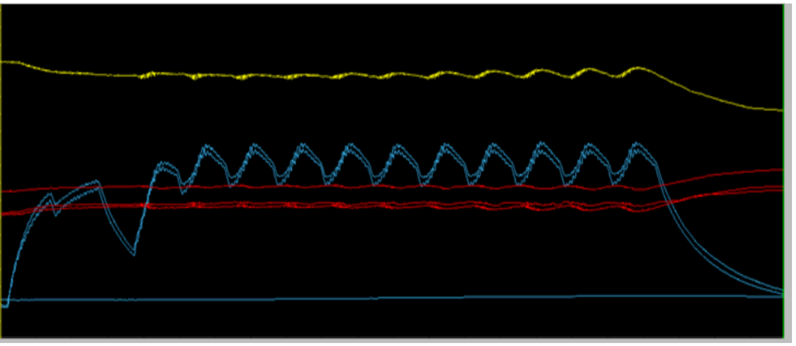
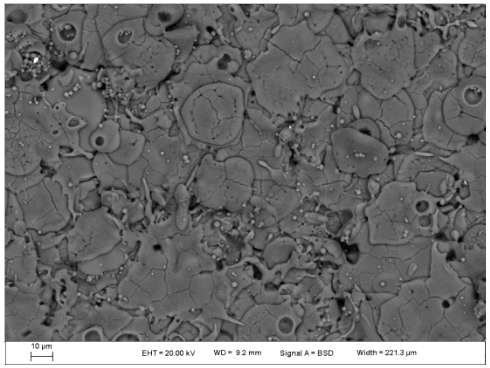
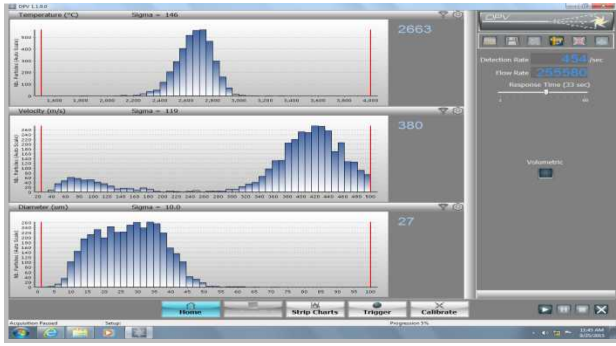
SAND2015-8446C

Exceptional service in the national interest

Sandia National Laboratories







Reproducibility in Plasma Spray Processes

<u>Andrew S. Miller</u>, Pylin Sarobol, Thomas Holmes, and Carlos Silva

Rio Grande Symposium on Advanced Materials, October 2, 2015

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. SAND NO. 2011-XXXXP

Motivations

Control of Thermal Spray process parameters presents challenges, often done in reaction to results measured after the fact.

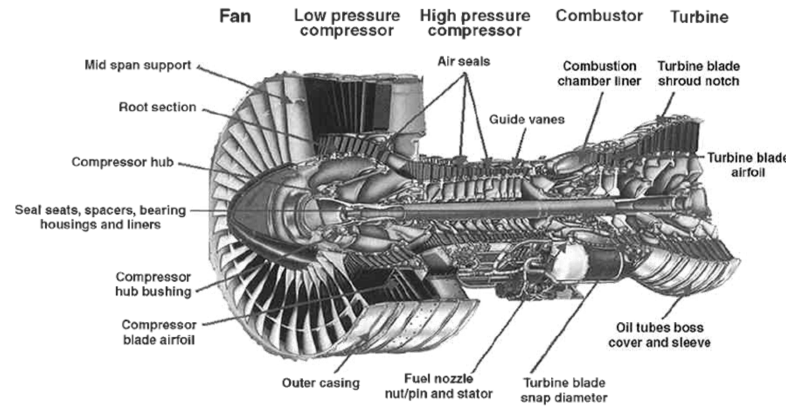
Measurement of coatings often involves off-site SEM and metallographic investigations.

Repeating a good result or adjusting for a poor one involves guess work and experienced “feel” for the process.

What are Thermal Spray Coatings Used For?

- Aerospace
  - Gas Turbines
  - Landing Gear
- Automotive
- Biomedical
- Computers
- Electronics
- Infrastructure
- Marine
- Paper Making
- Petrochemical
- Power Generation
- Printing
- Textiles

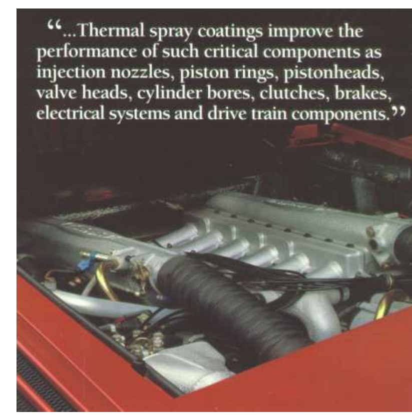
Hydroxyapatite coating on a hip implant



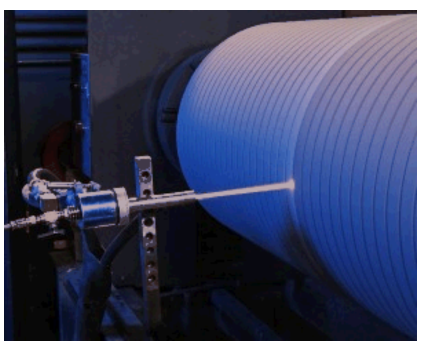
Gas Turbines are full of sprayed coatings!

Hard Chrome Replacement on a Landing Gear Strut

The Wuhan Junshan Bridge over the Yangtze River is covered with 35,000 m^2 (~8.5 acres) of thermal sprayed zinc coating!



Auto applications include pistons, valves, cylinder bores, clutches, and drive train components.



Ceramic Coating a Printing Roll

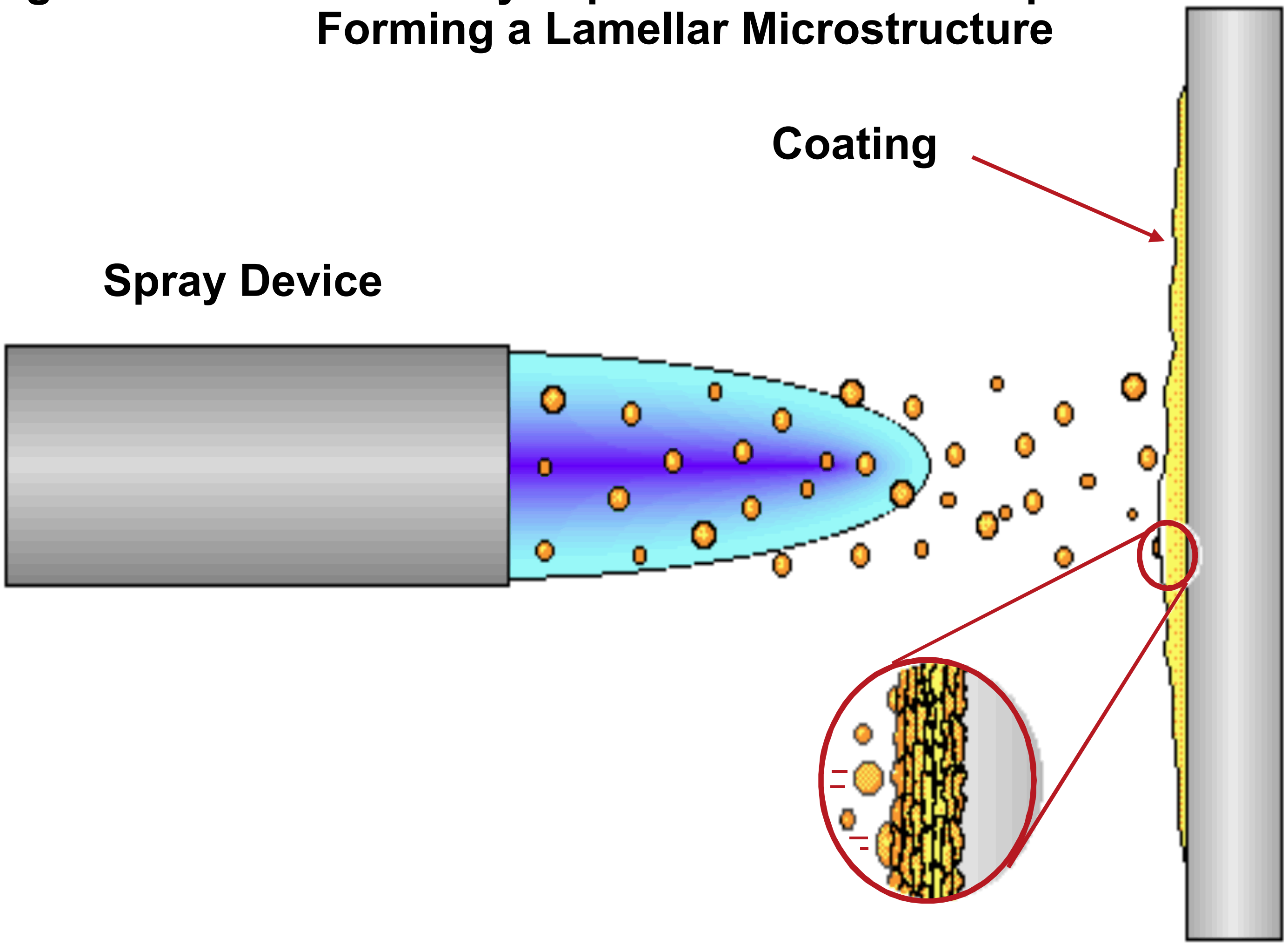
Thermal Spray Coatings find niche surface modification applications in many, many industries!

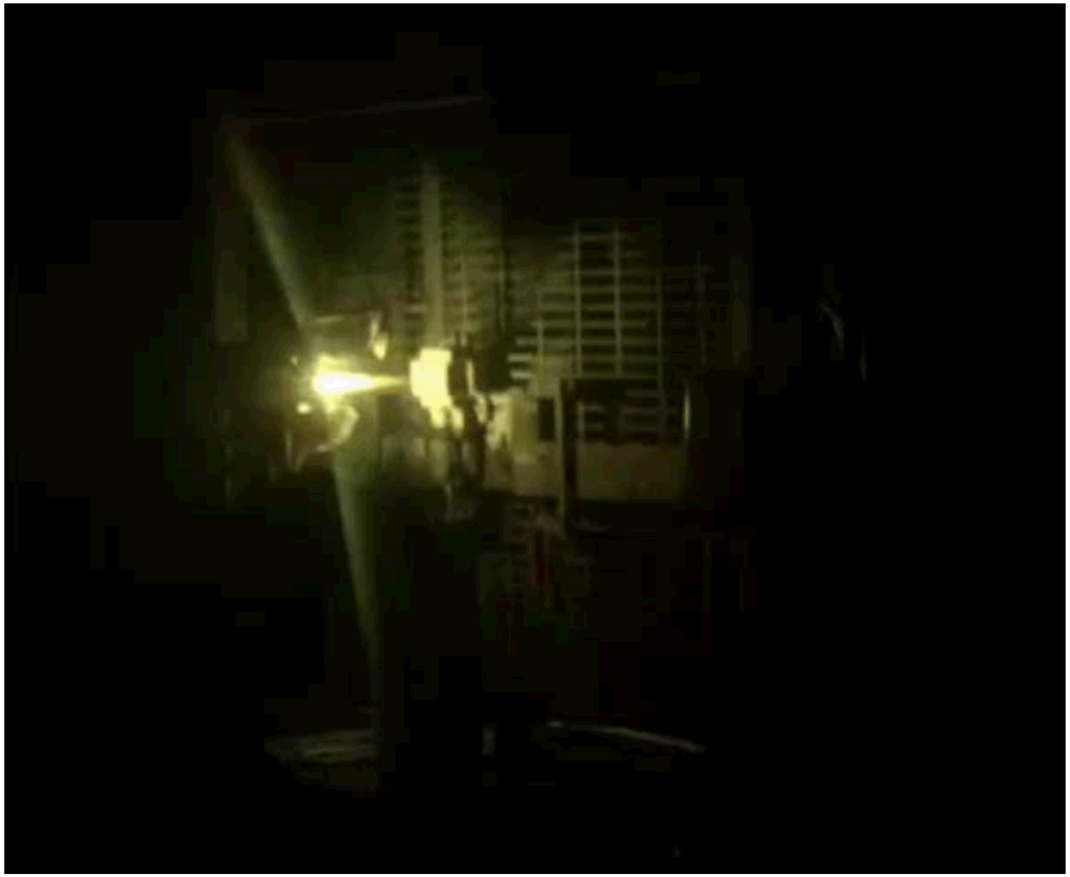
Handbook of Thermal Spray Technology, 2005, ASM International

What is Thermal Spray?

Sandia National Laboratories

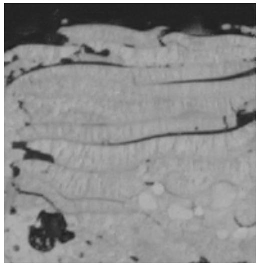
~ 10 - 100 µm Molten or Semi-Molten Droplets are Sprayed onto a Target Surface Where they "Splat" Cool at Rates up to 10^4 - 10^8 K/sec Forming a Lamellar Microstructure



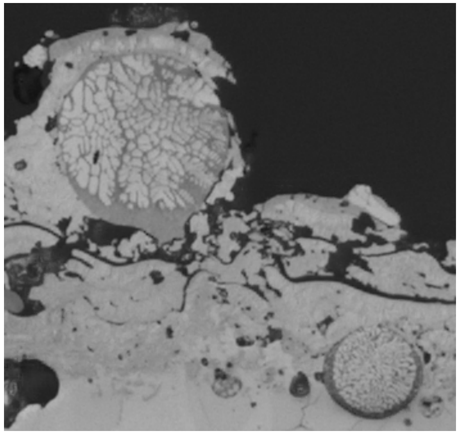


Nuts & Bolts of Thermal Spray

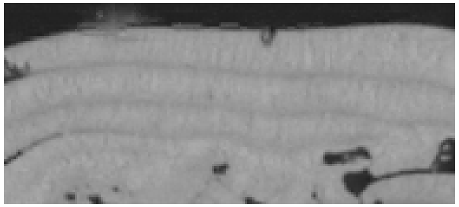
Heavier particles:
- Lower velocity
- Shorter travel distance
- Heat up slowly
- May not be melted at impact



Molten particles:
- "splash" on impact
- Make "pancake" deposits
- Splashed droplets may re-deposit into crevices and low spots
- Coatings have fewer voids



On impact, a molten particle rapidly cools and contracts while it is constrained by adherence to the substrate. This is "quenching stress" – part of "deposition stresses"

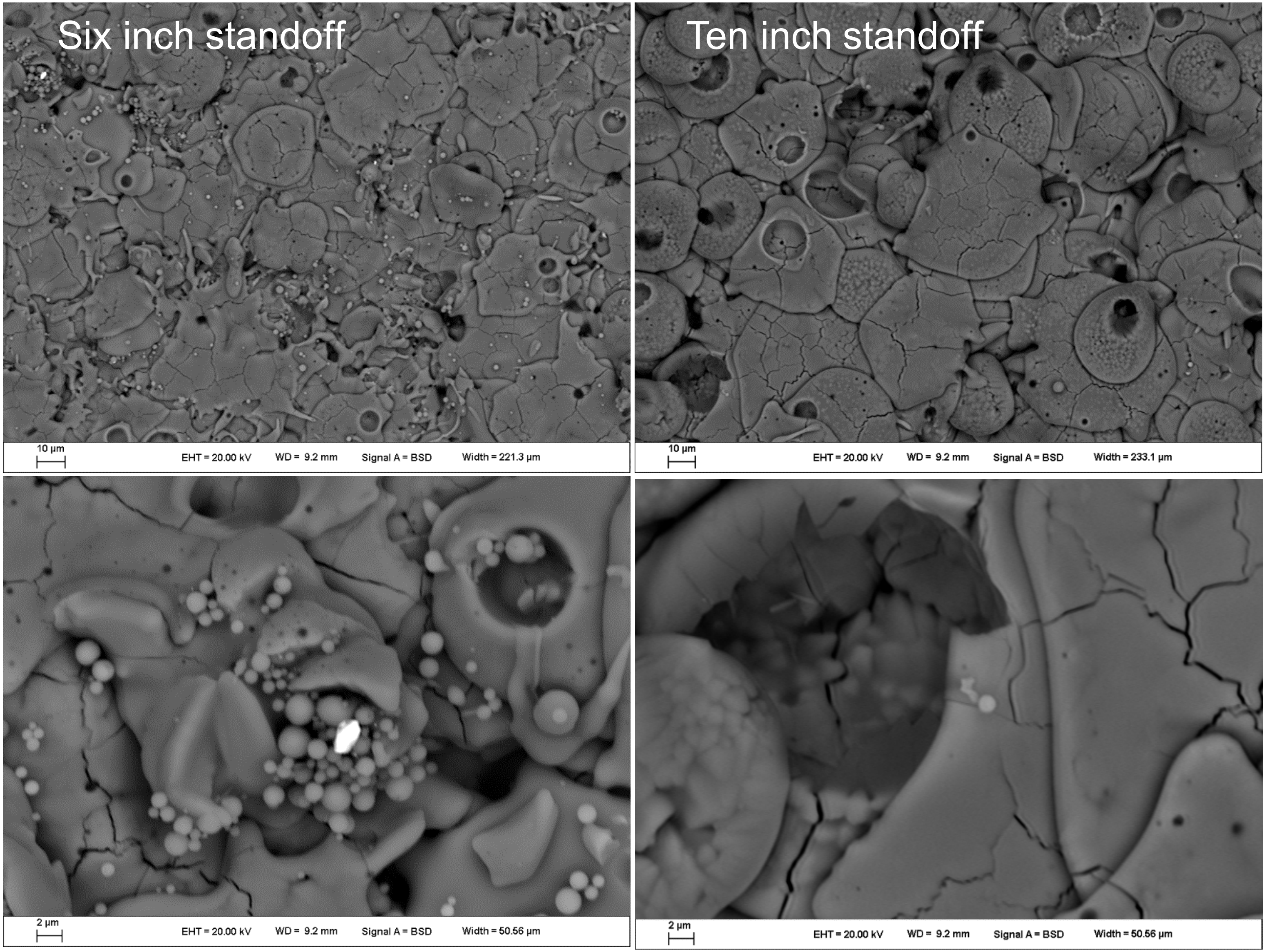


Un-melted particles:
- May fracture or crack on impact
- May not deposit
- Greater momentum at impact
- Coatings have more voids

Lighter particles:
- Higher velocity
- Greater travel distance
- Heat up quickly (melt faster)
- May vaporize

Sandia National Laboratories

zoom

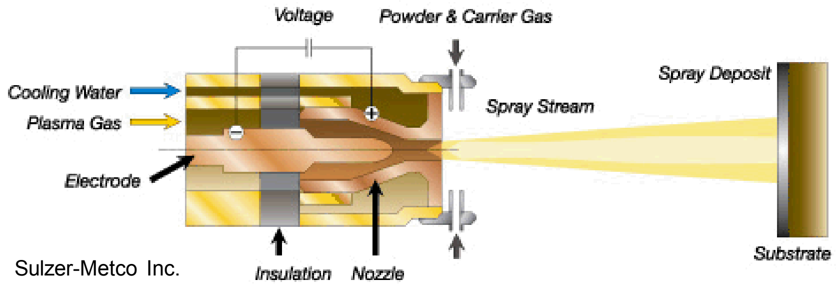


Smaller splat diameters. More splashing.
Less craters. Micro-cracks.
More fine spherical particles (not splats).

Larger splat diameters. No splashing.
More and larger craters.
Substructures, Micro-cracks

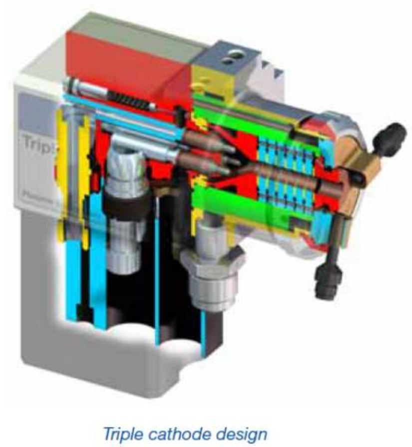
Plasma Spray Processes

Sandia National Laboratories



Sulzer-Metco Inc.

SG-100 Praxair-Tafa Inc.



Triplex®Pro-200 Sulzer-Metco Inc.

Air Plasma Spray

- DC Plasma heat source
- SG-100, Triplex®Pro-200
- I, V, & Gas Composition affect T_p & V_p

"Vacuum" Plasma Spray

- Plasma spray at ~ ½ atmosphere (380 torr)
- Oxide-free coatings

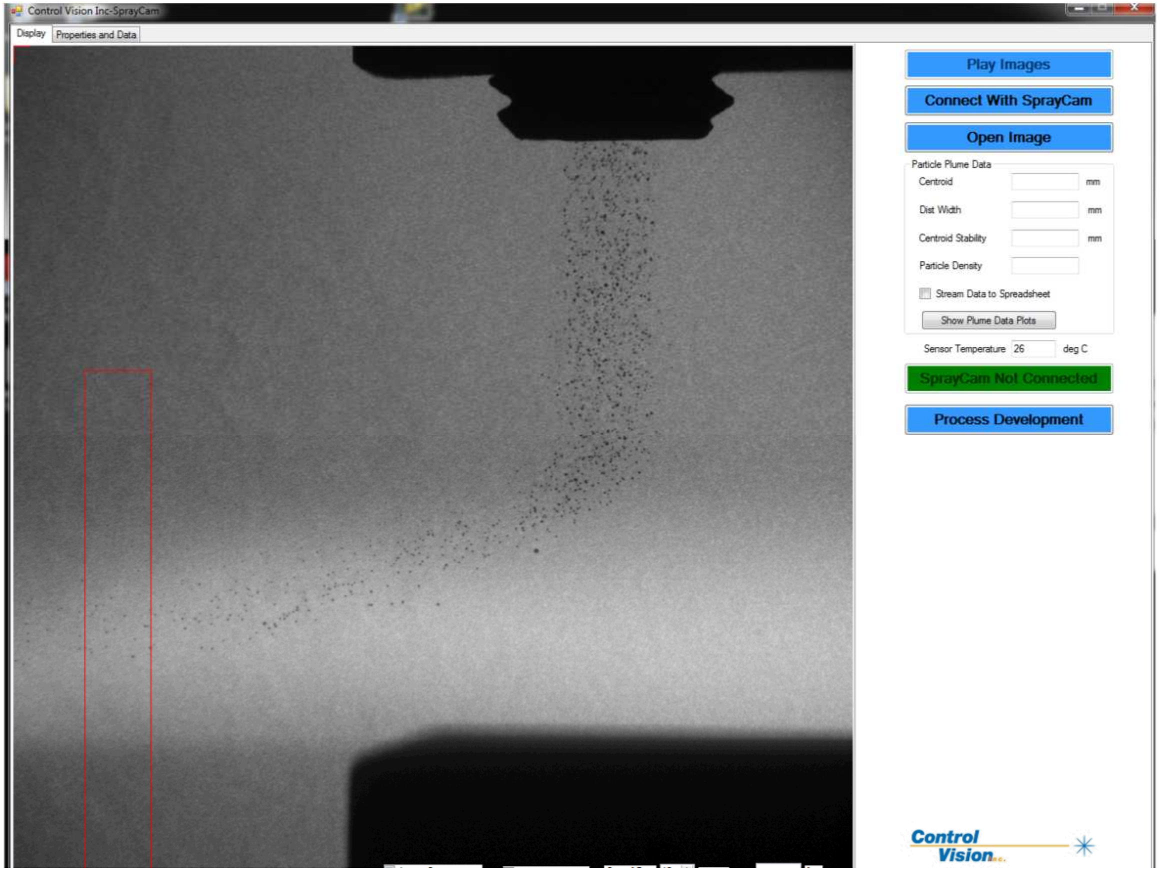
Very Low Pressure Plasma Spray

- Plasma spray at 1.0 Torr (0.001 atm)
- Emerging Technology
- SNL has one of two systems in U.S.
- *Droplet Deposition*
- *Vapor Deposition!*
- *Thin coatings (< 50 microns)*

O3CA Suzler-Metco Inc.

Measuring the parameters between torch and substrate

TUNING THE PROCESS



Control Vision

- Optimize particle insertion to the plume
- Quantify flux at a point in the plume

Particle Temperature (Tp) and Particle Velocity (Vp) directly affect coating microstructure and properties.

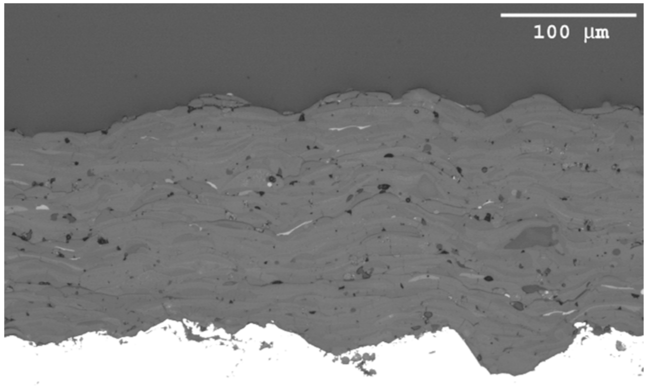
Sandia National Laboratories

Tp: Particle Thermal energy
Vp: Particle Kinetic energy

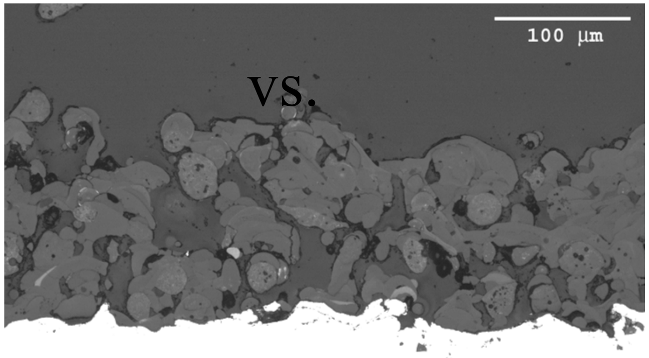
- *Are controllable*
- *Are measureable*
- *Make sense*

Increasing Tp or Vp

- *Increases deposition efficiency*
- *Reduces coating porosity*
- *May increase residual stress*
- *May increase substrate damage*

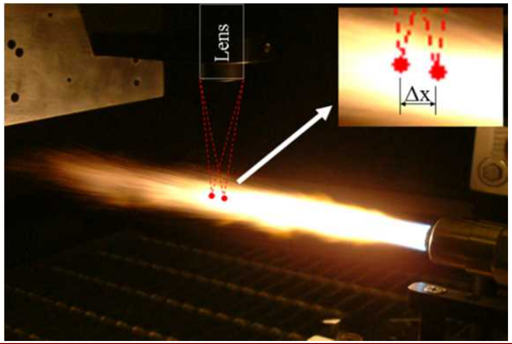


vs.



Sensor-Based Particle Characterization

- *Simultaneous time of flight and two color pyrometry measurement*



$$Vp = \Delta x / \Delta t$$

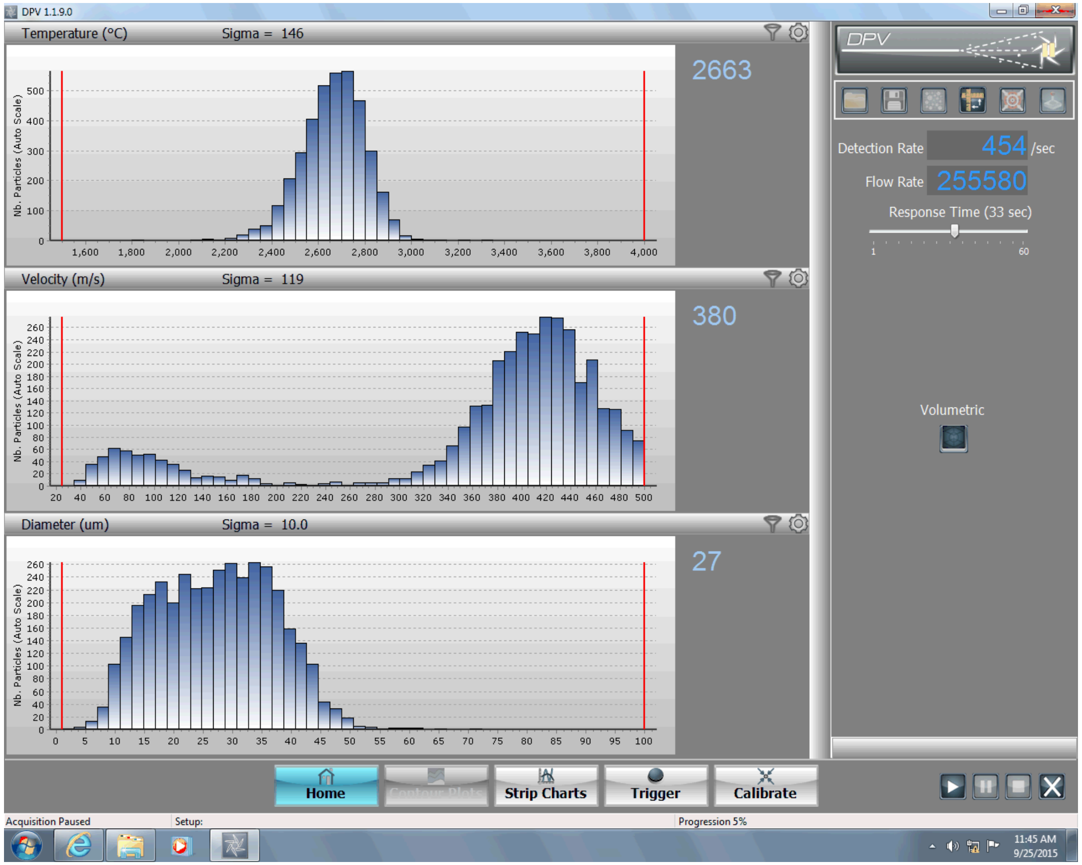
$$Tp = \lambda 1 / \lambda 2$$

DPV Histograms

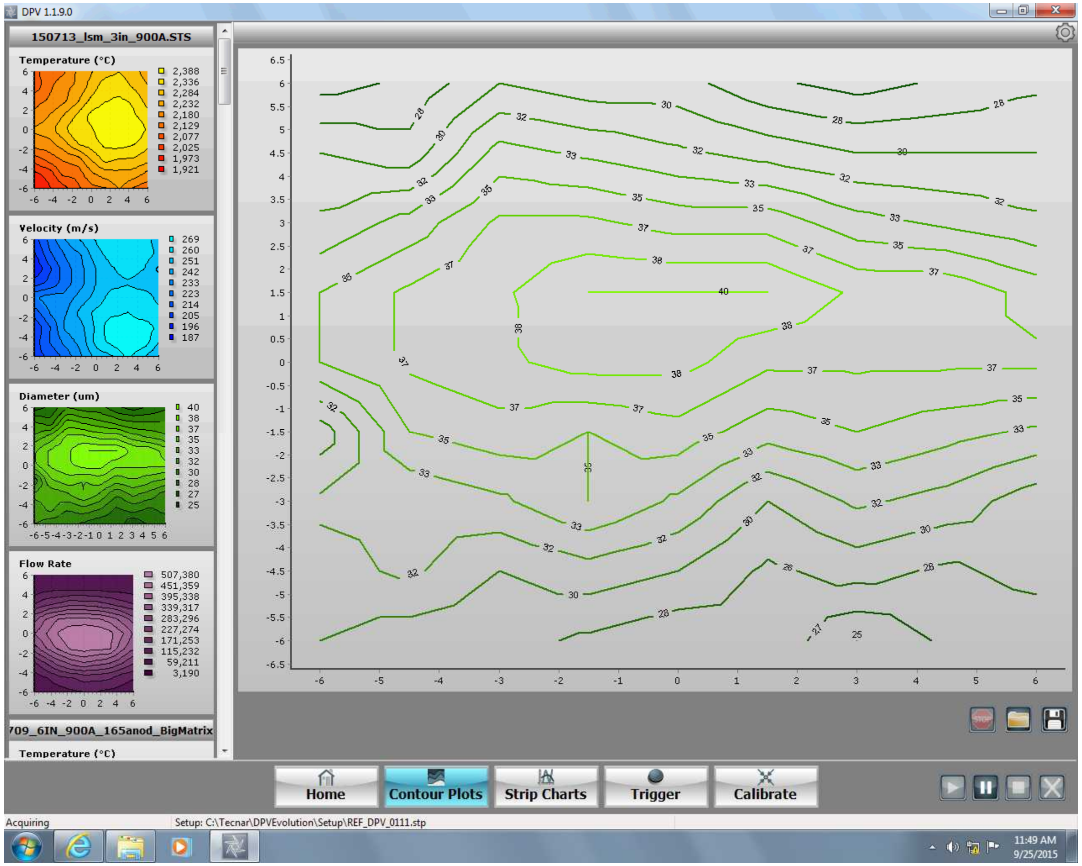
Precise measurement of velocity and temperature of up to 4000 individual particles per second

Auto-center function ensures measurement is centered on point of highest particle flux

Measurement taken at specified standoff distance provides details about particle state at impact plane



DPV Contour



2D cross-section through the plume. Temp, Velocity, Particle Diameter, Particle Flow Rate at specified standoff distance

Less precise than histogram

Process Conditions

Sandia National Laboratories

Torch Parameter	Value
Standoff	6 inch, 10 inch
Traverse Speed	200 mm/s
Amperage	450A
Argon Process Gas	40 SLPM
Helium Process Gas	20 SLPM
Powder Feed Rate	20 g/min
Powder Gas Flow	4 SLPM
Cooling Air	4 NLPM

Particle Parameter	Mean	St. Dev.
Temp	1608°C	603
Velocity	62 m/s	12
Diameter	77 µm	50.6

How can we know that one coating is the same as another?

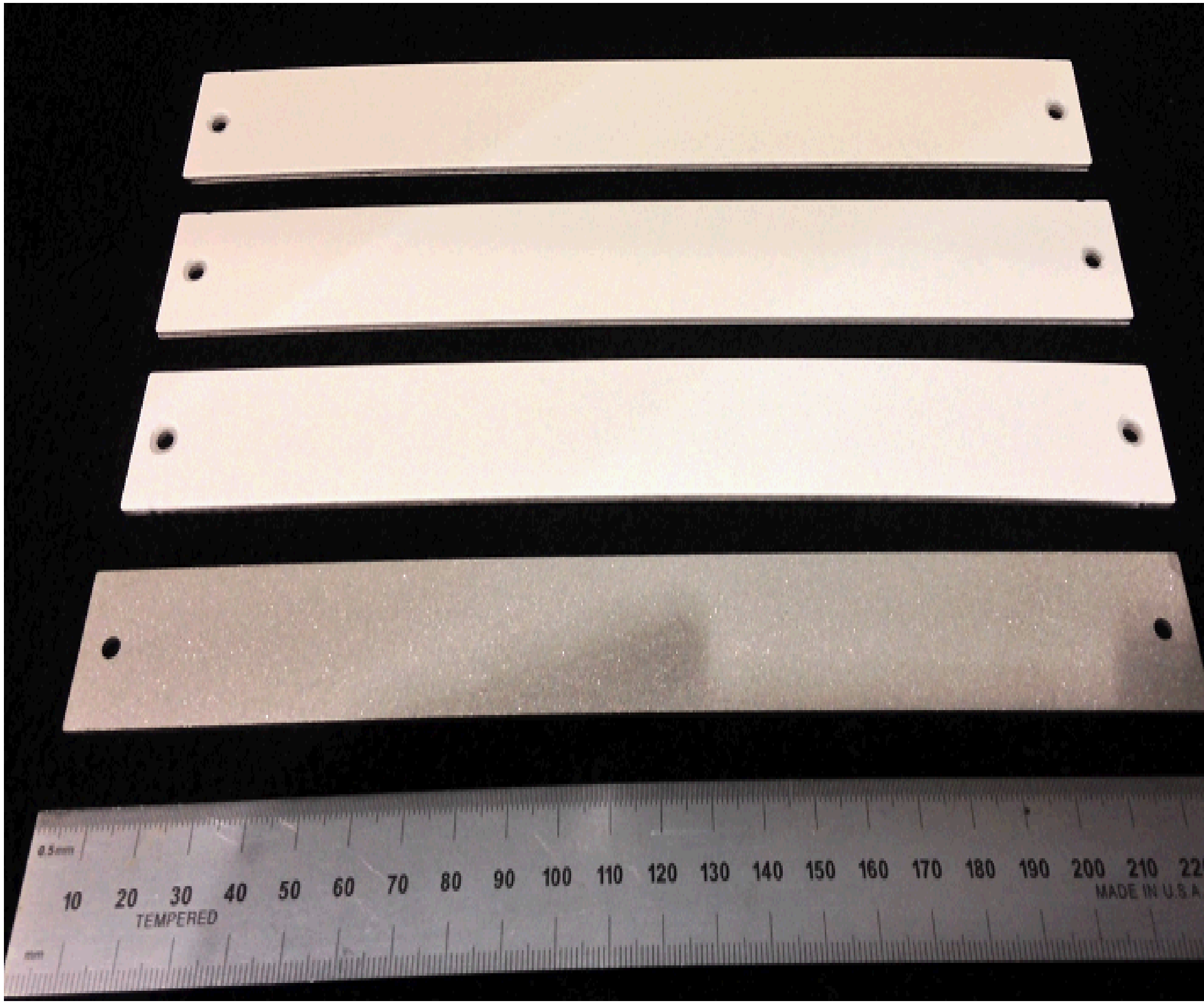
MEASURING COATINGS IN-SITU

ICP

New instrument measures curvature and temperature real time, in-situ to determine residual stress and elastic modulus of sprayed coating

In-situ coating properties (ICP) sensor can be used to determine repeatability of coating based on these parameters with much faster turn around.

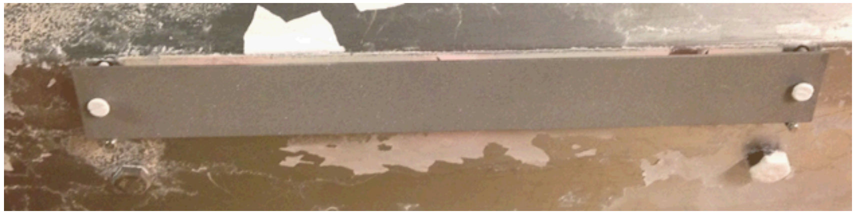
Limitations of the instrument require knowledgable user and some institutional experience to effectively quantify and produce repeatable coatings



Instrument Layout

Three displacement laser ports directly behind substrate

Two loose pin connections prevent binding as beam curves during deposition run



ICP Data & Clyne Equation

$$\Delta\kappa = \frac{6E_0E_S(h+H)hH\Delta\alpha\Delta T}{E_D^2h^4 + 4E_DE_Sh^3H + 6E_DE_Sh^2H^2 + 4E_DE_ShH^3 + E_S^2H^4}$$

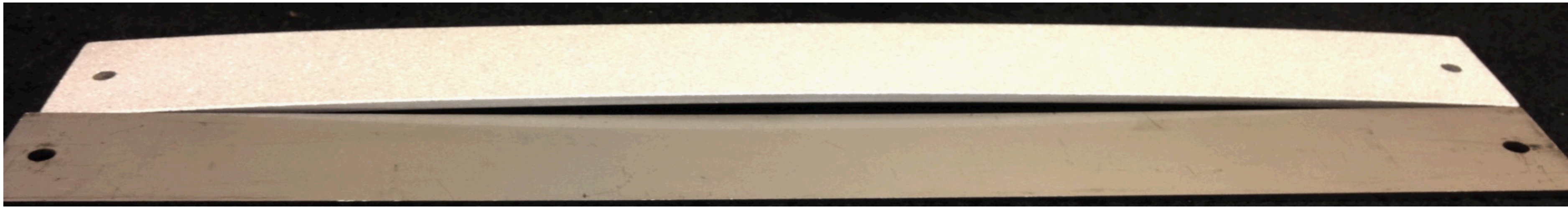
$$\Delta\kappa \propto \frac{hH\,\Delta T}{(h+H)^3}$$

Curvature and Temp are measured directly.

Bulk moduli, substrate and coating thicknesses, and CTEs are inputs.

Iterative use of equation yields calculated coating modulus; this in turn is used to determine stresses

Subjectivities of ICP

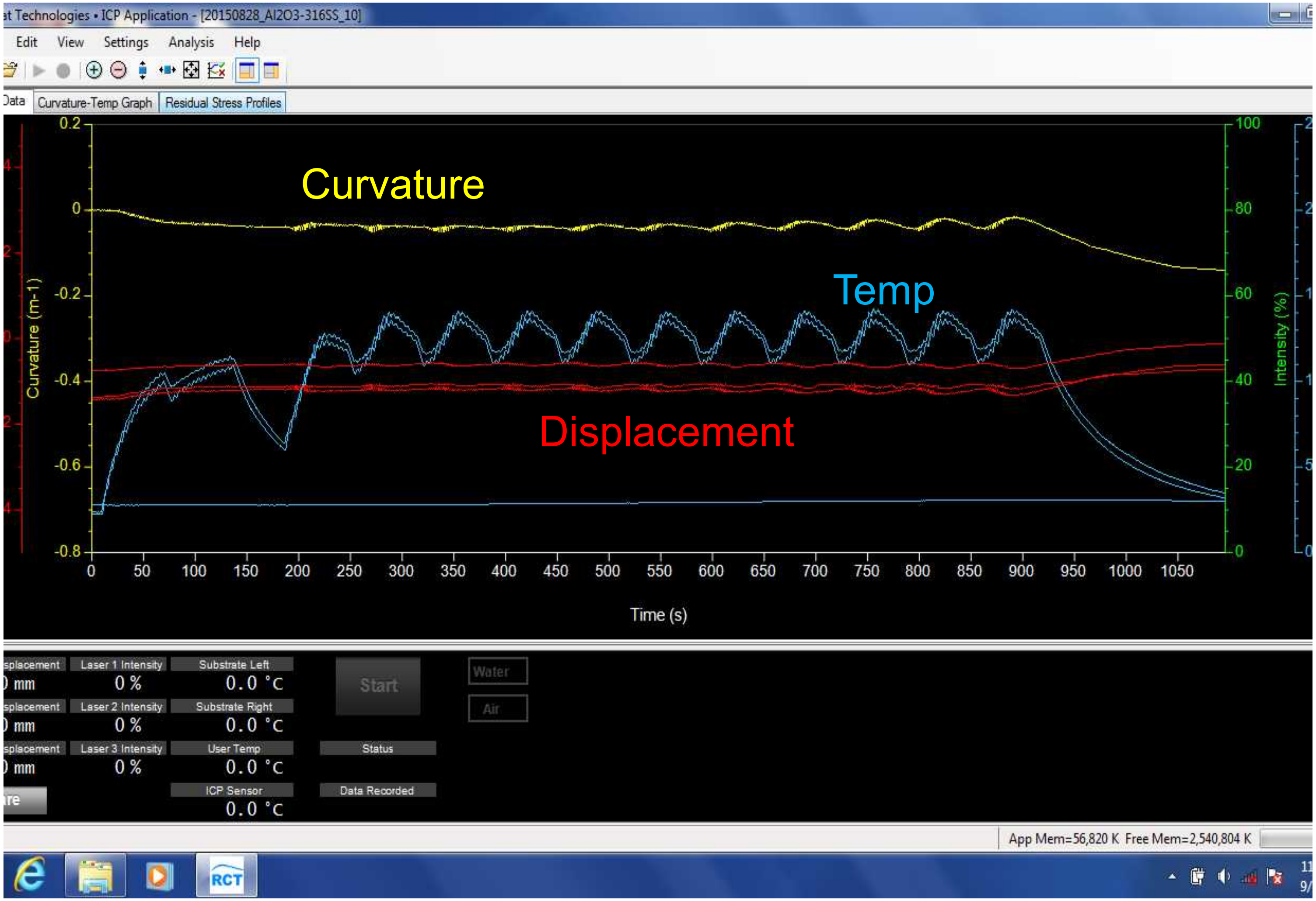


Sample Prep:

- Grit blasting removes surface oxides and introduces surface texture for mechanical adhesion
- Induces offset curvature that will offset the curvature caused by the coating stresses

Solution:

- Automate grit blasting process (costly)
- Apply to both sides of beam in attempt to “balance” the induced curvature
- Use one operator to perform all grit blasting for a given project



Temp (blue) note pre-heat, steady state through several raster passes

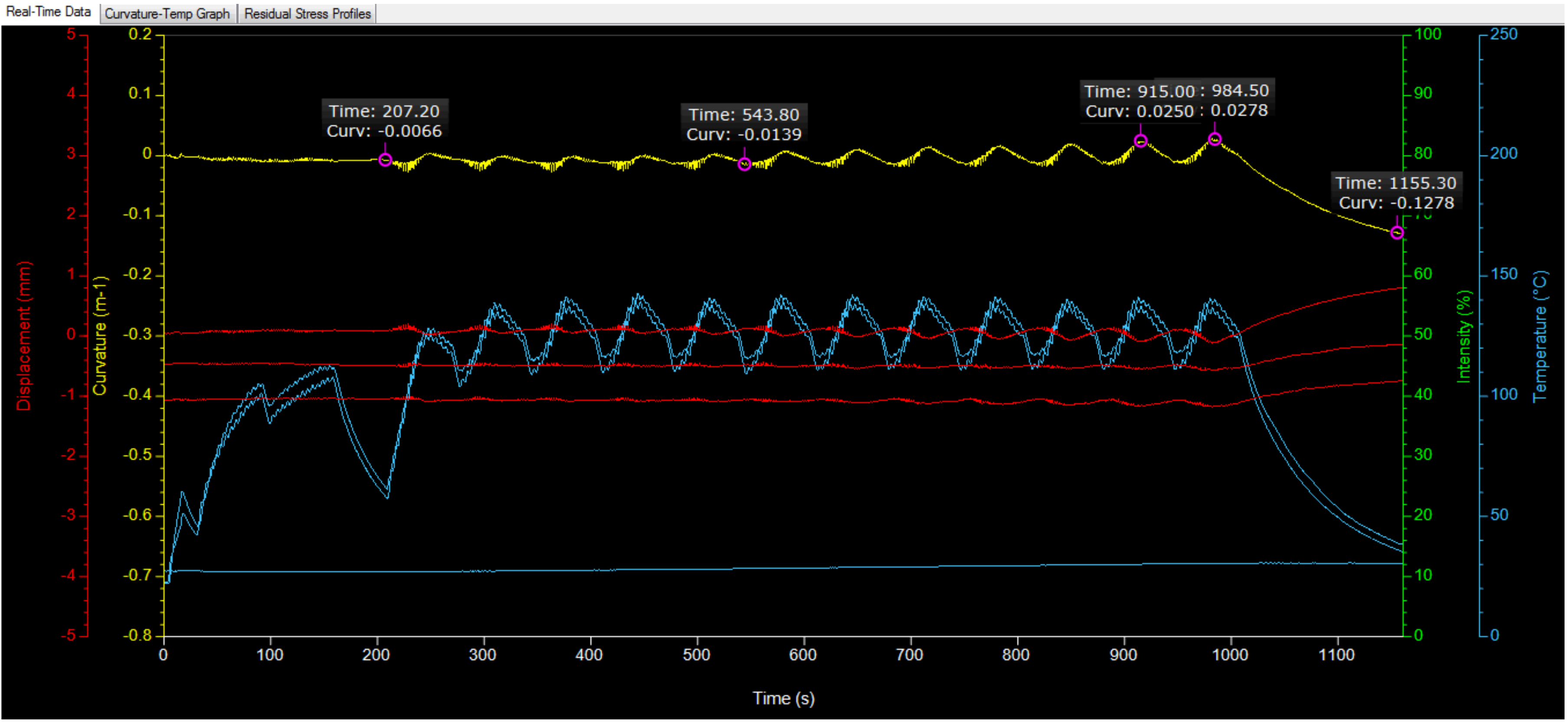
Three lasers (red) two at ends of lesser magnitude than center shows convex bending

Curvature (yellow) shows increasing flex with thermal cycling as spray run progresses, then slowly increases as thermal stress builds on cooling

Real-Time Data Collection Screen

Deposition Stress + Thermal Stress = Residual Stress

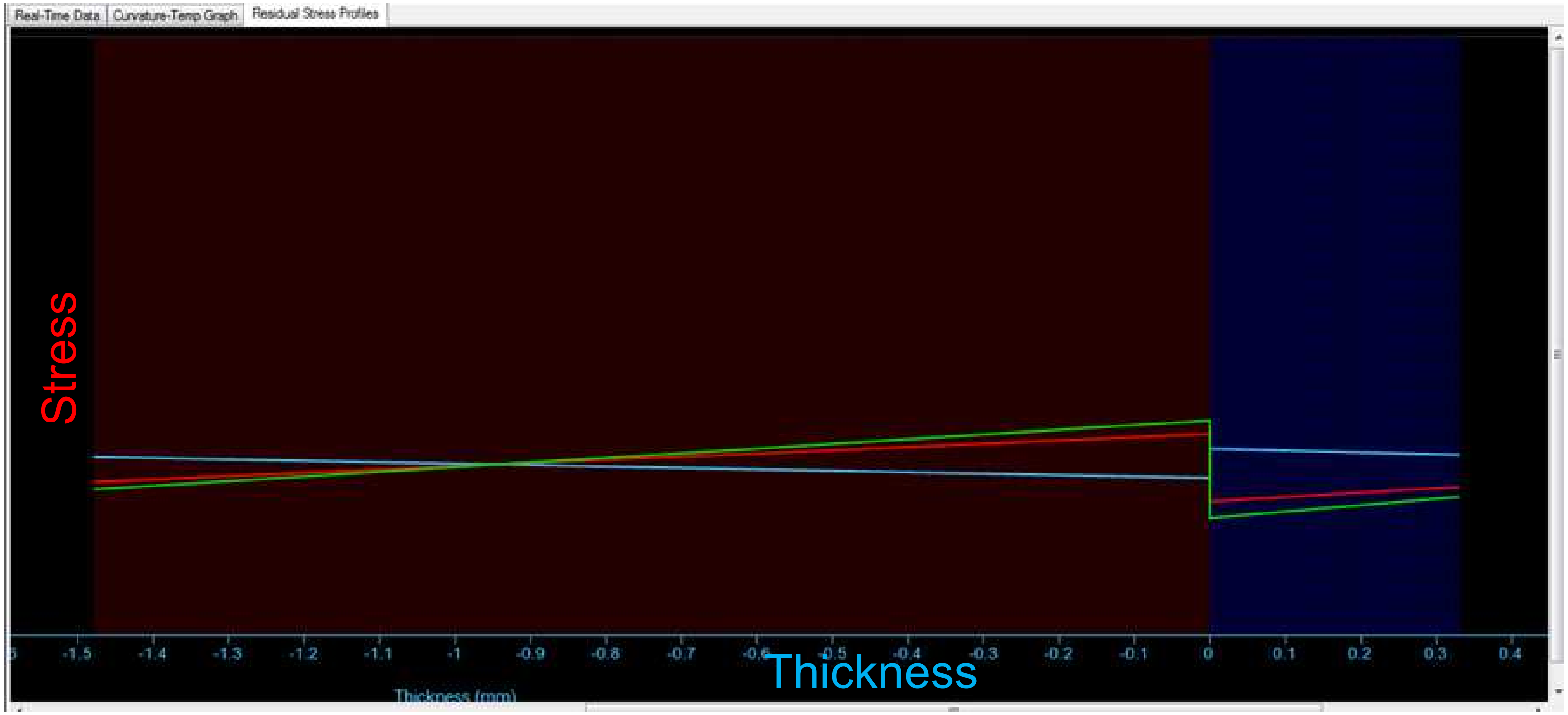
Subjectivities of ICP



Selection of points performed by operator:

- Beginning and end of spraying
- Beginning and end of deposition stress regime
- End of cooling (approx. room temp)

Stress Profile Plot

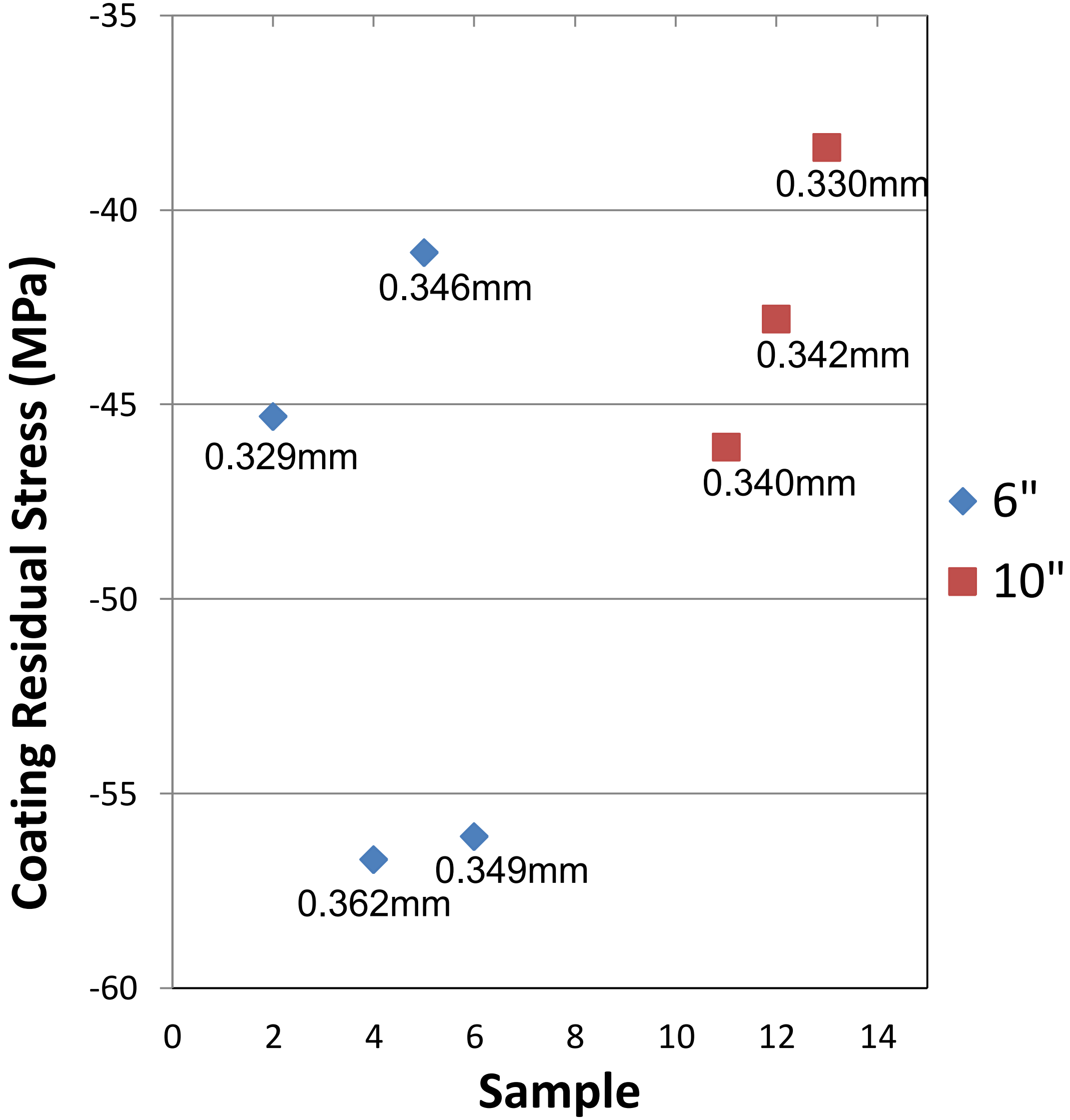


Stress profile shows build of stress through thickness of substrate and coating

Note differing slope of deposition stress (blue) and thermal stress (red)

What does all of this tell us?

LOOKING AT THE PRODUCT



Residual Stress = Quenching Stress + Thermal Stress

Large variability due to process variation

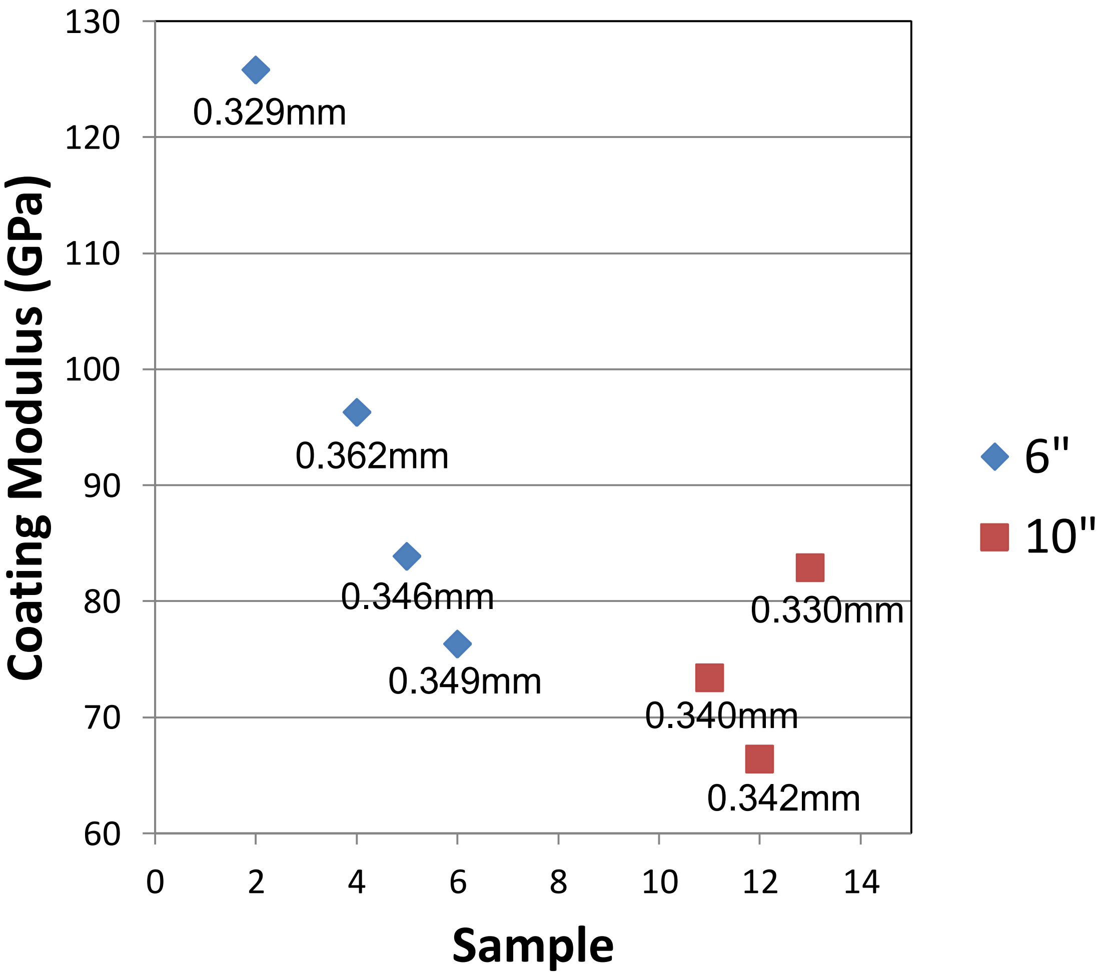
Reproducibility of process measured by variability of residual stress

Elastic modulus determined for linear range selected from Curvature – Temp plot.

Compare to bulk alumina modulus ~ 300 GPa. [accuratus.com]

Large variability due to process variation

Reproducibility of process indicated by variation of coating modulus



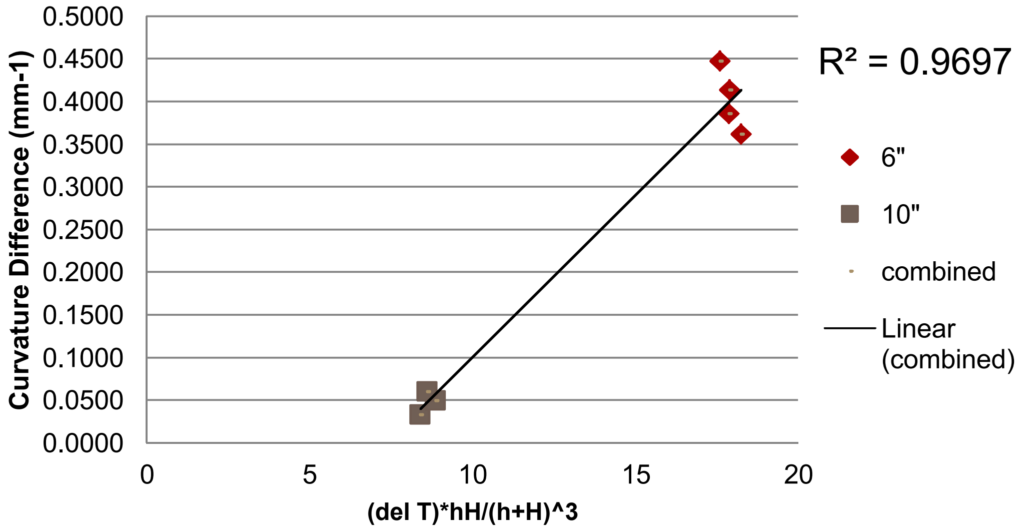
Conclusions

TSRL currently has capacity to capture relevant process parameters and variations.

TSRL now has instrumentation to measure coating product properties in-situ, at the point of production

For this particular experiment, 6” standoff yielded particles with higher velocity and temperatures, resulting in coatings with higher magnitude of compressive residual stress and higher modulus.

Future work will focus on tuning these input and output parameters to determine repeatability of sprayed coatings for increased efficiency and higher quality products



$$\Delta\kappa \propto \frac{hH\,\Delta T}{(h+H)^3}$$

Questions

?

Sandia National Laboratories

Clyne Equation for Thin Films

$$\Delta\varsigma = \frac{6E_D E_S(h+H)hH\Delta\alpha\Delta T}{E_D^2 h^4 + 4E_D E_S h^3 H + 6E_D E_S h^2 H^2 + 4E_D E_S hH^3 + E_S^2 H^4}$$

$$\Delta\varsigma \propto \frac{hH\ \Delta T}{(h+H)^3}$$

Linearity demonstrates validity of data

- Variation in modulus and residual stresses are indicative of variations in process
- Use of the linear relationship allows detection of whether data is real or just instrument error

Clyne, 1997

ICP Data & Clyne Equation

$$\Delta\kappa = \frac{6E_0 E_S (h + H) hH \Delta\alpha \Delta T}{E_D^2 h^4 + 4E_D E_S h^3 H + 6E_D E_S h^2 H^2 + 4E_D E_S h H^3 + E_S^2 H^4}$$

$$\Delta\kappa \propto \frac{hH\ \Delta T}{(h + H)^3}$$

Input

- $E_{substrate}$
- $E_{coating\ (bulk)}$
- Substrate Thickness (H)
- Coating Thickness (h)
- Coating Weight
- Feedstock Flow Rate
- Traverse Speed

Output

- $E_{substrate}$ (calculated)
- $E_{coating}$ (calculated)
- Curvature (K)
- ΔT
- Deposition Efficiency