Microstructure, Processing, Performance Relationships for High Temperature Coatings

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Goals

- Develop coatings for high temperature service in fossil fuel environments
  - Develop coating materials – Iron aluminide (Fe_3Al)
  - Develop methods for applying coatings – HVOF
  - Understand factors that affect the reliability of HVOF coatings
- Transfer the techniques and coatings to industry
  - Demonstrate reproducibility and reliability of coatings
  - Field testing – industrial partner
  - Demonstrate repair methodologies

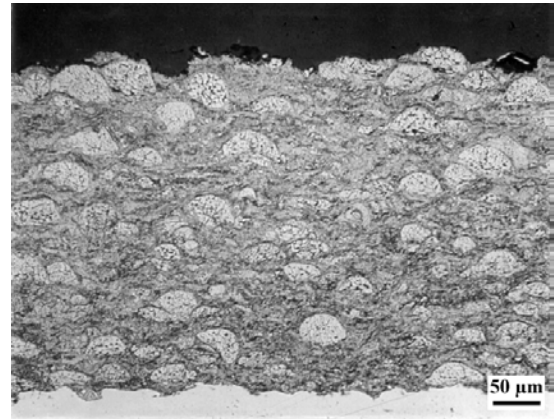
Past Results

- Thermal spray parameters can be used to generate highly dense coating with varying levels of residual stress

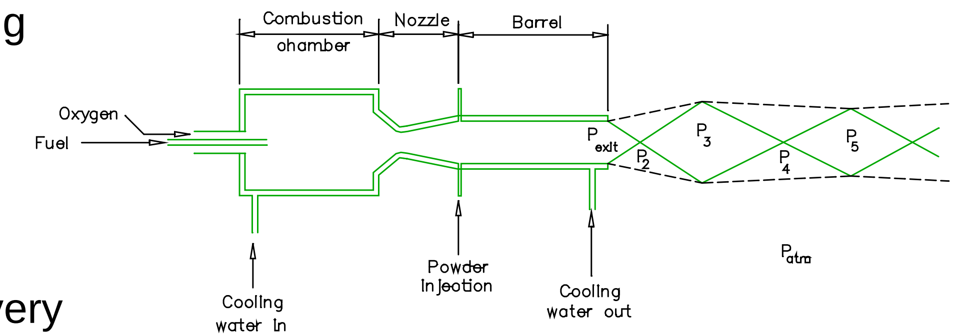
- Residual stresses in coating arise from three sources
  - CTE mismatch between coating and substrate
  - Quench stresses
  - “Peening” stress

- Corrosion resistance of coating is very close to wrought material

- Coating failure governed by cracking and delamination



Fe_3Al Coating



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*

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
- Determine the influence of substrate properties on coating durability during thermal cycling
- Determine the influence of thermal spray parameters and substrate properties on coating adhesion

Parameters of Interest

Objective: Identify parameters that result in adherent, high-durability coatings

- Materials parameters
  - CTE difference between coating and substrate
  - Microstructure stability
- High-Velocity Oxy-Fuel (HVOF) thermal spray parameters
  - Chamber pressure – particle velocity
  - Fuel/oxygen ratio – particle temperature
  - Substrate temperature during spraying – standoff distance, traverse speed, preheat/active cooling
  - Coating thickness - # of passes

Effects of Substrate Material, Substrate Thickness and Coating Thickness on Thermal Cycling Durability

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Substrate Temperature During Spraying

- Maximum temperature attained by the substrate varied from sample to sample.
- In general, substrate thickness and material had little effect on max. temperature attained during spraying.
- Coating thickness did not strongly influence max. temperature.
- Lack of cooling air (Plate 9) resulted in a significantly higher max. temperature.
- Chamber pressure/particle velocity was constant during application of coating

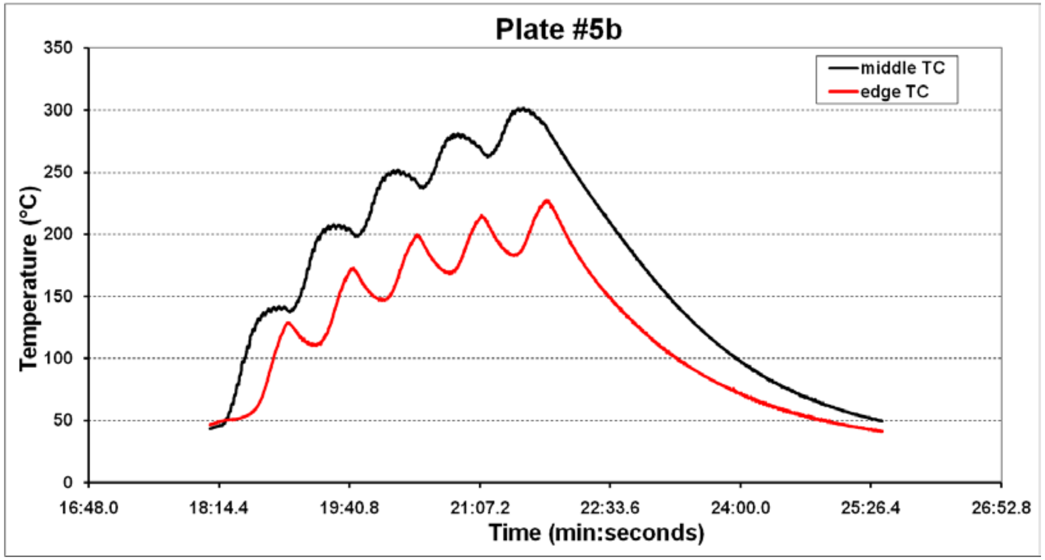

Plate #	Plate Material	Plate Thickness, mm	Number of Layers	Average, microns	Approx. Max Temp., °C
3	Carbon Steel	12.7	3	218	220
9*	Carbon Steel	12.7	5	364	540
4	Carbon Steel	12.7	5	402	280
8	Carbon Steel	19.1	3	249	210
6	Carbon Steel	19.1	3	439	240
7	Grd. 91	19.1	3	223	210
1	Grd. 91	19.1	3	246	190
2	Grd. 91	19.1	5	385	190
5	Grd. 91	19.1	5	416	300

* No cooling air to the substrate

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Microstructural Characterization

Plate #	Plate Material	Plate Thickness, mm	Number of Layers	Coating Thickness from optical image analysis #1	#2	#3	#4	Average, microns	Phase Fraction by Image Analysis Porosity, %	Melted, %	Solid, %
3	Carbon Steel	12.7	3	224	225	212	209	**218**	0.2	23.3	76.6
9	Carbon Steel	12.7	5	384	371	359	342	**364**	0.1	22.3	77.7
4	Carbon Steel	12.7	5	430	397	403	379	**402**	0.6	20.3	79.1
8	Carbon Steel	19.1	3	228	271	245	252	**249**	0.1	21.9	78.0
6	Carbon Steel	19.1	3	423	430	448	453	**439**	0.1	19.8	80.1
7	Grd. 91	19.1	3	235	195	236	224	**223**	0.9	23.2	75.9
1	Grd. 91	19.1	3	240	241	252	251	**246**	0.1	18.9	81.0
2	Grd. 91	19.1	5	398	397	368	376	**385**	0.1	21.5	78.4
5	Grd. 91	19.1	5	387	426	421	430	**416**	0.2	20.9	78.9

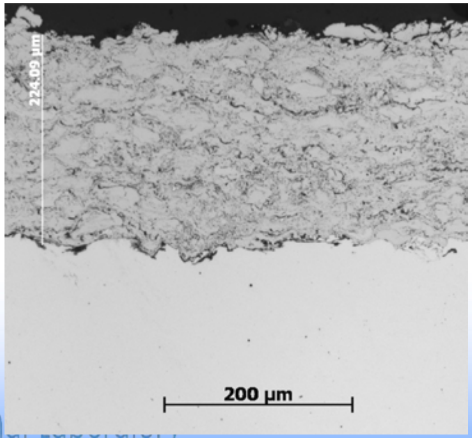


Plate 7

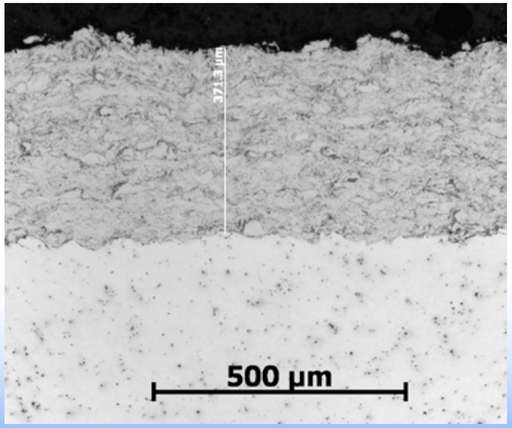
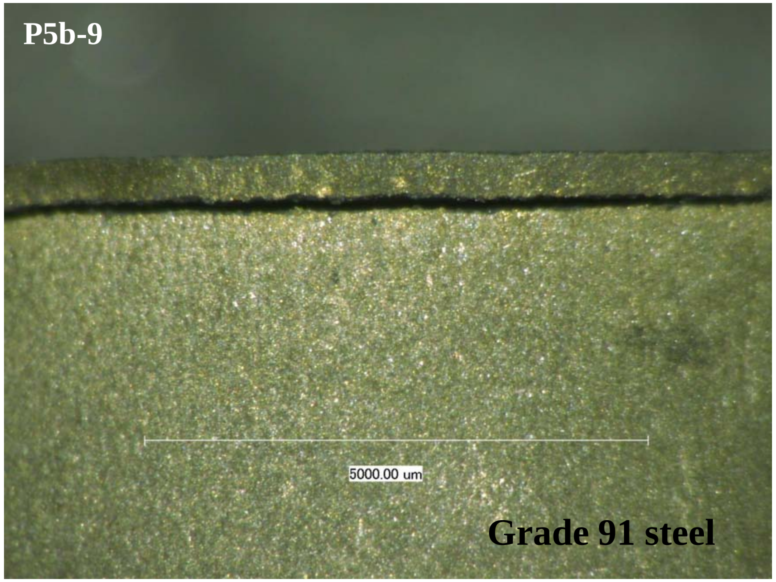
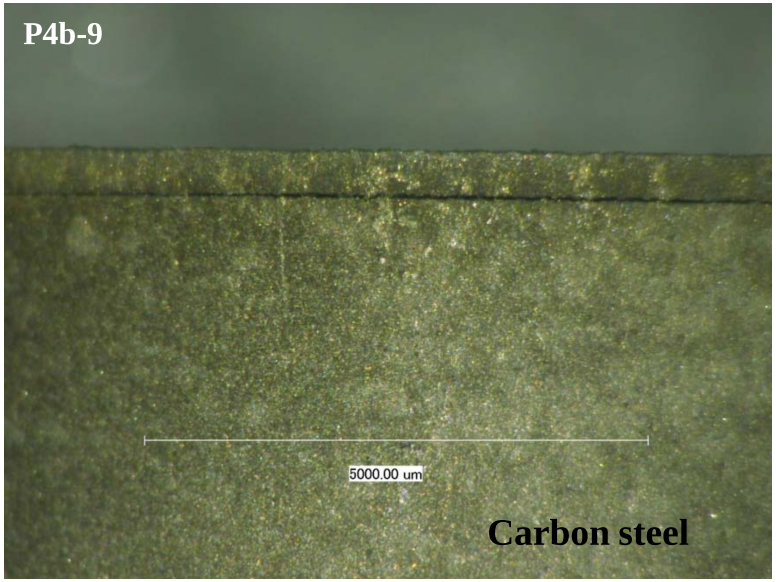
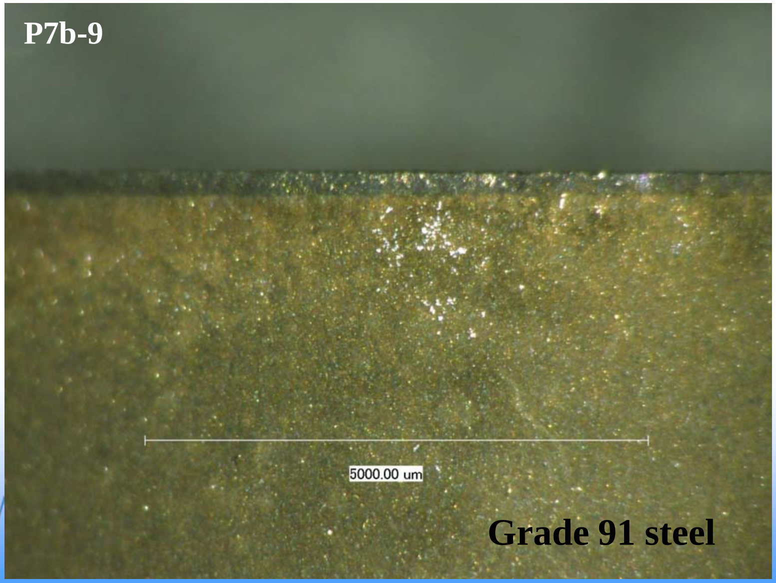


Plate 9

Coating Failure Modes During Thermal Cycling







All were thermal cycled at 700°C for up to 500 cycles – encapsulated with UHP Ar atmosphere

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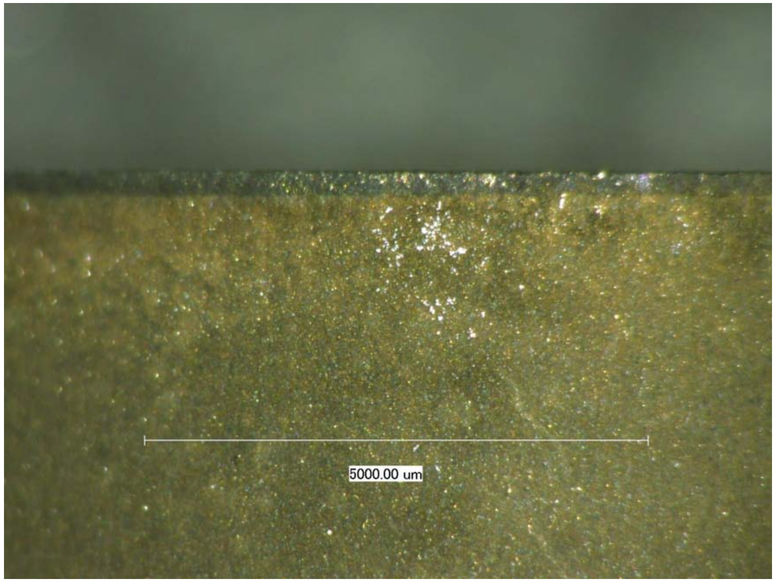
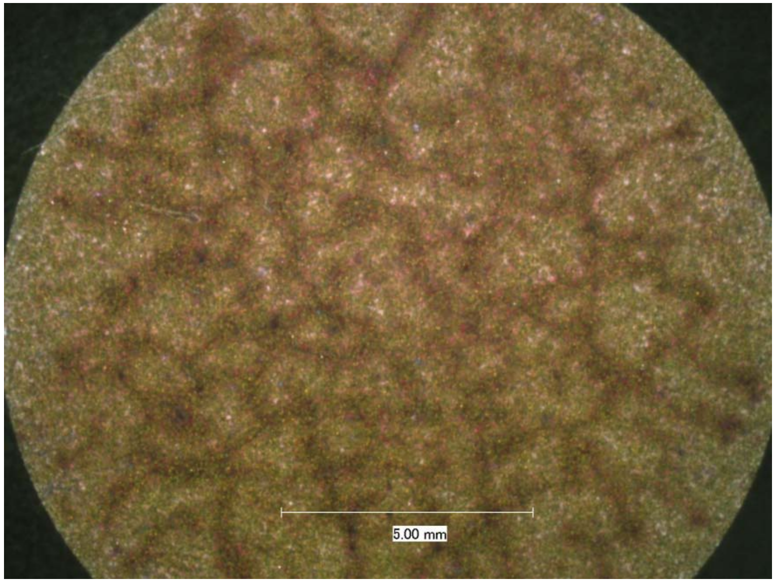
Coating Failure Modes During Thermal Cycling – cont.





Sample P7b-9, thermal cycled at 700^{o}C for up to 500 cycles – encapsulated with UHP Ar atmosphere. Fe_3Al on 19.1 mm thick Grade 91 steel plate, coating thickness ~220 microns.

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Current Thermal Cycling Results

Sample ID	Plate Material	Plate thickness, mm	Max. Temp., ºC	Coating Thickness, microns	Pass/Fail	Cycle Temp, ºC	Cycles to failure
P8b-6	Carbon Steel	19.1	210	249	*	620	300+
P3b-6	Carbon Steel	12.7	220	218	*	620	300+
P6b-6	Carbon Steel	19.1	240	439	*	620	300+
P4b-6	Carbon Steel	12.7	280	402	F	620	300
P9b-6	Carbon Steel	12.7	540	364	F	620	300
P8b-9	Carbon Steel	19.1	210	249	F	700	300
P3b-9	Carbon Steel	12.7	220	218	P	700	500+
P6b-9	Carbon Steel	19.1	240	439	F	700	100
P4b-9	Carbon Steel	12.7	280	402	F	700	100
P9b-9	Carbon Steel	12.7	540	364	F	700	100
P1b-6	Grd. 91	19.1	190	246	*	620	300+
P2b-6	Grd. 91	19.1	190	385	*	620	300+
P7b-6	Grd. 91	19.1	210	223	*	620	300+
P5b-6	Grd. 91	19.1	300	416	F	620	100
P1b-9	Grd. 91	19.1	190	246	P	700	500+
P2b-9	Grd. 91	19.1	190	385	P	700	500+
P7b-9	Grd. 91	19.1	210	223	F	700	<500
P5b-9	Grd. 91	19.1	300	416	F	700	100

* Samples have survived 300 cycles and are undergoing the remaining 200 cycles.

Current Thermal Cycling Results – cont.

Sample ID	Plate Matl	Plate thickness, mm	Max. Temp., °C	Coating Thickness, microns	Pass/Fail	Cycle Temp, °C	Cycles to failure
P1b-6	Grd. 91	19.1	190	246	*	620	
P2b-6	Grd. 91	19.1	190	385	*	620	
P8b-6	Carbon Steel	19.1	210	249	*	620	
P7b-6	Grd. 91	19.1	210	223	*	620	
P3b-6	Carbon Steel	12.7	220	218	*	620	
P6b-6	Carbon Steel	19.1	240	439	*	620	
P4b-6	Carbon Steel	12.7	280	402	F	620	300
P5b-6	Grd. 91	19.1	300	416	F	620	100
P9b-6	Carbon Steel	12.7	540	364	F	620	300
P1b-9	Grd. 91	19.1	190	246	P	700	500+
P2b-9	Grd. 91	19.1	190	385	P	700	500+
P8b-9	Carbon Steel	19.1	210	249	F	700	300
P7b-9	Grd. 91	19.1	210	223	F**	700	<500
P3b-9	Carbon Steel	12.7	220	218	P	700	500+
P6b-9	Carbon Steel	19.1	240	439	F	700	100
P4b-9	Carbon Steel	12.7	280	402	F	700	100
P5b-9	Grd. 91	19.1	300	416	F	700	100
P9b-9	Carbon Steel	12.7	540	364	F	700	100

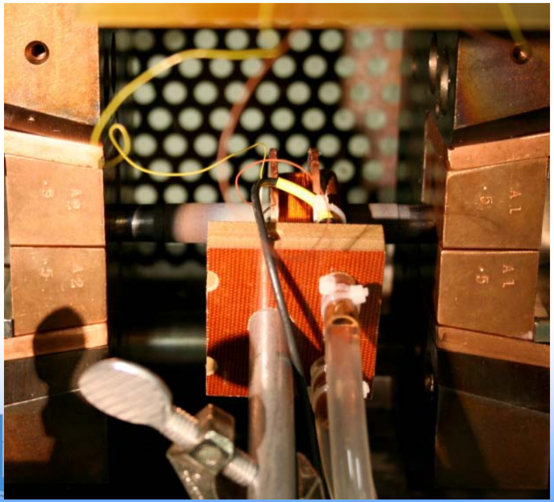
* Samples have survived 300 cycles and are undergoing the remaining 200 cycles.

**Failure by through-thickness cracks

Thermal Cycling – Gleeble Results

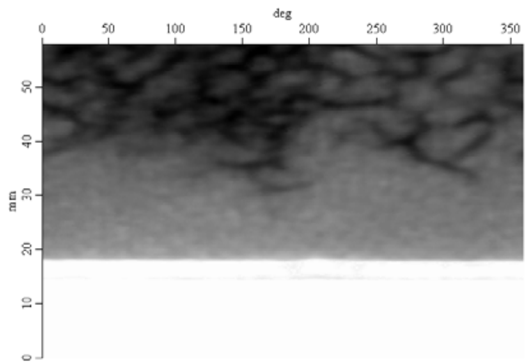
Effect of Chamber Pressure

Sample ID	Substrate Material	Chamber Pressure, MPa	Cycle Temerature, ^{o}C	Pass/Fail	Comments
SS-50-1	Stainless Steel	0.3	650	Pass	
SS-50-5	Stainless Steel	0.3	700	Pass	Microstructural changes in the substrate
SS-90-4	Stainless Steel	0.6	700	Pass	Microstructural changes in the substrate
600-50-1	Inconel 600	0.3	650	Pass	
600-50-5	Inconel 600	0.3	700	Pass	Optical metallography shows delam
600-90-4	Inconel 600	0.6	700	Pass	No delam in Optical metallography
600-50-4	Inconel 600	0.3	800	Pass	Metallography not complete
600-90-3	Inconel 600	0.6	800	Pass	Metallography not complete
91-50-1	Grade 91 steel	0.3	650	Failed	Okay after 250 cycles
91-90-1	Grade 91 steel	0.6	650	Failed	Cracks initiated at the TC weld - okay after 250 cycles
91-50-5	Grade 91 steel	0.3	700	Failed	Did not survive even 250 cycles
91-90-5	Grade 91 steel	0.6	700	Failed	Cracks initiated at the TC weld - okay after 250 cycles

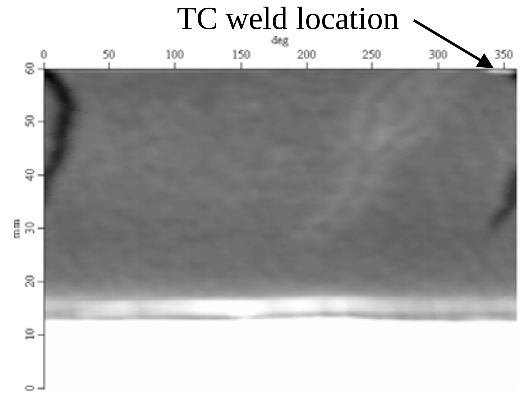


- Fe_3Al coatings
- Results are for 500 cycles
- All coatings approximately the same thickness, ~250 microns
- Particle velocity:
  - @0.3 MPa = 570 m/s
  - @ 0.6 MPa = 630 m/s
- Particle Temperature:
  - @0.3 MPa = 1750^{o}C
  - @ 0.6 MPa = 1600^{o}C

Eddy Current Scans for Grade 91

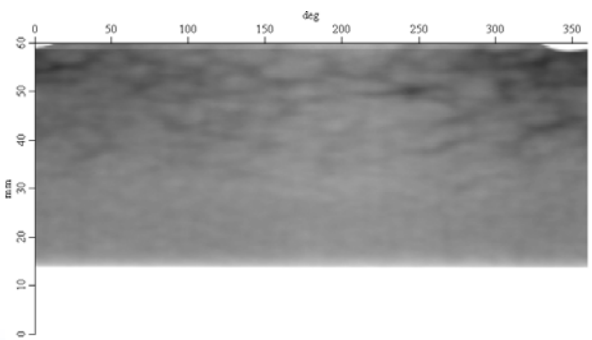


91-50-1 after 500 cycles
(0.3 MPa Chamber Pressure)

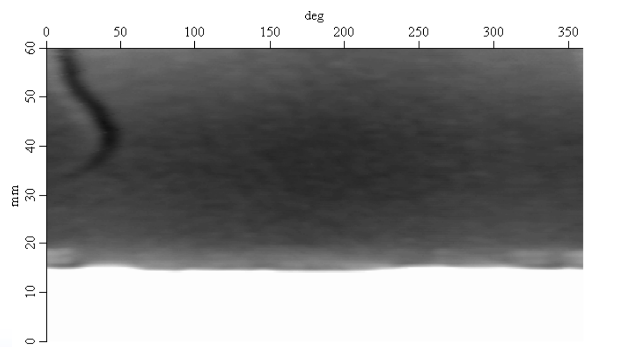


91-90-1 after 500 cycles
(0.6 MPa Chamber Pressure)

650ºC



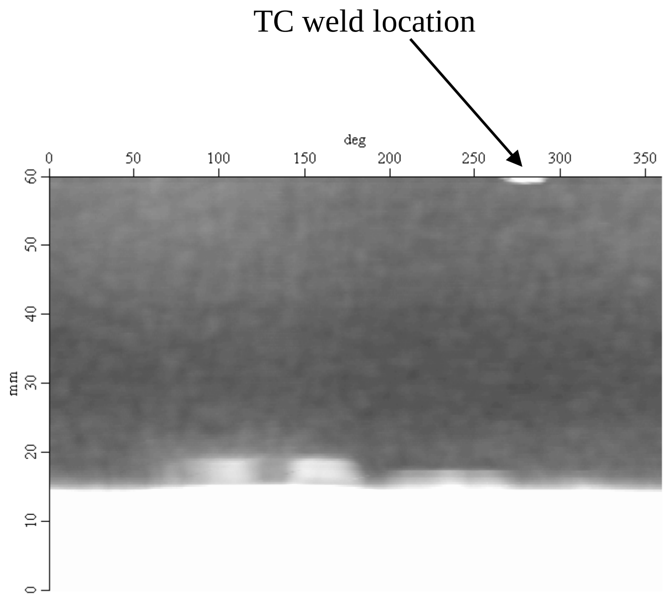
91-50-5 After 250 cycles
(0.3 MPa Chamber Pressure)



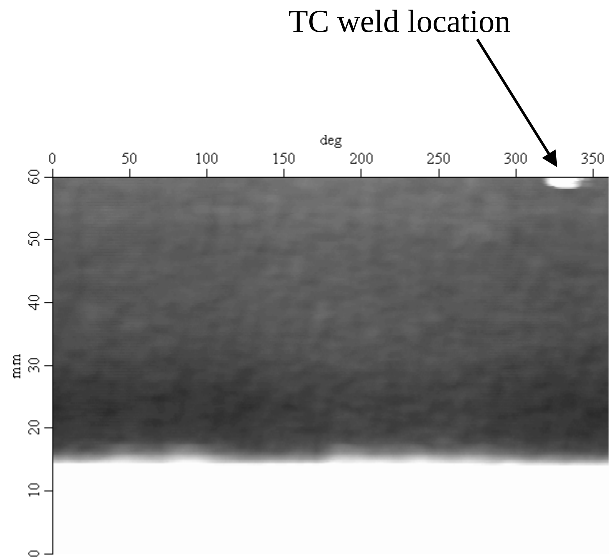
91-90-5 After 500 cycles
(0.6 MPa Chamber Pressure)

700ºC

Eddy Current Scans for Stainless Steel

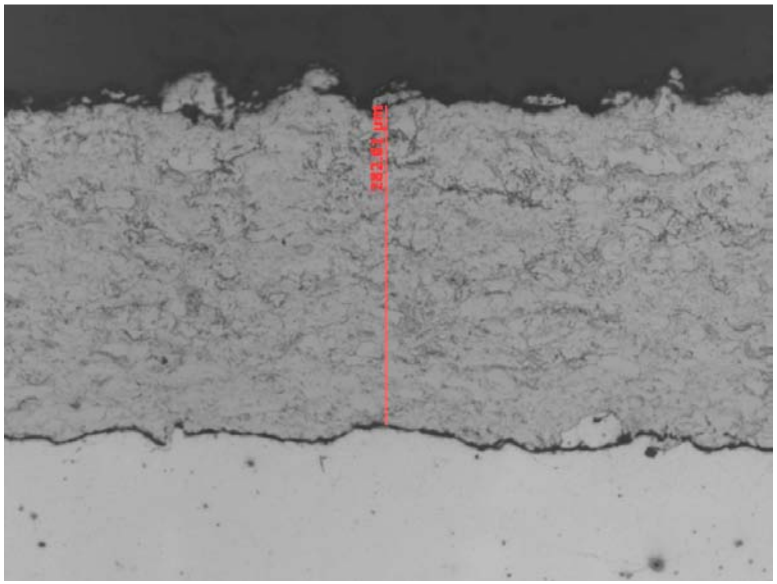


SS-90-4 after 500 cycles to 700^{o}C
(0.6 MPa Chamber Pressure)

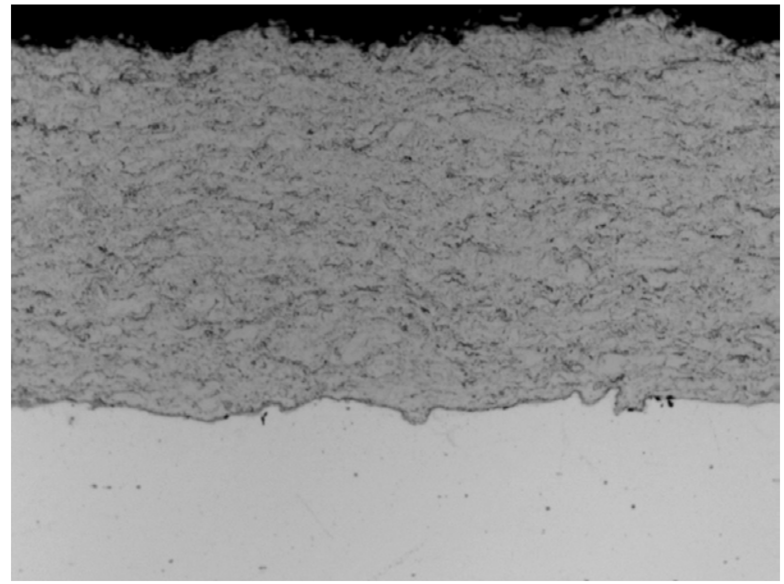


SS-50-5 after 500 cycles to 700^{o}C
(0.3 MPa Chamber Pressure)

Evidence of Delamination



600-50-5: 500 cycles @ 700^{o}C
Chamber Pressure = 0.3 MPa



600-90-5: 500 cycles @ 700^{o}C
Chamber Pressure = 0.6 MPa

Inconel 600

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Potential Causes for Observed Behavior

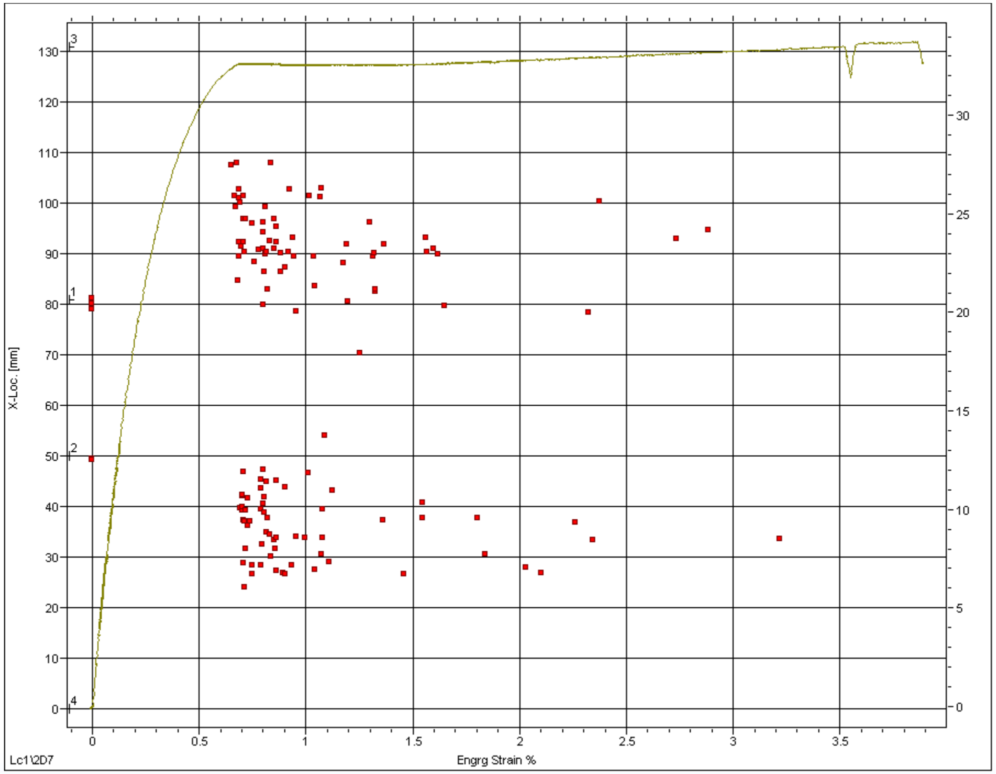
- **Differences between CTE of coating and base metal:**

Fe_3Al Coating (0.3 MPa)	= 13 x $10^{-6}/^{o}C$
Fe_3Al Coating (0.6 MPa)	= 12 x $10^{-6}/^{o}C$
Carbon Steel (1080)	= 12.2 x $10^{-6}/^{o}C$
Stainless Steel (316SS)	= 18.2 x $10^{-6}/^{o}C$
Grade 91 Steel (9Cr-1Mo)	= 13.3 x $10^{-6}/^{o}C$
Inconel 600 (Ni-base)	= 14.0 x $10^{-6}/^{o}C$

- **Heat capacity/conductivity of substrate – substrate temperature rise.**
- **Oxidation of base metal during application**
- **Diffusion between coating and substrate**
- **Strength of coating**
- **Substrate strength/deformation of substrate material (?)**

Coating Adhesion Via Tensile Testing

- Coating strain to fracture measured using acoustic emission monitoring
- 300 µm coatings applied to round tensile specimen substrates
- Two AE sensors attached to each end of substrate
- Coating cracking produces clear AE signals
- Crack initiation appears to be concentrated at ends of the coating.
- Modifications to specimen geometery are currently being made.



Cracking strain ~0.7%

Cracking stress ~477 MP (SS – $\sigma_{YS} \approx$ 415 MPa)

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Summary & Conclusions

- **It appears that substrate temperature – as affected by cooling air, substrate thickness and # of coating layers – influences coating durability during cycling.**
- **Chamber pressure/particle velocity during HVOF coating application influences the durability of the coating during thermal cycling with a higher particle velocity producing a more durable coating.**
- **Coating failure occurs by either through-thickness cracking or delamination at the coating/substrate interface.**
- **Coating have been prepared that are capable of withstanding 500 thermal cycles to temperatures up to 800^{o}C.**

Summary & Conclusions

- **Complete thermal cycling at two additional temperatures on both the coated plates and rods.**
- **Analysis of the coating/substrate interface of cycled samples.**
- **Analysis of interface stability samples (650^{o}C for 5000 hrs).**
- **Apply coatings to temperature controlled substrates.**
- **Evaluate coating strength as a function of chamber pressure using tensile testing with AE crack detection.**