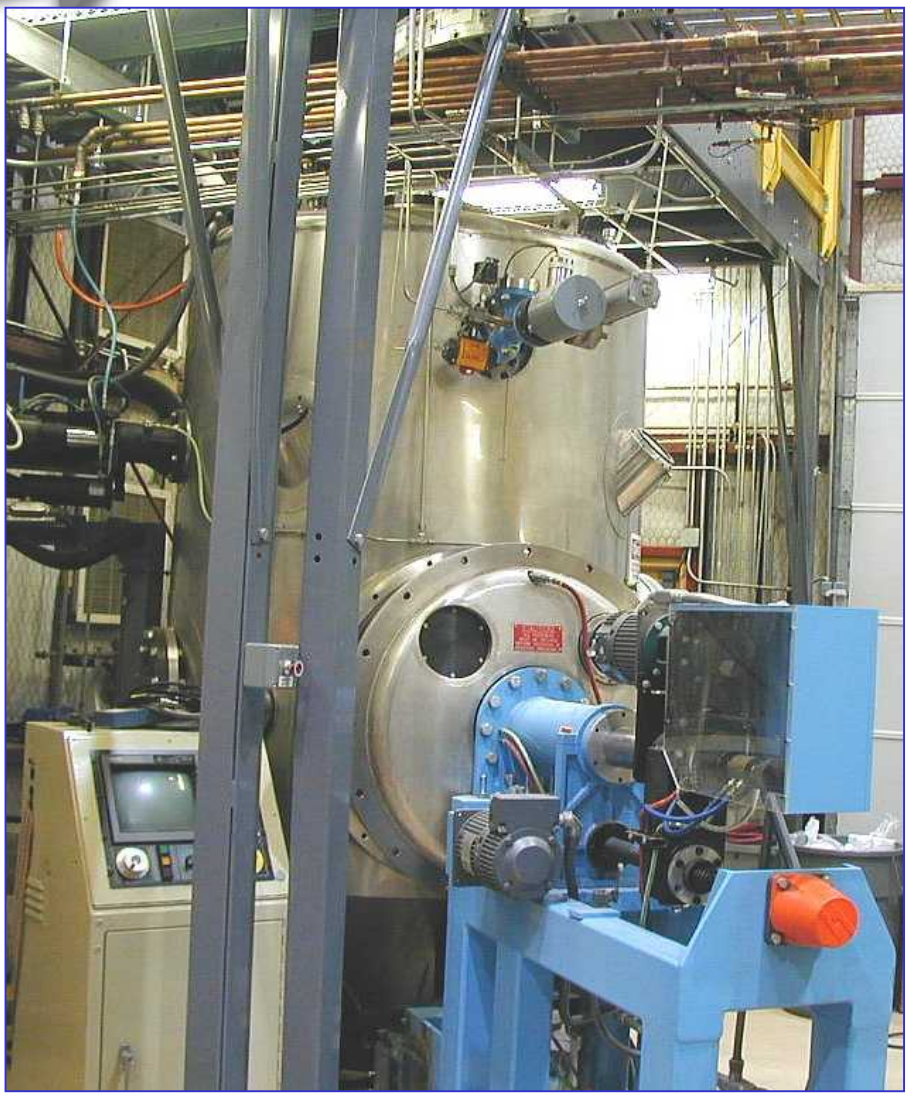
Why is SNL interested?

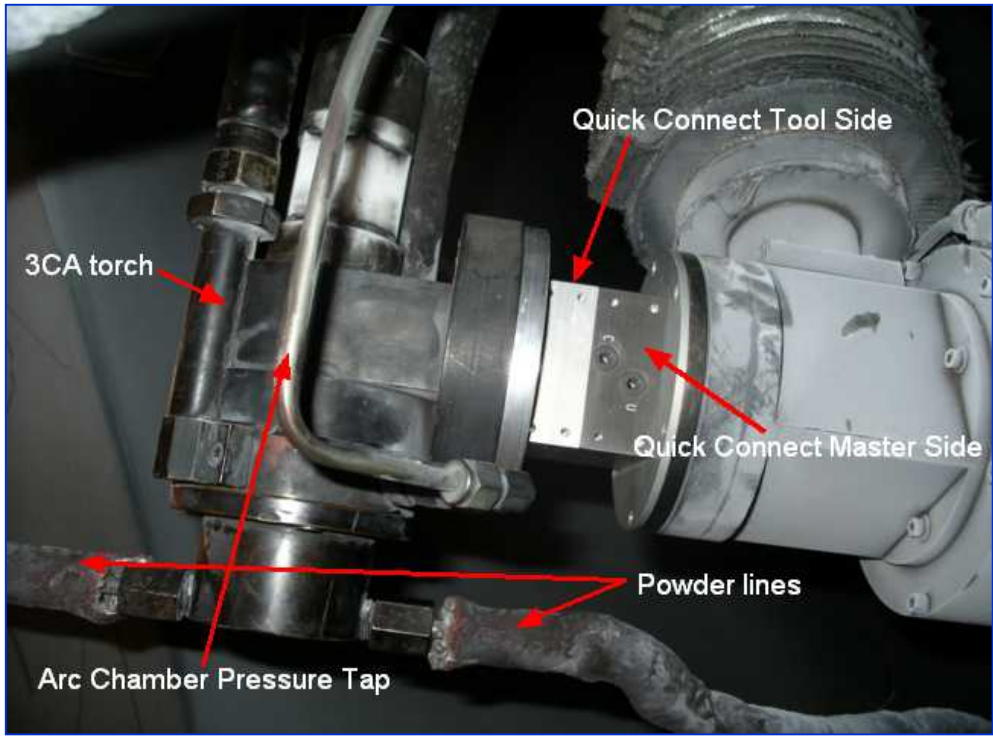
- Fundamentally new spray process
- Unique deposition mechanisms
- Unique microstructures
- Unique plasma physics
- Thinner coatings than any other spray process
- Lots of potential for good science…



LPPS Plume at Sandia

Sandia's LPPS System: 03C Plasma torch; 2000A, 50V Halmar Power Supply, & an ~ 1000 L^3 Chamber





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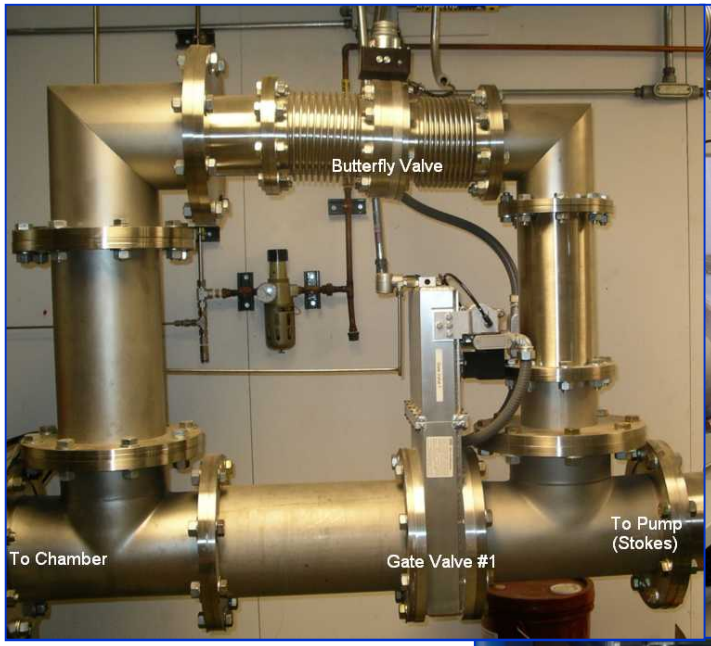
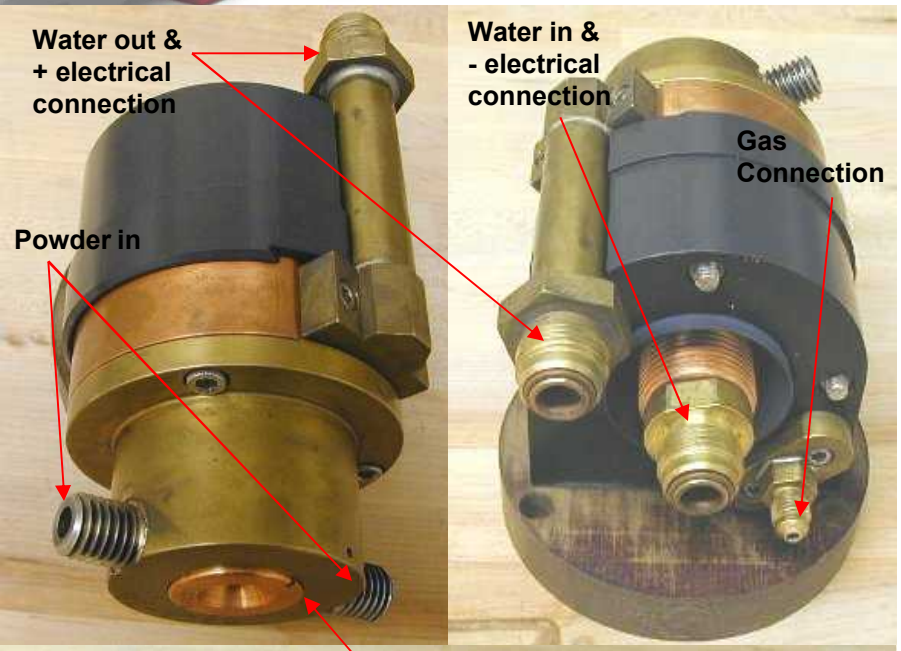
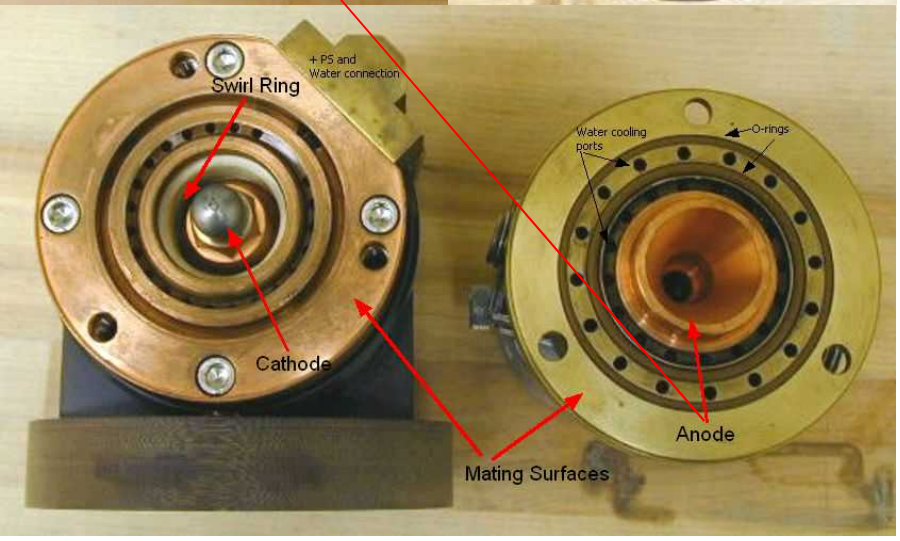
Sandia's LPPS System is outfitted with two vacuum pumping systems allowing torch operation at pressures below 1 Torr

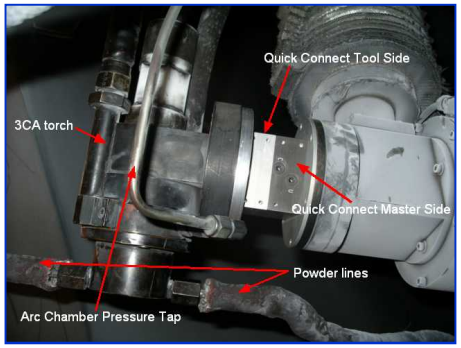


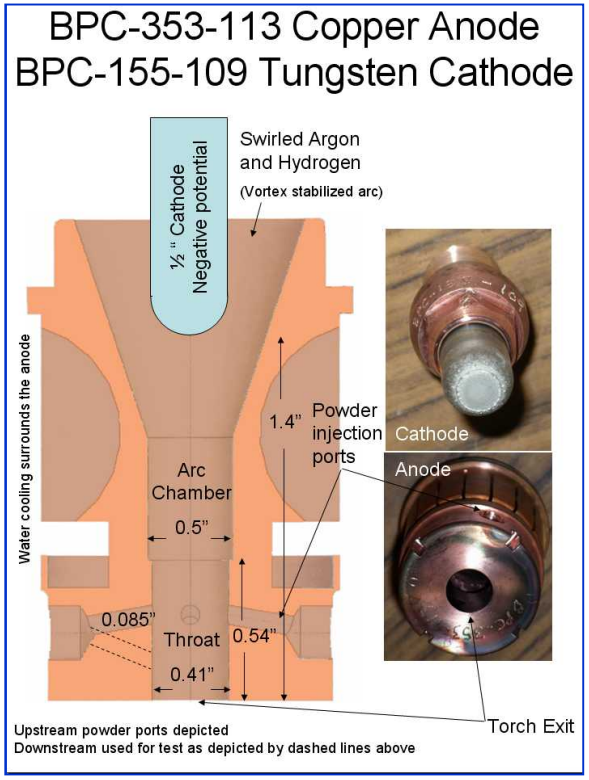
- Butterfly valve is used to throttle pumping system.
- Feedback control maintains operating pressure between 1 Torr to ~ 625 Torr (Atmospheric in Albuquerque, NM)

03C Sulzer-Metco Plasma Torch









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Anode wear after 30 minutes was an issue with early anode geometries.

- **Arc Current ~1800 amps, Ar/H Plasma**
- **Both runs show wear at two distinct locations indicating attached arc.**
- **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

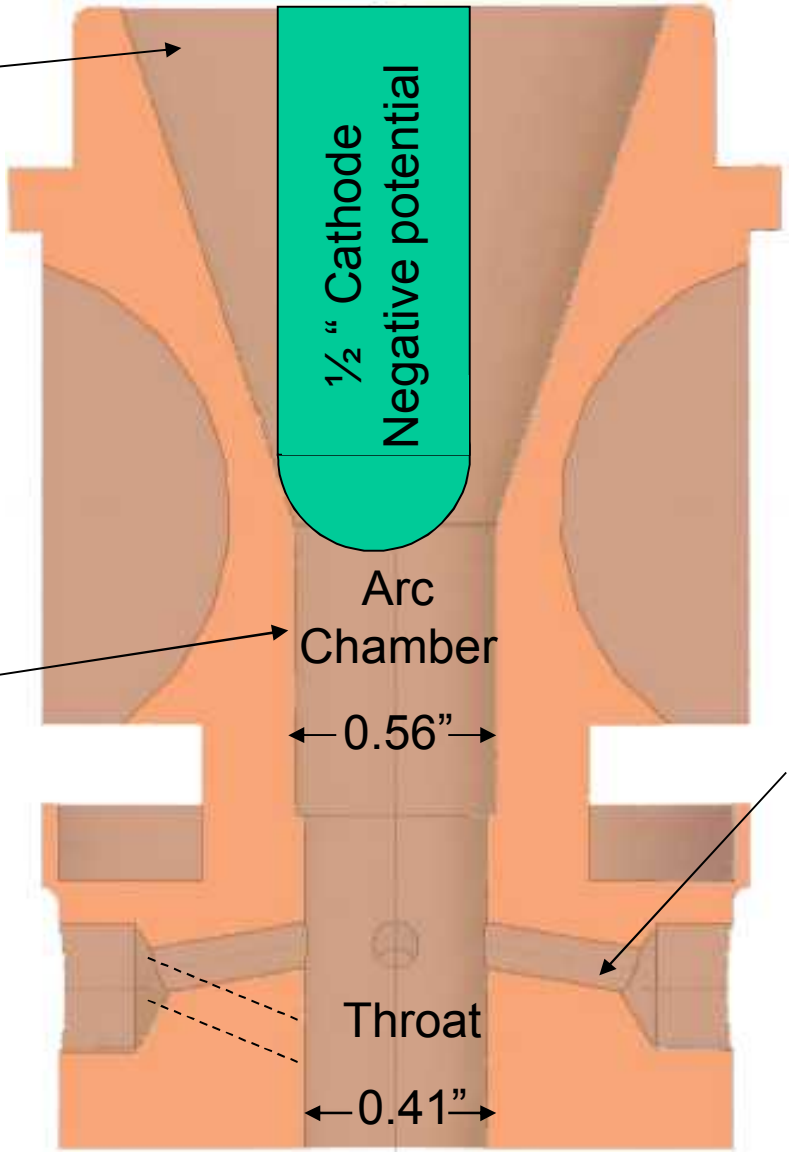
2nd run 0.525” arc chamber, 0.41” throat: 38 minutes

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

Swirled Argon and Hydrogen

(Vortex stabilized arc)

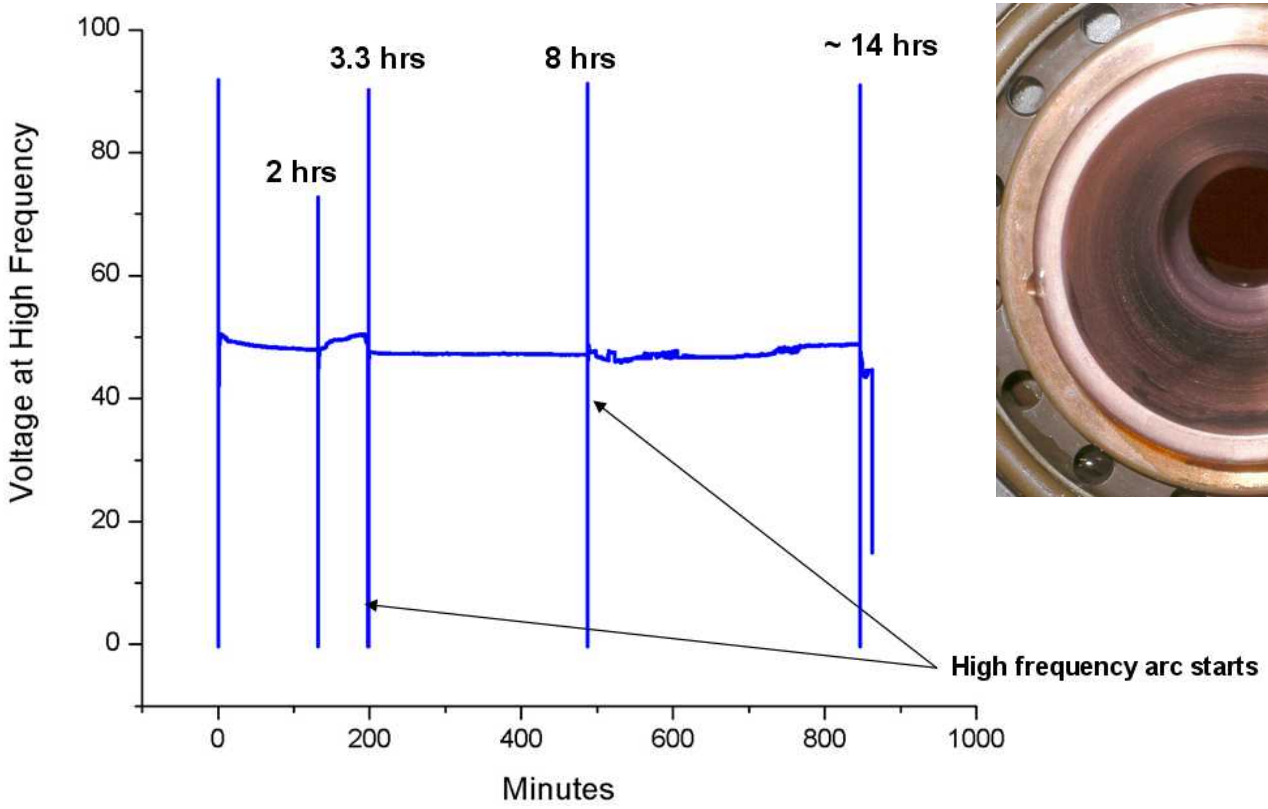
Larger arc chamber results in lower energy density inside arc chamber and less anode wear.



Sandia's LPPS system has been operated for 8 hours using a single anode.

- **BPC-353 Anode with BPC-155 Cathode**
- **0.56" Arc Chamber**
- **0.41" Throat**
- **Current: 1750 Amps**
- **Main Argon: 59 SLPM**
- **Main Hydrogen: 7.0 SLPM**
- **Powder Gas (per each of the 2 sides): 12.5 SLPM**
- **Powder Gas Composition: Argon**
- **Total Gas Flow Through Torch: 91 SLPM**
- **Vacuum Chamber Pressure: ~1.3 torr**
- **Water Temperature Rise: 28.5° F (15.8° C)**
- **Water Flow Rate: 9.5 gpm**
- **Resulting Voltage: ~43 Volts**
- **Resulting Arc Chamber Pressure: ~9.5 PSIA**
- **Visible Plume Diameter: ~30 cm**
- **Visible Plume Length: > 1.3 m**
- **Tests conducted in Albuquerque NM. Ambient pressure 12.18 PSIA**

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



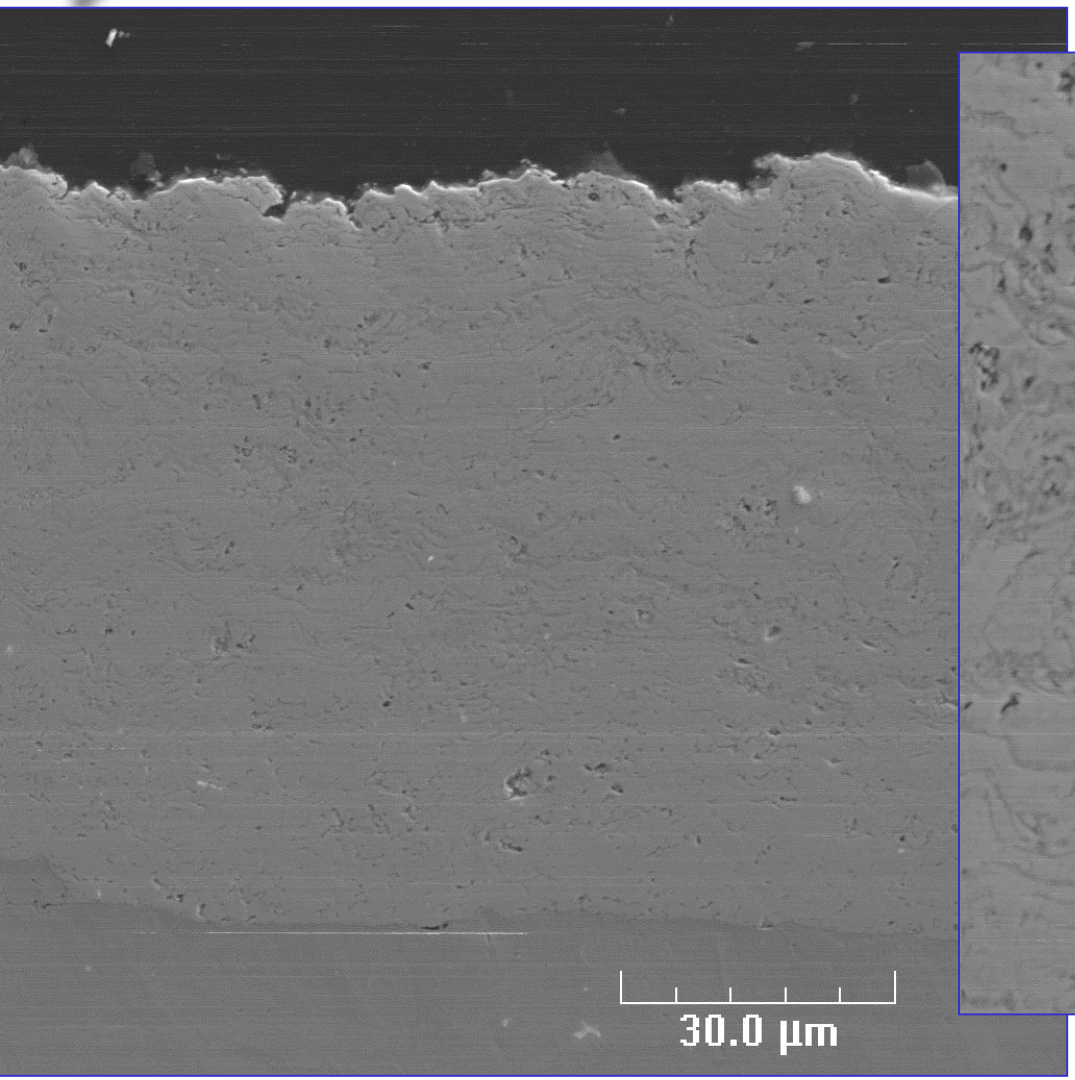
3.3 hours

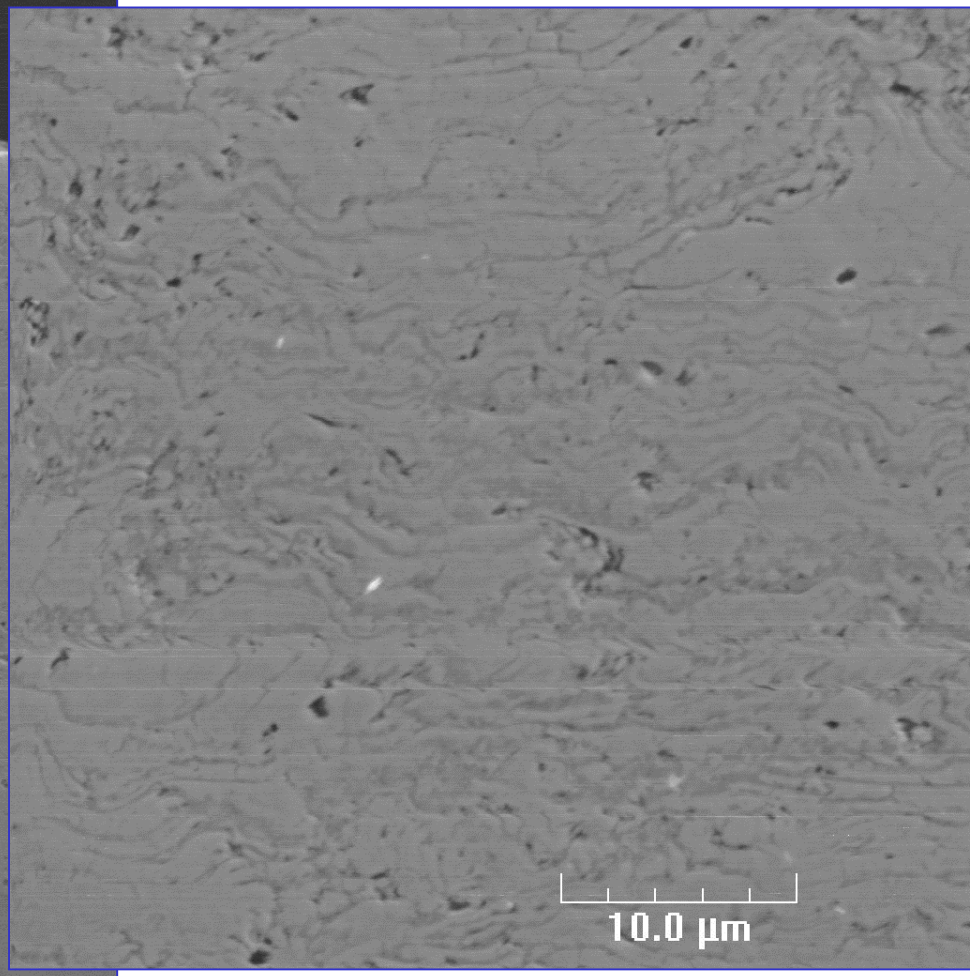
The arc normally circulates about half way down the arc chamber

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

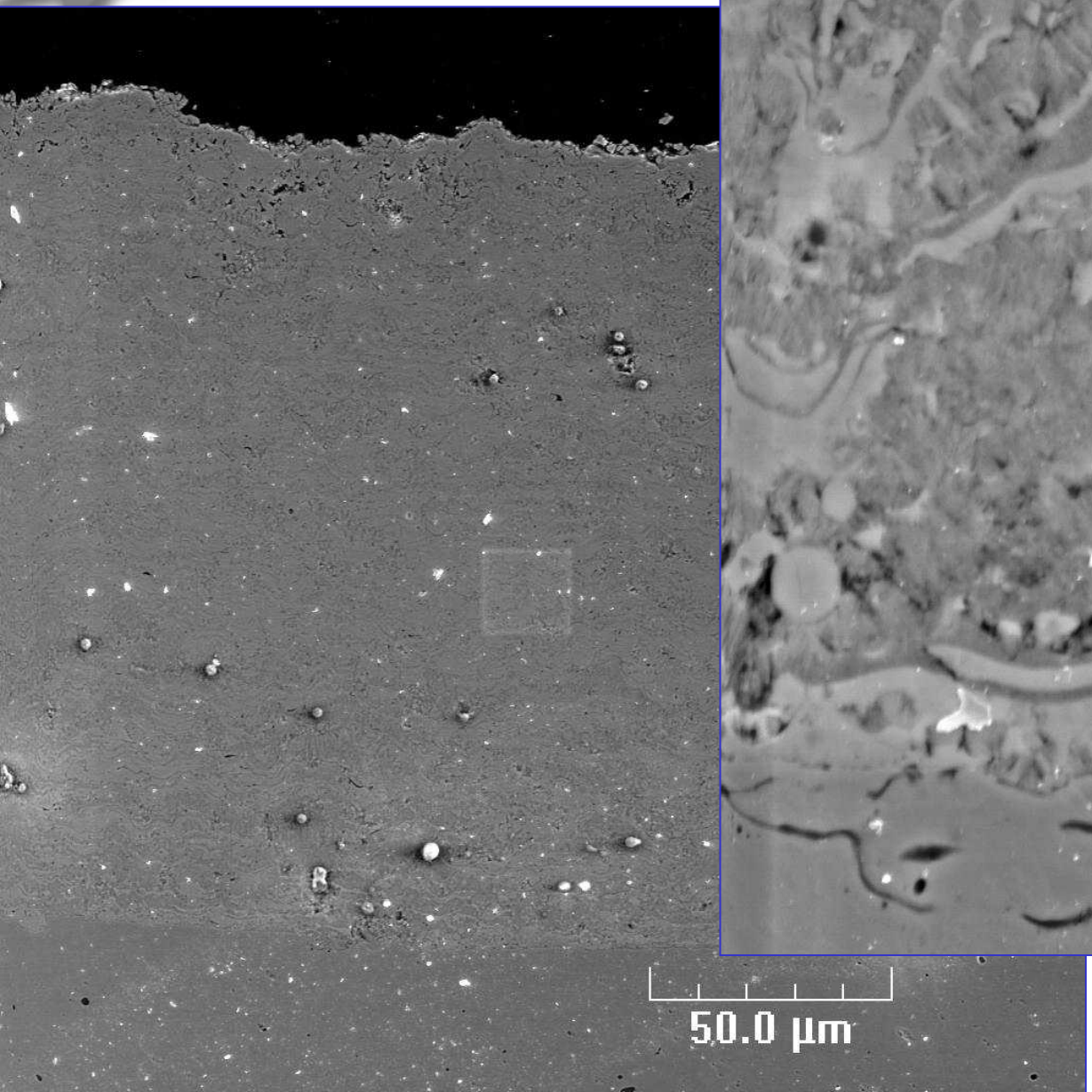
Arc chamber still intact and showing little wear

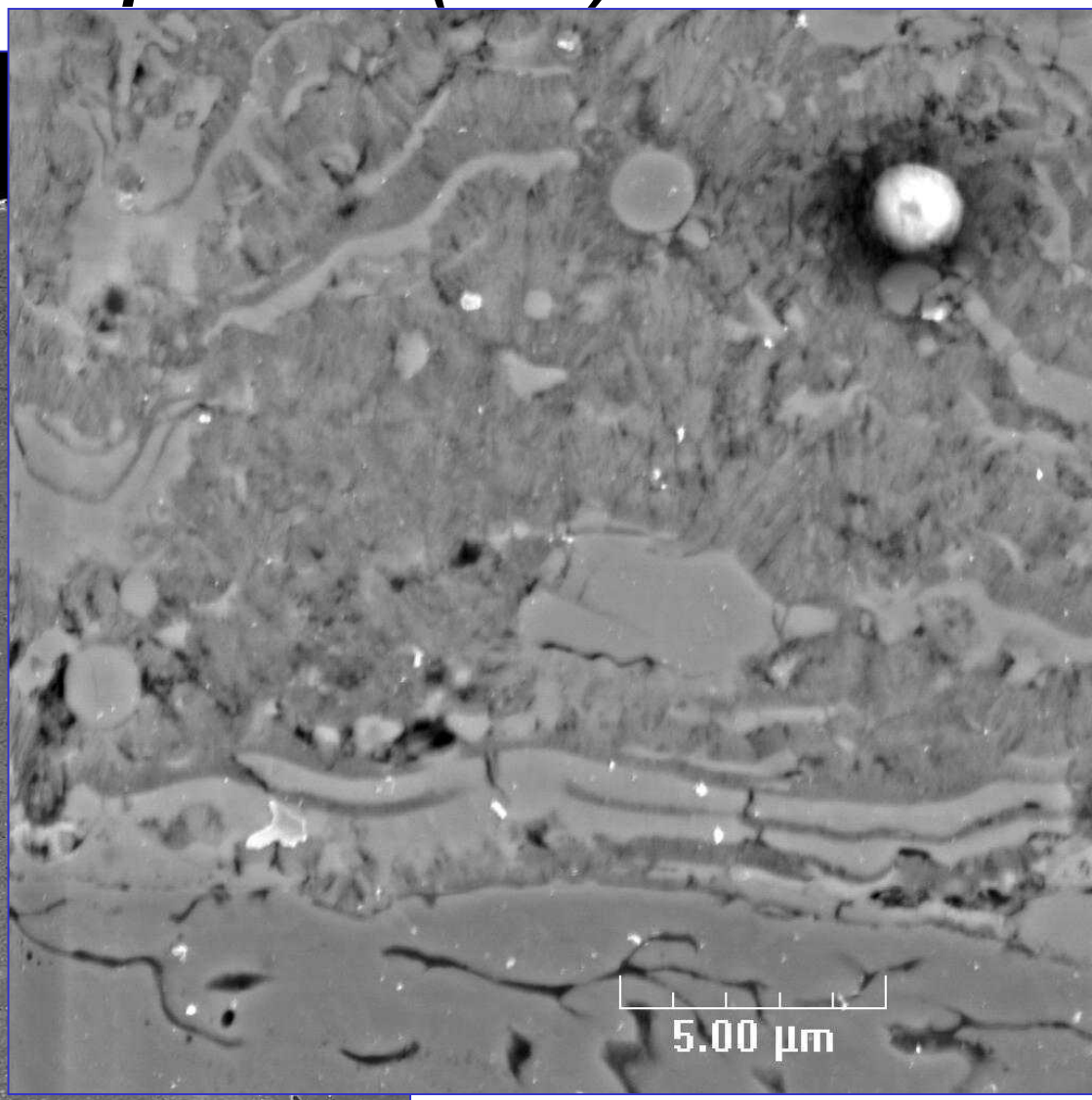
Droplet Deposition (YSZ)





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





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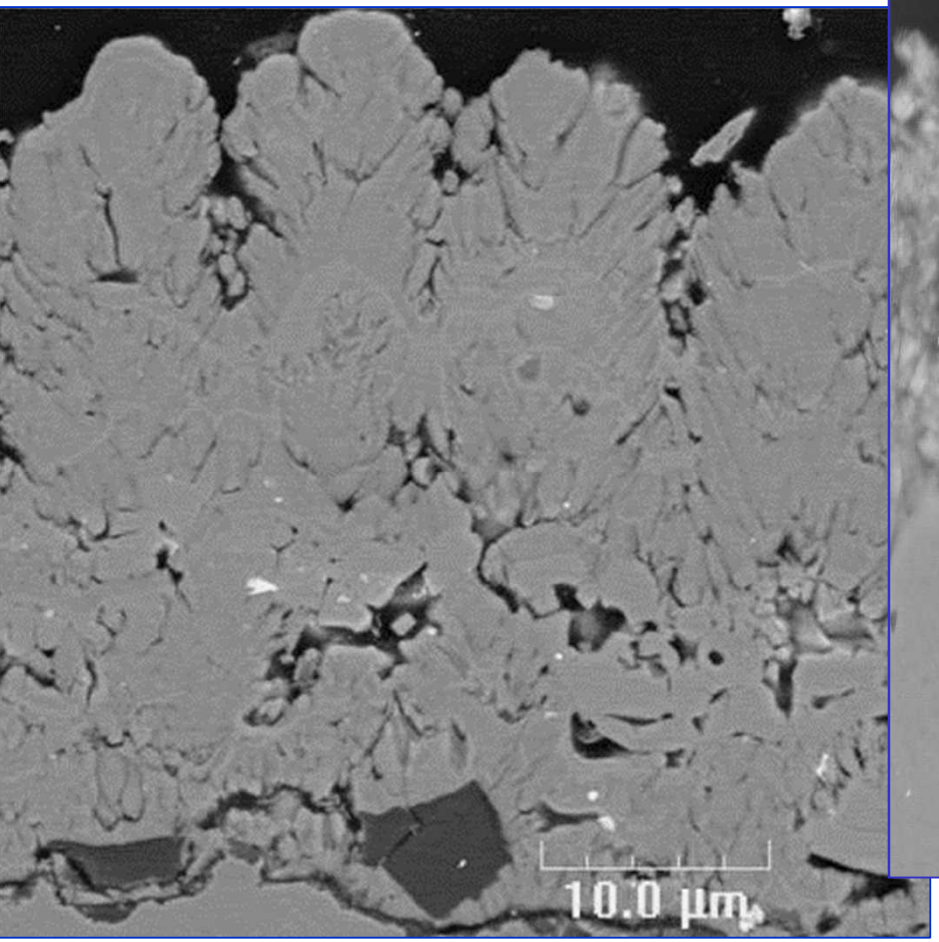
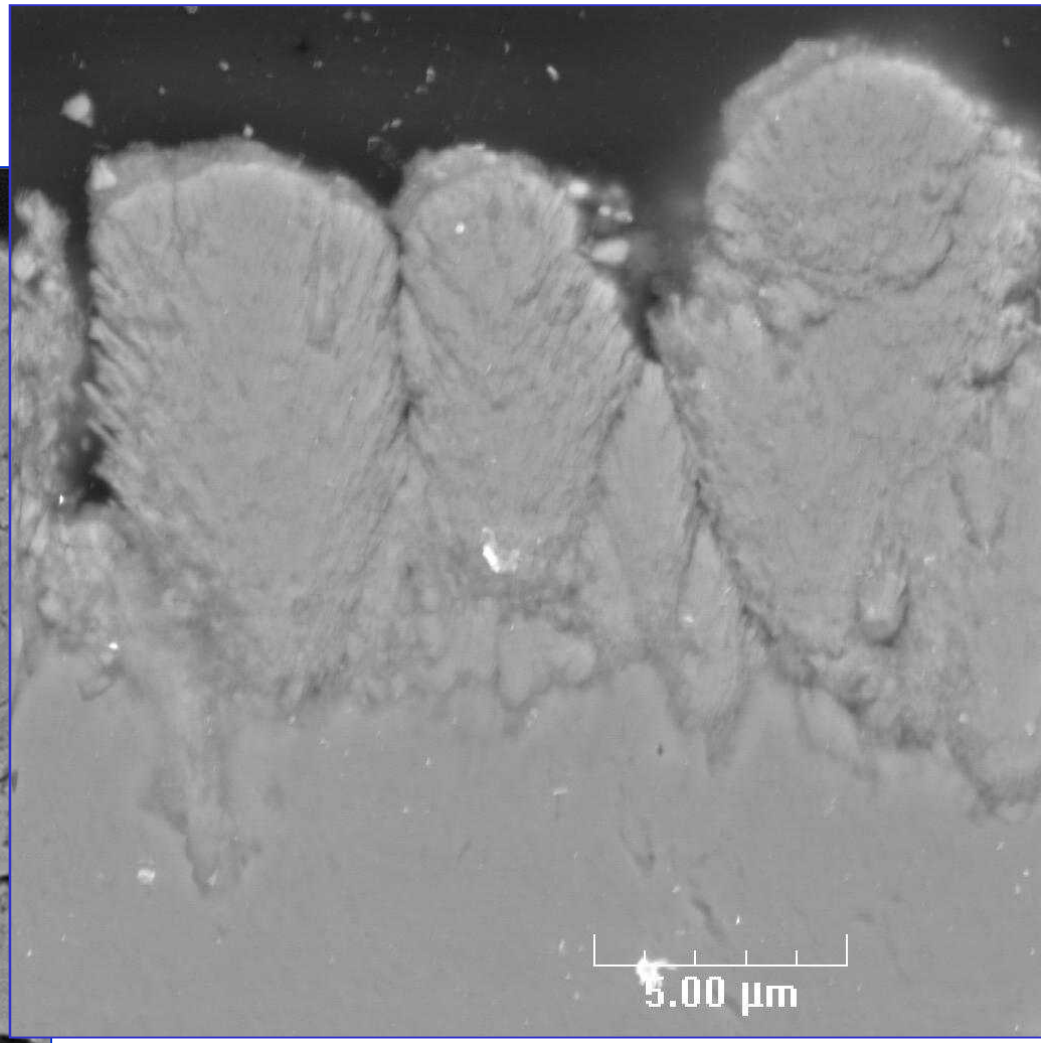
Vapor Growth (YSZ)



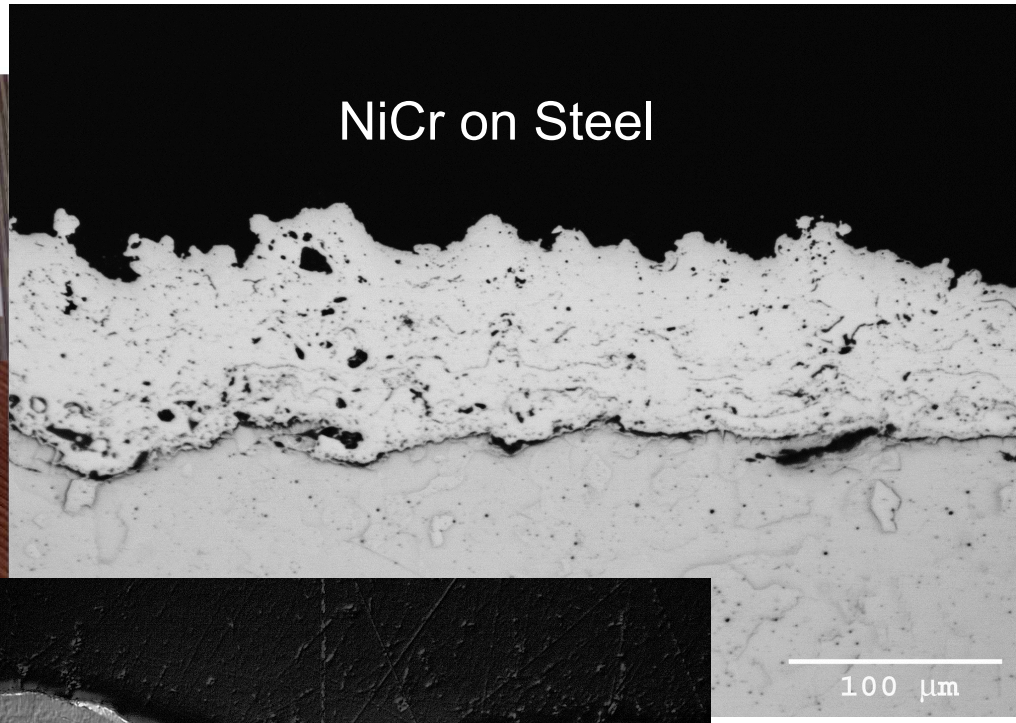


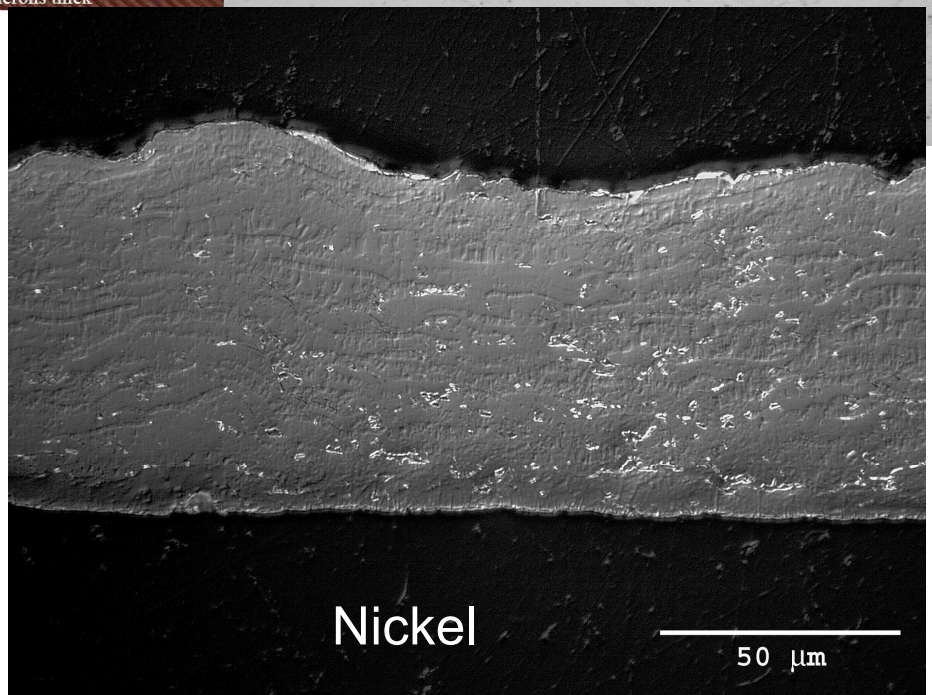
Materials other than YSZ have been sprayed using SNL's LPPS System.

Ar – 59 SLPM
H2 – 7 SLPM
I – 1600A
2.6 Torr
49.2 V, 78.8 KW

Cu powder in jet

CAPS Thin Film – First Coatings





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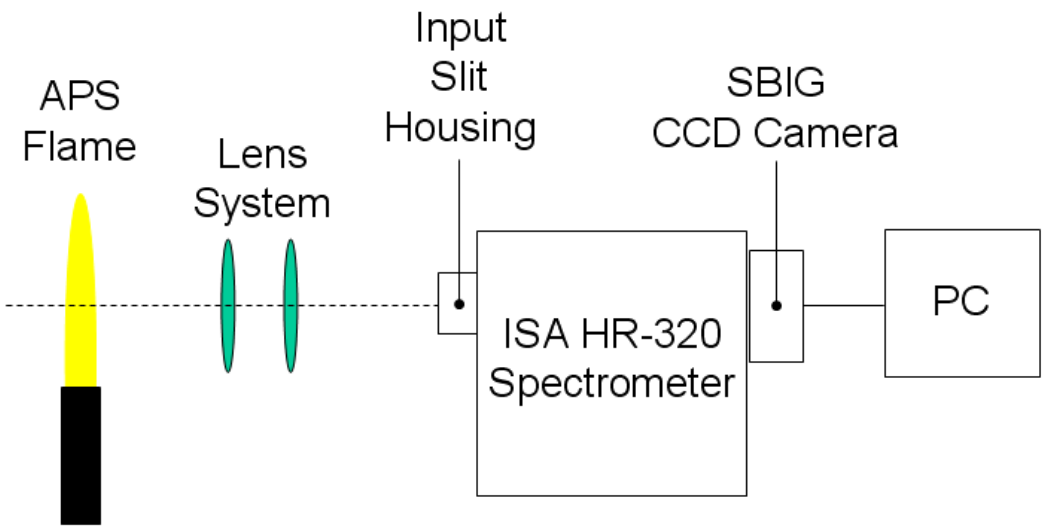
Plasma temperature measurement using optical spectroscopy

Objective:

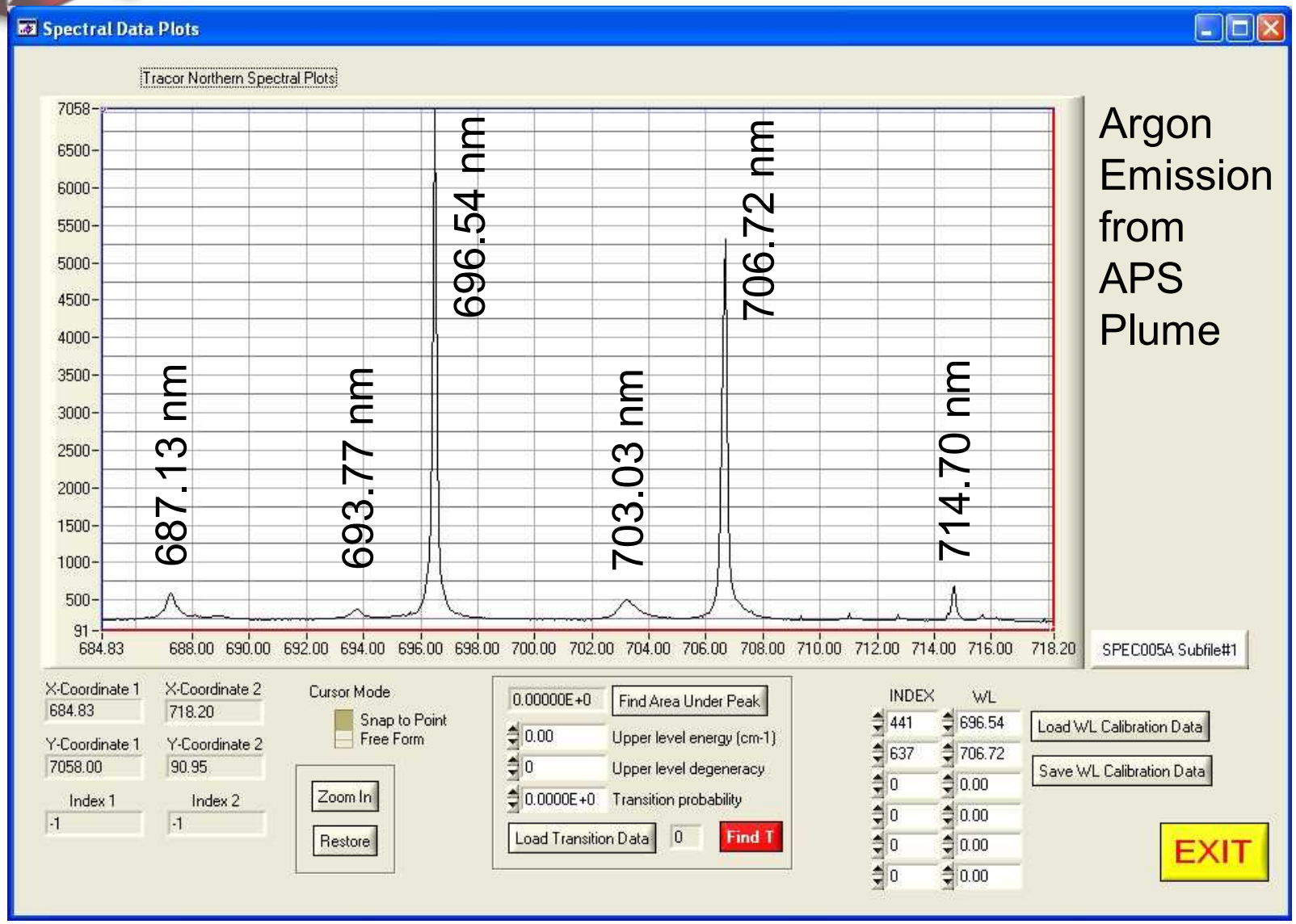
- Develop a laboratory diagnostic tool to measure the radial temperature distribution of argon in an air plasma spray (APS) plume
  - Use optical spectroscopy
  - "One shot" i.e. no scanning to different wavelength regions to get data

Approach

- Image source onto the slit of an optical spectrometer coupled to a calibrated CCD array
- Analyze slit image assuming a cylindrically symmetric source
- User Abel inversion to acquire the radial intensity distribution
- Obtain radial temperature distribution from spectroscopic line intensities



Spectroscopic Window



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Spectroscopic Data

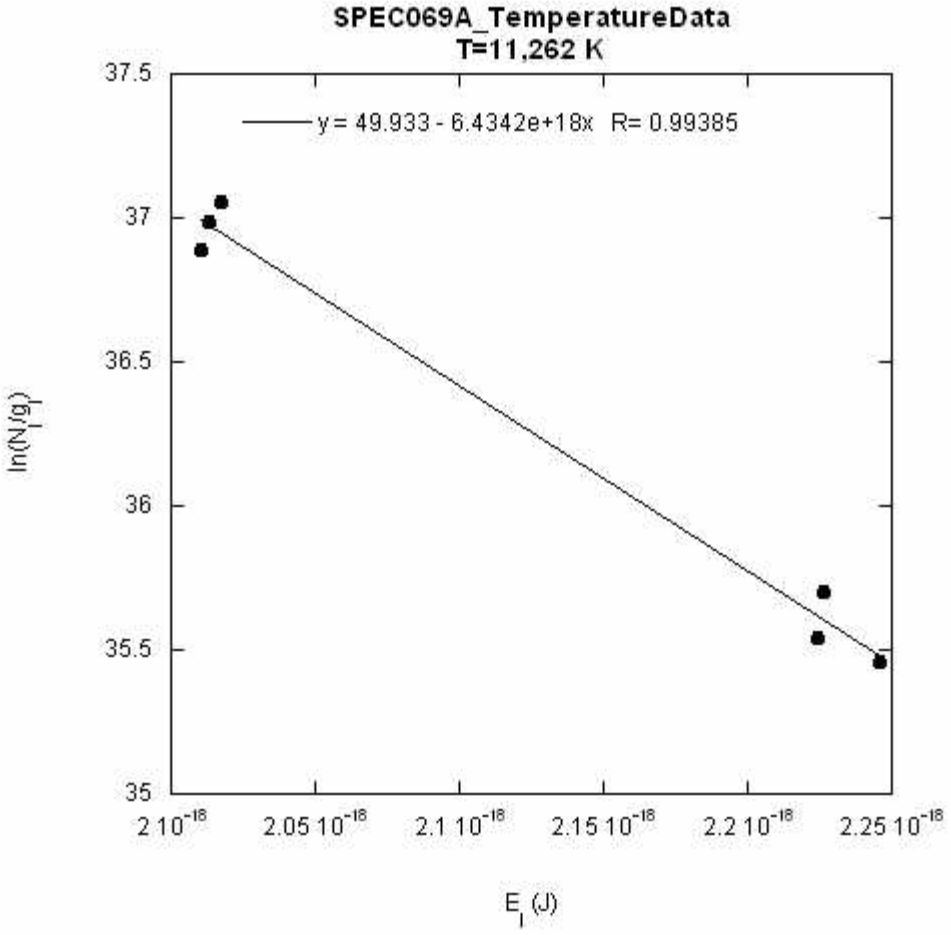
W.L. (nm)	E_{upper} (cm^{-1})	g_{upper}	A_{ij} (s^{-1})
687.13	118,651	3	2.78×10^6
693.77	118,512	1	3.08×10^6
696.54	107,496	3	6.39×10^6
703.03	119,683	5	2.67×10^6
706.72	107,290	5	3.80×10^6
714.70	107,132	3	6.25×10^6

Avg. T Calculation Using Selected Ar Lines

From Boltzmann's equation we have:

$$\ln\left(\frac{N_j}{g_j}\right) = K - \left(\frac{1}{k_B T}\right) E_j$$

where N_j is the number density in energy level j (derived from the spectroscopic line intensities) and k_B is Boltzmann's constant.



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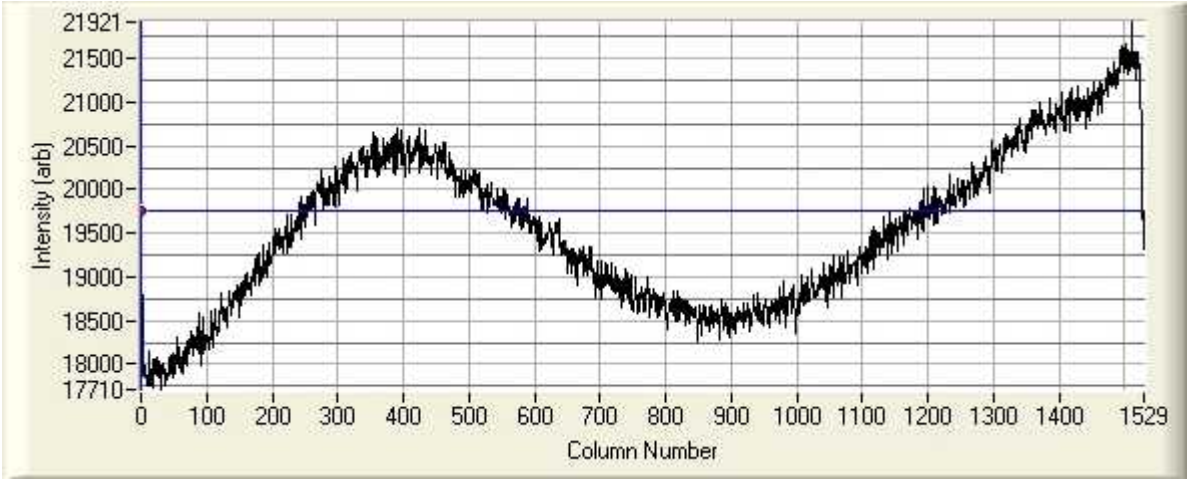
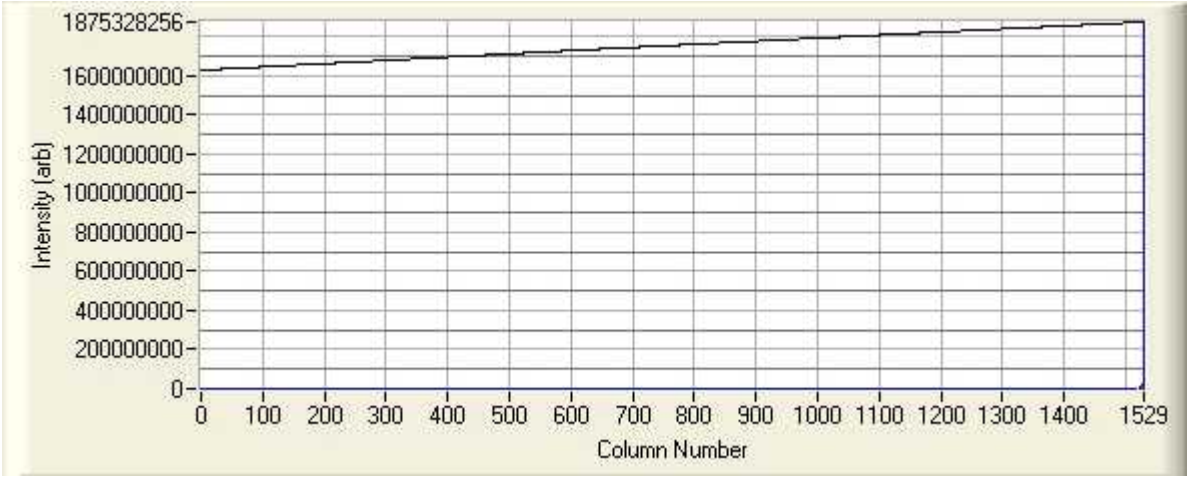
System Calibration

The filament of a tungsten ribbon lamp of known radiance is imaged onto the spectrometer slit and the pixel response of the CCD array is calibrated from the tabulated radiance curve data.

Uncalibrated



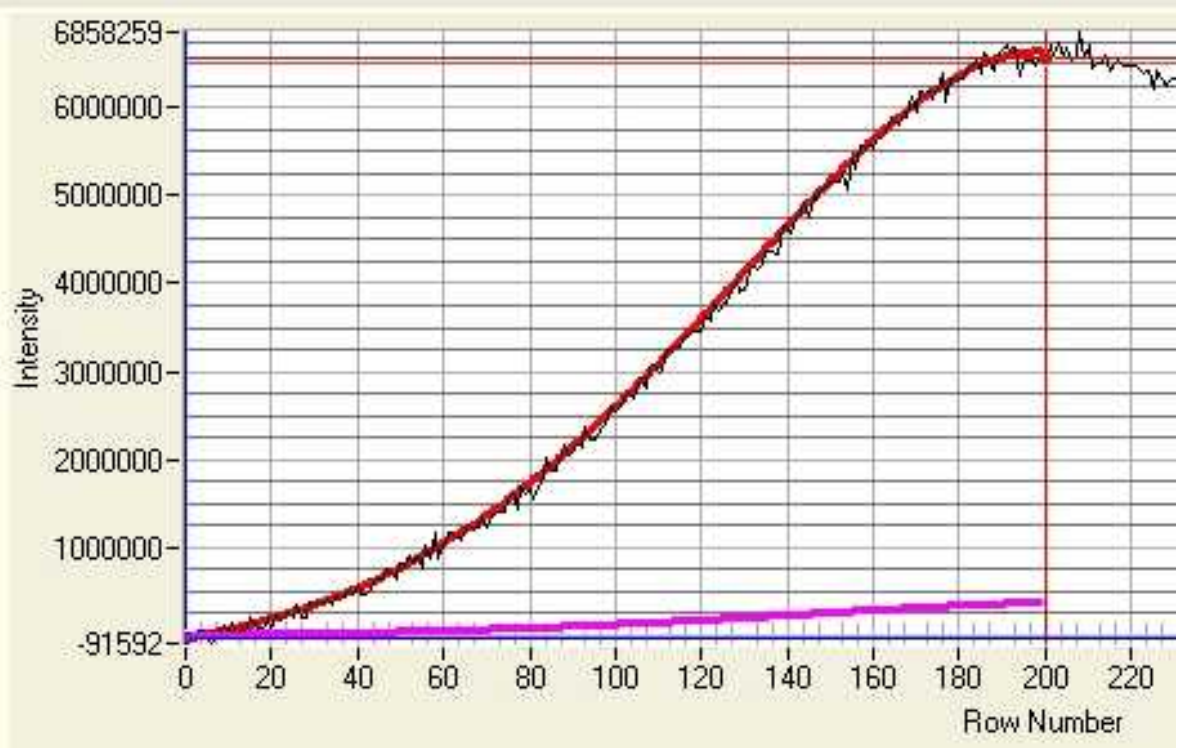
Calibrated



Abel Inversion

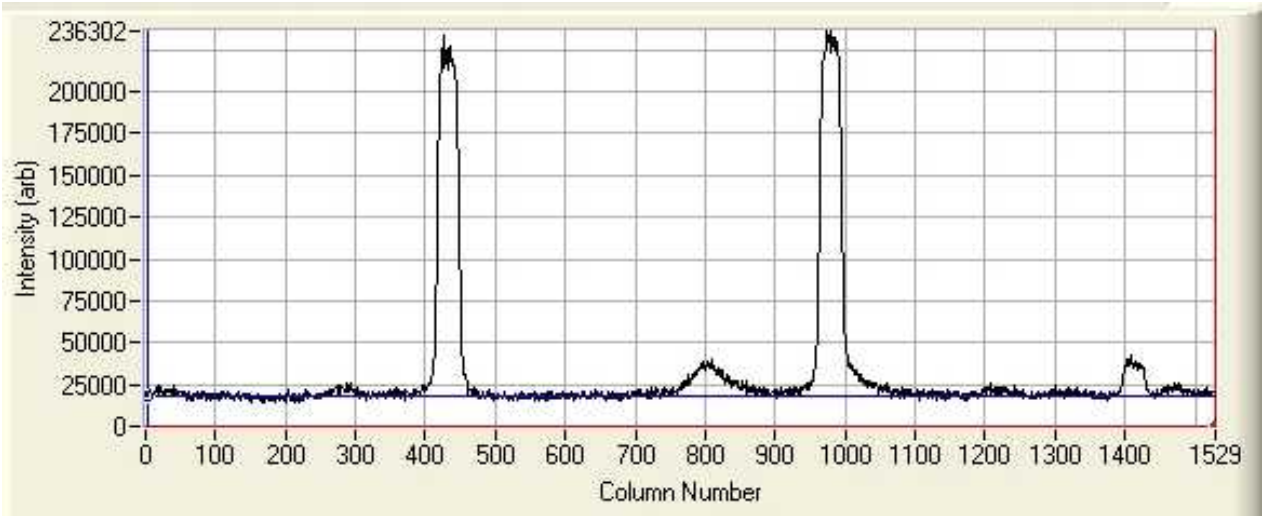
The integrated intensity data are fit with a 4th order polynomial with the 1st order coefficient constrained to be zero. The inversion is performed on this polynomial to obtain I(r).

$$I(r) = -\frac{1}{\pi}\int_{r}^{R}\frac{dI(y)}{dy}\left(y^2 - r^2\right)^{-1/2} dy$$

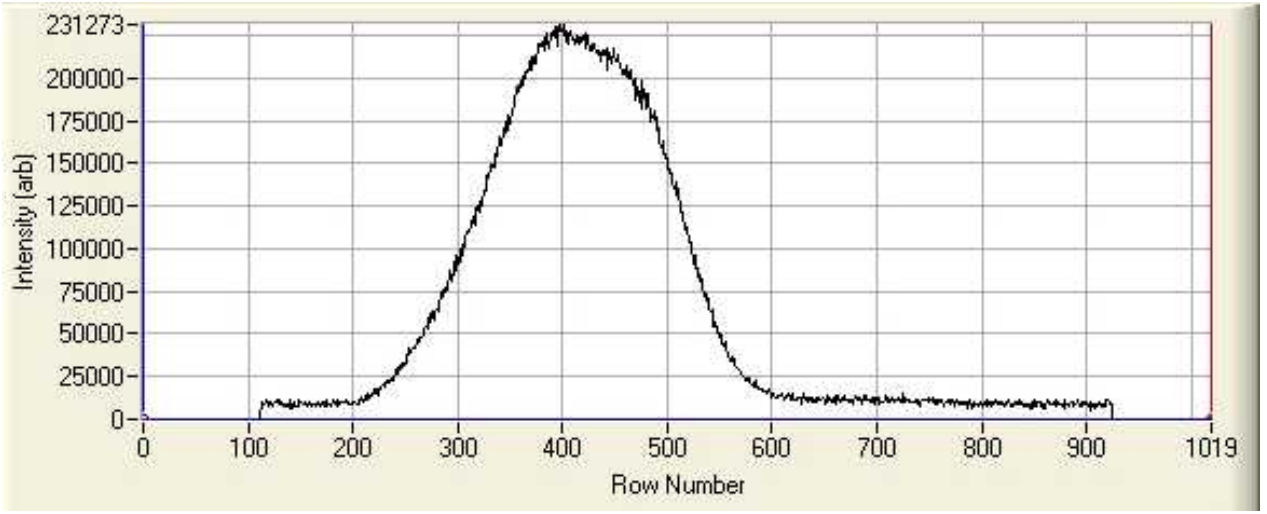


Slit Image Data

Single row of data from the CCD array containing I_{max}.



Single column of data from the CCD array containing I_{max}.



Radial Temperature Data

APS Conditions:

- 800 A
- 34.7 V
- 55 slpm Ar
- 16 slpm He

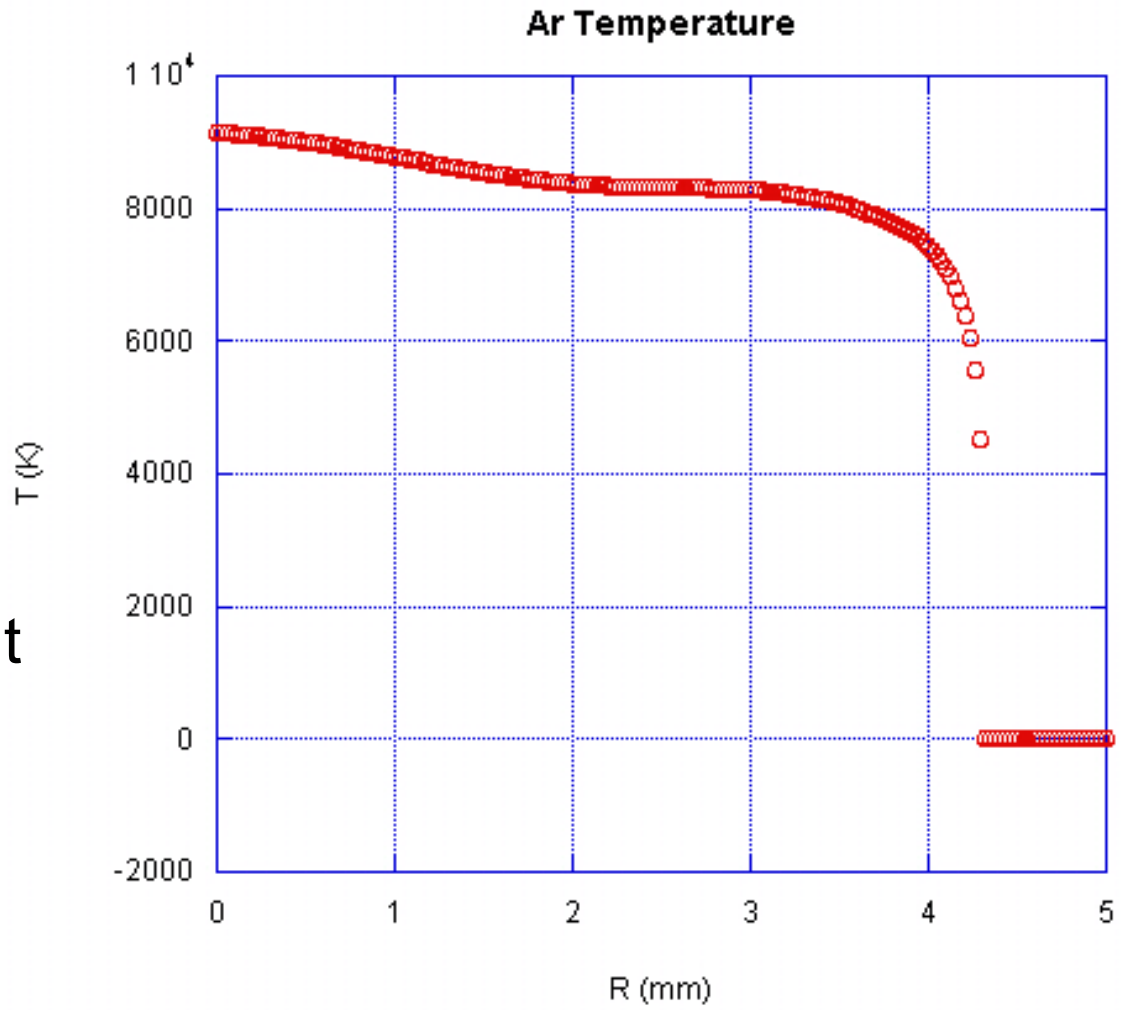
Image:

0.25 mm x 5 mm slit

1.6 s exposure

5.0 OD n.d. filter

455 nm l.p. filter



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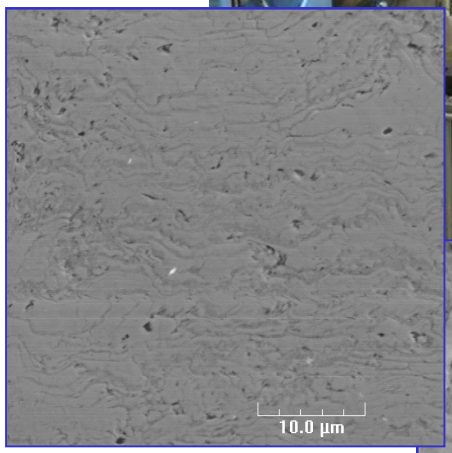
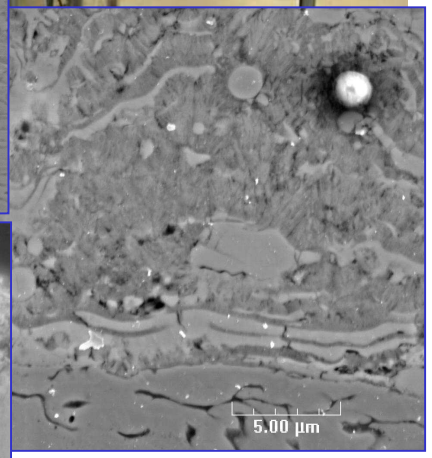
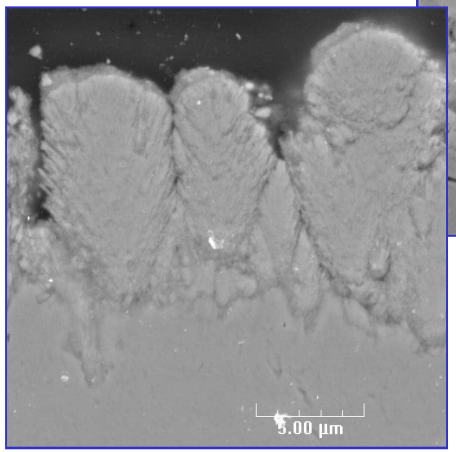
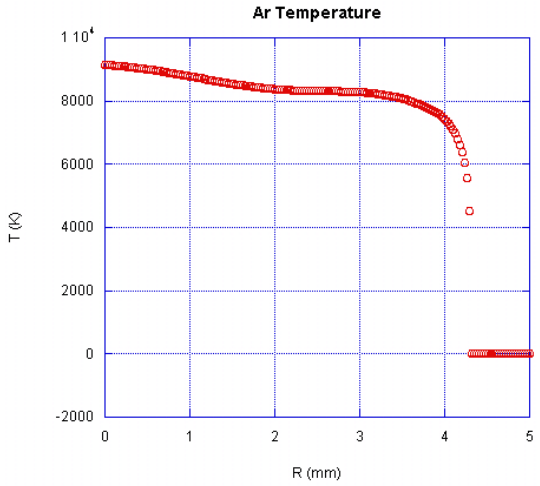
Summary

- 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.
- A spectroscopic tool has been developed that allows measurement of radial Ar temperature distributions from APS & LPPS spray plumes.









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Questions?

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