Microstructure, Processing, Performance Relationships for High Temperature Coatings

Thomas M. Lillo

Materials Science & Engineering
Idaho National Laboratory

Co-Investigators:
Dennis C. Kunerth
W. David Swank
D.C. Haggard

25th Annual Conference on Fossil Energy Materials
April 26-28, 2011, Portland, Oregon

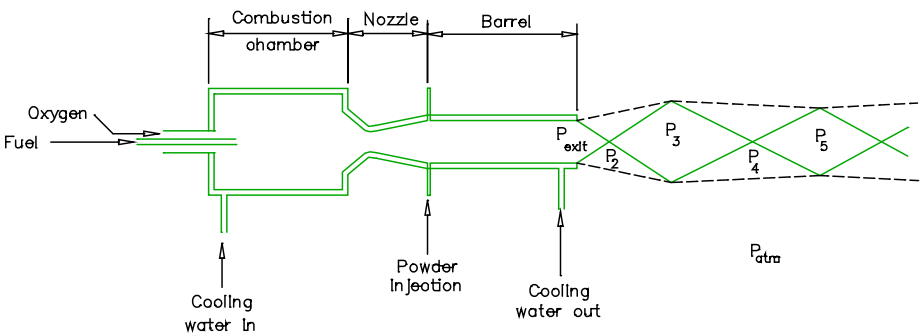
Prepared for the U.S. Department of Energy, Office of Fossil Energy, under DOE Idaho Operations Office, Contract DE-AC07-05ID14517

Goals

- Develop Fe_3Al coatings for high temperature service in fossil fuel environments
  - Develop High Velocity Oxy-Fuel (HVOF) thermal spray techniques for applying the coating
  - Understand factors and thermal spray parameters that affect the reliability of this coating
- Transfer the techniques and Fe_3Al coatings to industry
  - Demonstrate reproducibility and reliability of coatings
  - Field testing – industrial partner
  - Demonstrate repair methodologies

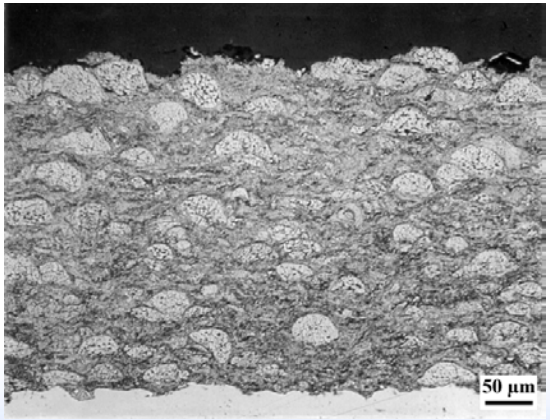
Past Results

- Thermal spray parameters can be used to generate highly dense coating with varying levels of residual stress
- Residual stresses in coating arise from three sources
  - CTE mismatch between coating and substrate
  - Quench stresses
  - “Peening” stress
- Corrosion resistance of coating is very close to wrought material
- **Coating failure governed by cracking and delamination**



High-Velocity Oxy-Fuel (HVOF) thermal spray

- Equivalence ratio (phi)- $\Phi = \dfrac{Fuel/Oxygen}{(Fuel/Oxygen)_{Stoich}}$
- Combustion chamber pressure
  P_C – Determined by total mass flow of O_2 and fuel



Fe_3Al Coating

Current Project Focus

Goal:

Determine factors affecting the mechanical stability of HVOF thermal spray coatings

Tasks:

- Characterize the influence of thermal spray parameters on the mechanical stability of coatings - Completed
- Determine the influence of substrate properties on coating durability during thermal cycling – Completed
- Prepare manuscript for peer-reviewed journal – Completed (submitted to J. Thermal Spray Tech.)
- Determine the influence of thermal spray parameters and substrate properties on coating adhesion – Completed
- Determine whether coating corrosion resistance is influenced by thermal spray parameters – simulated fossil fuel combustion environments, decarburizing, carburizing and oxidizing environments – In progress

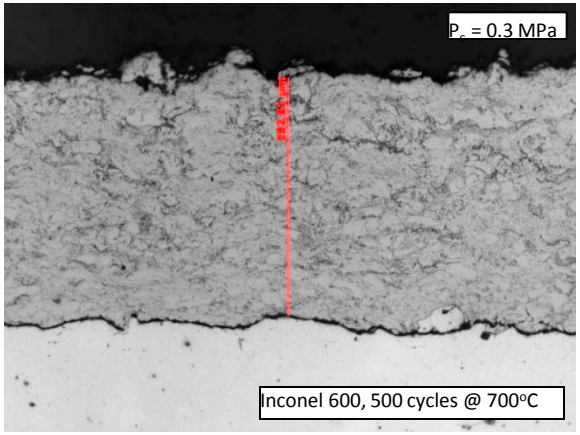
Coating Durability – Thermal Cycling

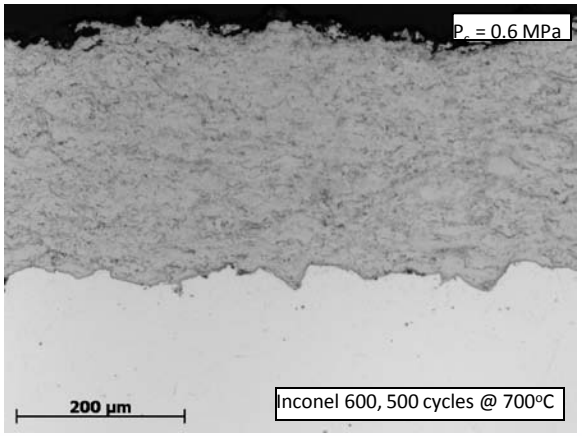
- **Parameters investigated:**
  - **Substrate material: Carbon steel, 9Cr-1Mo steel, 316 SS, Inconel 600**
  - **Thermal Spray Chamber pressure: 0.3 or 0.6 Mpa**
  - **Coating Thickness: 200-400 microns thick**
  - **Substrate Thickness: 12.7 mm & 19.1 mm**
  - **Substrate temperature during deposition – 190 to 540°C**
  - **Thermal cycling temperature: 550 to 900°C**

Coating Durability – Thermal Cycling – results

Thermal cycling produced these general results:

- Coatings survived lower thermal cycling temperatures
- A higher thermal spray chamber pressure (higher particle velocity) produced a more durable coating
- Coating failure was predominantly by delamination
- A lower substrate temperature during coating deposition produced more durable coatings
- Coatings on thicker substrates performed slightly better
- Inconel 600 and 316 SS outperformed 9Cr-1Mo and carbon steels

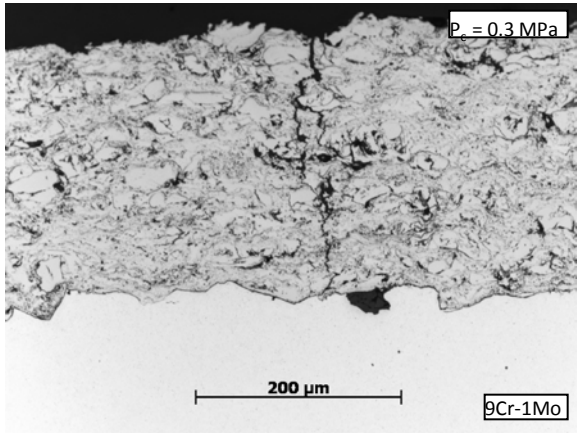




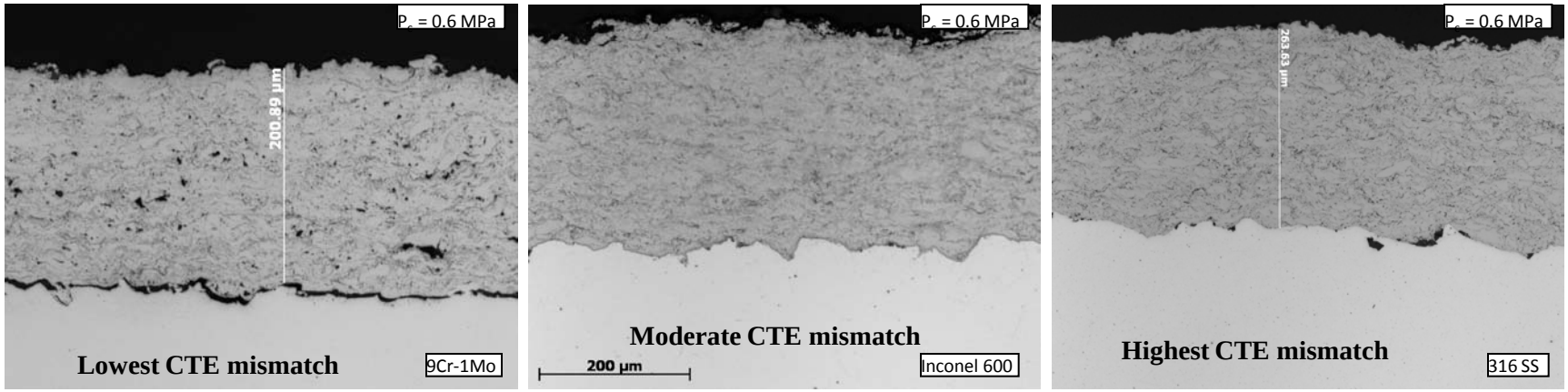
Coating Durability – Thermal Cycling – Discussion

- **CTE mismatch affects stress state**
  - CTE of coating is less than substrate materials
  - Expect reduced performance at higher cycling temperatures
- **Higher chamber pressure**
  - Results in larger compressive stresses – inhibits cracking
  - Lower coating porosity – stronger coating

Material	Coefficient of Thermal Expansion, ppm/°C
Carbon Steel (1018)	14
316 Stainless Steel	18
9Cr-1Mo (Grade 91)	13
Inconel 600	15
Fe_3Al - wrought	20.5
Fe_3Al - Coating, P_c=0.3 MPa	13
Fe_3Al - Coating, P_c=0.6 MPa	12



Coating Durability – Thermal Cycling – Discussion



After 500 cycles at 700°C

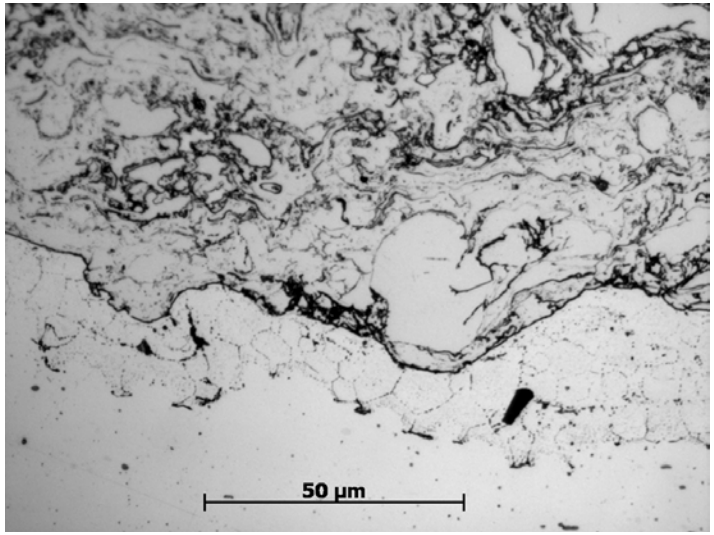
- **The poor durability shown by coatings on 9Cr-1Mo steel cannot be explained by CTE mismatch**
  - CTE for 9Cr-1Mo steel is closest to Fe_3Al coatings
  - Performed worse than Inconel 600 which has a larger CTE

Coating Durability – Thermal Cycling – Surface Roughness

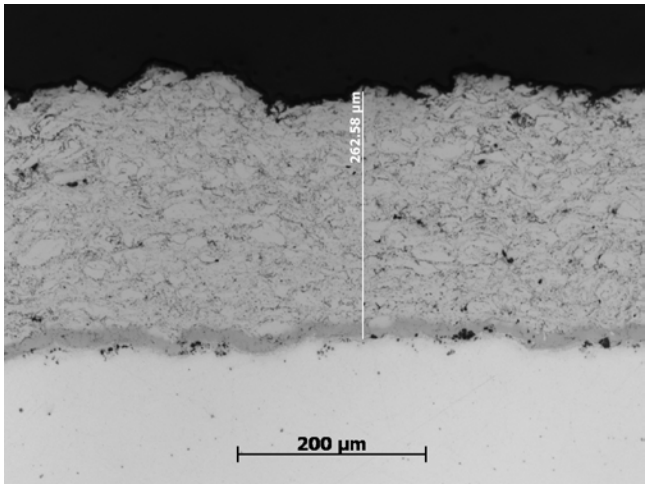
- **Major factor in coating adhesion is substrate roughness**
- **All materials prepared by automated grit blasting**
  - 24 grit Alundum (Norton St. Gobain)
  - 480 kPa
  - 25 mm standoff distance
  - Traverse speed 4 mm/second
- **Resulting surface roughness:**

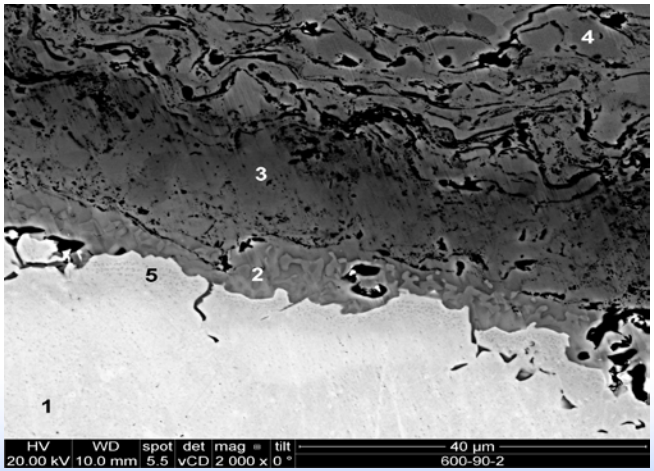
Grit Blasted Substrate	R_{RMS}, microns	R_t, microns
9Cr-1Mo steel	7.24	59.24
Carbon steel (1018)	7.65	58.75
316 Stainless Steel	7.73	81.62
Inconel 600	8.23	81.91

Coating Durability – Thermal Cycling – Interfacial reactions



Interface of Fe_3Al coating on carbon steel after 500 thermal cycles at 700°C





Interface of Fe_3Al coating on I-600 after 500 thermal cycles at 900°C

Idaho National Laboratory

Coating Durability – Thermal Cycling – Conclusions

- **A higher thermal spray chamber pressure results in more durable Fe_3Al coatings**
- **Surface roughness of the substrate is critical prior to deposition**
- **Modification of the surface preparation methods for 9Cr-1Mo steel should improve coating durability**
- **Some coatings/substrate systems were capable of withstanding 500 thermal cycles to 900°C**
- **Interface reactions were observed in high temperature thermal cycling of carbon steel and Inconel 600**

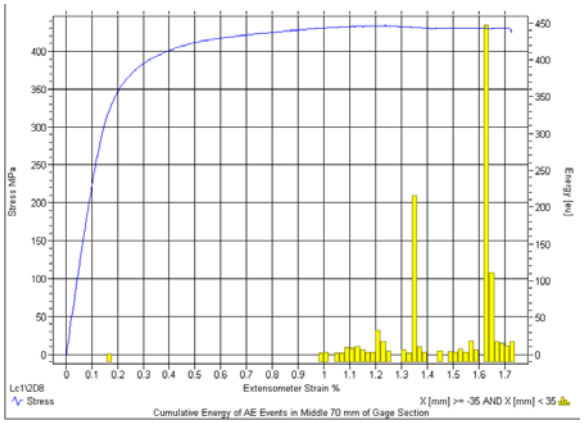
Coating Adhesion

Study the effect of substrate materials and thermal spray parameters on coating adhesion

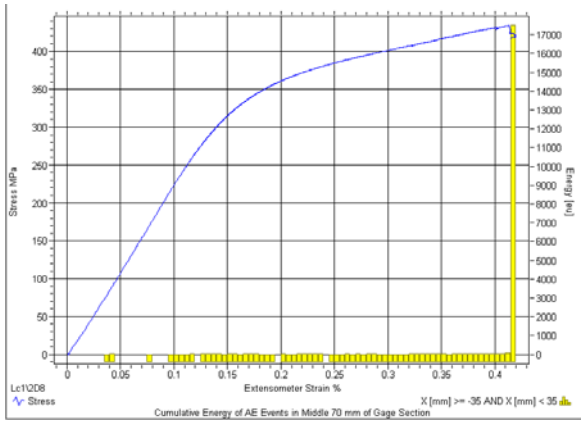
- **Substrate Materials: 316 SS, 9Cr-1Mo and Inconel 600**
- **Variables:**
  - Thermal spray chamber pressure (0.3, 0.6 & 0.7 MPa)
  - Coating thickness (250 and 500 microns)
- **Room temperature tensile tests using acoustic emission to identify coating failure**

Coating Adhesion - Results

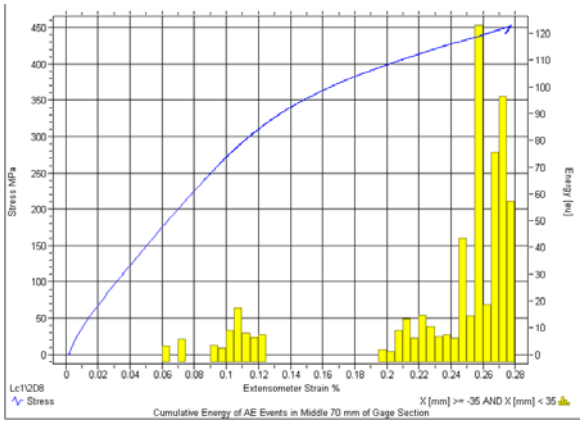
Inconel 600



P_C= 0.3 MPa, 250 microns thick (7 Passes)



P_C= 0.7 MPa, 250 microns thick (11 Passes)



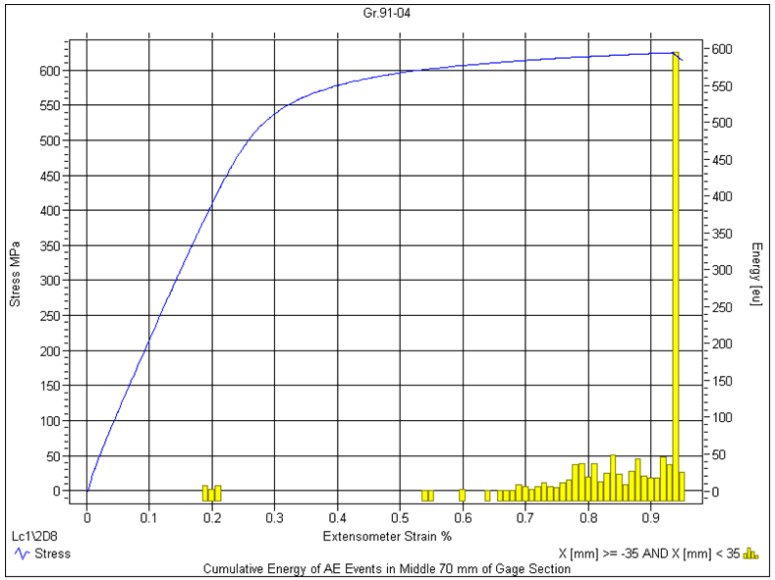
P_C= 0.6 MPa, 450 microns thick (20 Passes)

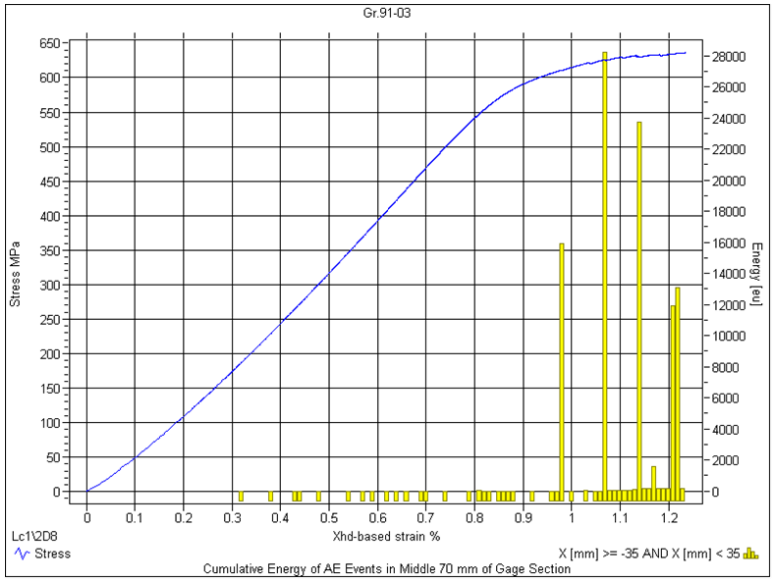
Results for 316 SS showed similar trends:

- Increasing P_C lowers strain to failure
- Increasing coating thickness lowers strain to failure

Idaho National Laboratory

Coating Adhesion – Results – 9Cr-1Mo Steel



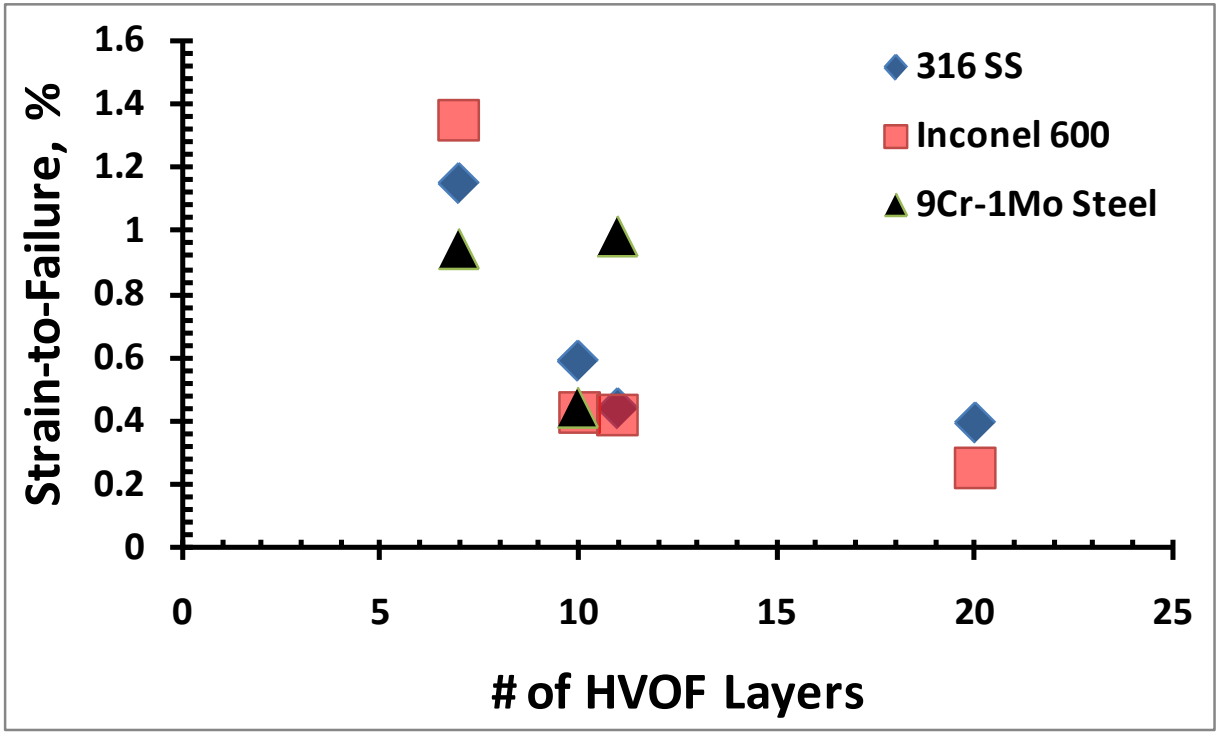


P_C= 0.3 MPa, 250 microns thick (7 Passes)

P_C= 0.7 MPa, 250 microns thick (11 Passes)

Different behavior results from different substrate surface roughness

Coating Adhesion – # of Layers



Combined Results

- **High chamber pressure improves thermal cycling durability**
- **Lower chamber pressure improves coating adhesion**
- **Thicker coatings decrease both thermal cycling durability and coating adhesion**
- **9Cr-1Mo steel results are different from 316 SS and Inconel 600 – seems to be related to surface roughness of the substrate.**

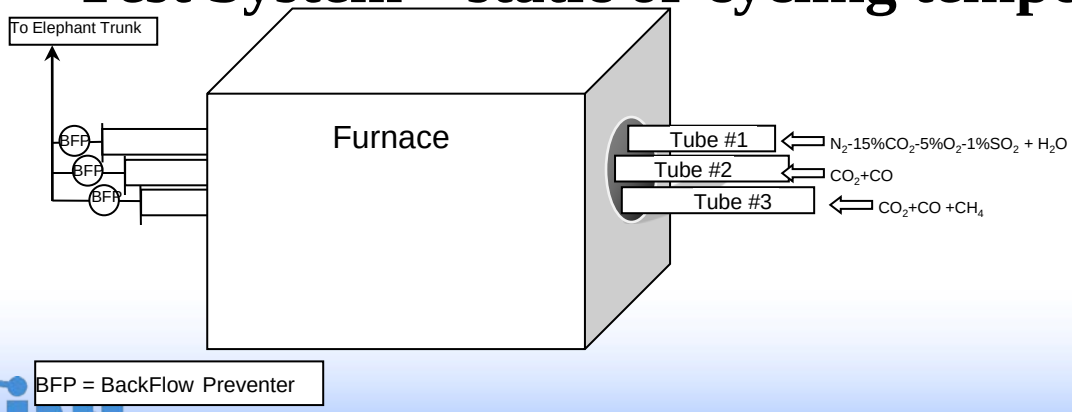
Additional Tasks

- **Metallography on coating adhesion samples**
  - Effect of thermal spray parameters on substrate surface roughness for various substrate materials
  - Effect of chamber pressure on oxide content in coating
- **CTE determination of coatings sprayed at different chamber pressures**
- **Elastic modulus of coatings**
- **Additional adhesion tests for 9Cr-1Mo steel substrates – including modified surface preparation**

Current Focus

Goal – Help define failure criteria

- **Coating behavior in:**
  - **Simulated fossil fuel combustion environment (N_2-15%CO_2-5%O_2-1%SO_2 + water vapor)**
  - **Reducing or oxidizing (mixtures of CO and CO_2)**
  - **Carburizing or de-carburizing (CH_4, CO and CO_2)**
- **Test System – static or cycling temperature**



Idaho National Laboratory

Summary & Conclusions

- The trends in chamber pressure, P_C, for thermal cycling durability and coating adhesion are opposite
- Surface condition of the substrate is critical and may have to be tailored to the specific substrate material
- Coating have been prepared that are capable of withstanding 500 thermal cycles to temperatures up to 900°C
- Fe_3Al coatings on nickel-based substrates exhibit significant reaction at high temperatures and may require a diffusion barrier