



# HCAT MEETING 16 March 2005



## In Search of the Holy Grail (of EHC Alternatives)

Joe Kolek  
AFRL/MLSC

# Report Documentation Page

*Form Approved*  
*OMB No. 0704-0188*

Public reporting burden for the collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to a penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.

1. REPORT DATE <b>16 MAR 2005</b>	2. REPORT TYPE	3. DATES COVERED <b>00-00-2005 to 00-00-2005</b>			
4. TITLE AND SUBTITLE <b>In Search of the Holy Grail (of EHC Alternatives)</b>		5a. CONTRACT NUMBER			
		5b. GRANT NUMBER			
		5c. PROGRAM ELEMENT NUMBER			
6. AUTHOR(S)		5d. PROJECT NUMBER			
		5e. TASK NUMBER			
		5f. WORK UNIT NUMBER			
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) <b>Air Force Research Laboratory, AFRL/MLSC, Wright Patterson AFB, OH, 45433</b>		8. PERFORMING ORGANIZATION REPORT NUMBER			
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)		10. SPONSOR/MONITOR'S ACRONYM(S)			
		11. SPONSOR/MONITOR'S REPORT NUMBER(S)			
12. DISTRIBUTION/AVAILABILITY STATEMENT <b>Approved for public release; distribution unlimited</b>					
13. SUPPLEMENTARY NOTES <b>25th Replacement of Hard Chrome and Cadmium Plating Program Review Meeting, March 15-17, 2005, Greensboro, NC. Sponsored by SERDP/ESTCP.</b>					
14. ABSTRACT					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT <b>Same as Report (SAR)</b>	18. NUMBER OF PAGES <b>46</b>	19a. NAME OF RESPONSIBLE PERSON
a. REPORT <b>unclassified</b>	b. ABSTRACT <b>unclassified</b>	c. THIS PAGE <b>unclassified</b>			



# Quests Underway



- Non-Line of Sight (NLOS) Hard Chrome Alternatives
- Advanced Non-Line of Sight (ANLOS) Hard Chrome Alternatives
- Alternative Spray Process Coatings Evaluation
- Metal-Coated Particles Evaluation
- Effects of Chemicals on HVOF Coatings Follow-up



# Non-Line of Sight (NLOS) Hard Chrome Alternatives



# NLOS Hard Chrome Alternatives



- High Velocity Oxygen Fuel (HVOF) thermal spray of Tungsten Carbide-Cobalt (WC-17Co) is being transitioned by the AF as the primary alternative to Electrolytic Hard Chrome (EHC) plating
- HVOF is a line of sight process unable to accommodate internal diameters, blind holes and complex shapes
- Non-Line of Sight (NLOS) plating requirements are 20 – 40% of the ALC workload and an alternative to EHC for these applications is required if the use of chrome plating is to be minimized / eliminated



# NLOS Hard Chrome Alternatives



- Potential alternatives search and initial down-selection
- Screening testing of 10 candidates
  - **Least costly testing done first**
  - **OC-ALC provided EHC coatings**
  - **Each vendor applied its own product in order to eliminate any chance of processing anomalies**
    - .005” thick coatings were requested
    - Coatings ground to .003”
    - 2 coating variations
      - As deposited
      - Baked at 375°F/24 hrs (Standard hydrogen relief bake)



# NLOS Hard Chrome Alternatives



## Initial Screening Testing

Adhesion: ASTM B571 Mandrel & Vise Bend tests

Hardness: HVN determination w/ 75 gram indenter

Profilometry: Smoothness / leveling ability of coating

Chemical composition: No unacceptable constituents

Quality: Visual analysis IAW Fed Spec QQ-C-320B

Taber wear index: Weight loss per 1000 cycles

- **Selected the 4 best processes for further evaluation**



# NLOS Hard Chrome Alternatives



## Niplate 700 by Surface Technology

- Electroless Nickel (95%) – Phosphorous (5%) with silicon carbide particles

## UltraCem by Universal Chemical

- Electroless Nickel (95%) – Boron (5%) that forms crystalline clusters of nickel boride

## Nanon 9 by Nanon Technologies

- Electrolytic Nickel (50-70%) – Cobalt (30-50%) that forms nanocrystalline microstructure

## NiCom by US Chrome

- Electrolytic Nickel w/ silicon carbide particles



# NLOS Hard Chrome Alternatives



## Mandrel Bend Adhesion Testing

**Mandrel radius 4 X thickness of 4130 steel substrate (0.090")**





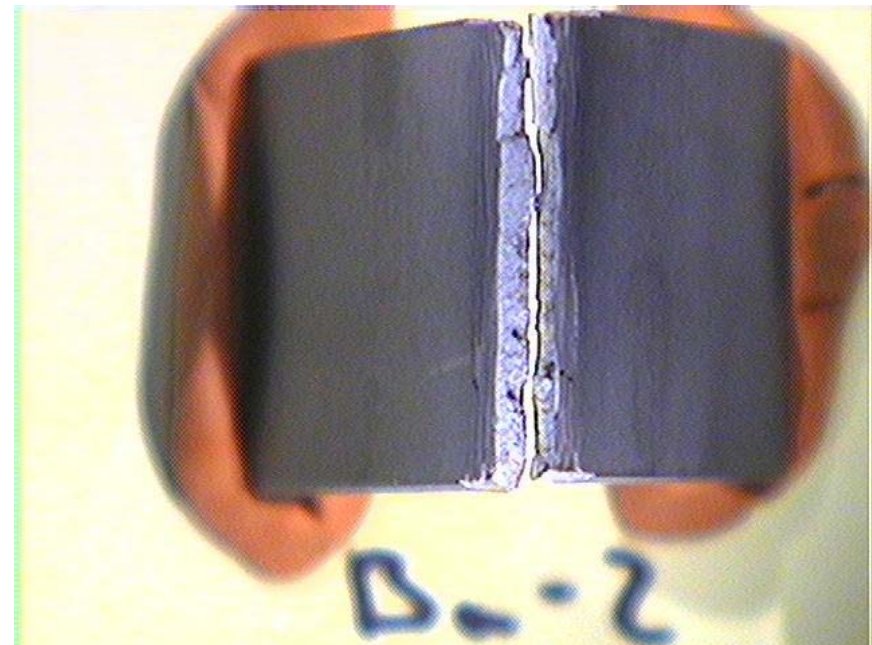
# NLOS Hard Chrome Alternatives



## Typical Mandrel Bend Test Results ASTM B571 Section 3.1



**EHC; Nanon 9; Unbaked UltraCem**



**Niplate 700; NiCom; Baked UltraCem**

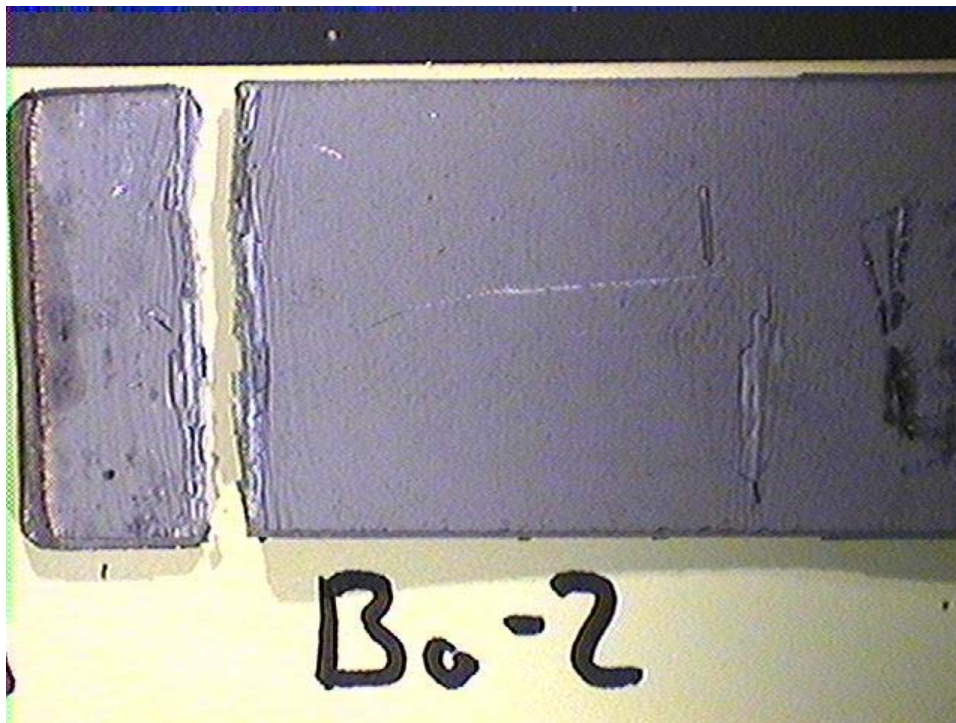


# NLOS Hard Chrome Alternatives



## Typical Vice Bend Test Results

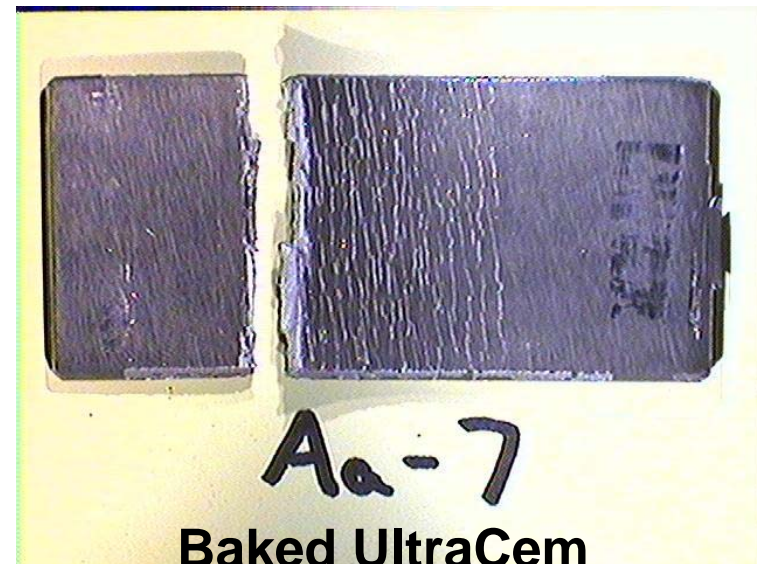
ASTM B571 Section 3.2



Niplate 700



Unbaked UltraCem



Baked UltraCem



# NLOS Hard Chrome Alternatives



## Initial Screening Test Results

	Niplate 700	UltraCem	Nanon 9	NiCom
Adhesion EHC Pass 3.1 Fail 3.2	As deposited & Baked Pass	As deposited: Pass 3.1 Fail 3.2 Baked: Pass	As deposited & Baked Pass	As deposited: Pass Baked: Pass 3.1 Fail 3.2
Hardness EHC = 895 HVN	781 (As deposited) 939 (Baked)	872 (As deposited) 910 (Baked)	934 (As deposited) 927 (Baked)	434 (As deposited) 541 (Baked)
Profilometry	Acceptable	Acceptable	Acceptable	Acceptable
Composition	As Claimed	As Claimed	As Claimed	As Claimed
Quality	Acceptable	Acceptable	Acceptable	Acceptable
Taber Wear Index EHC = 2.2 (10 or less acceptable)	2.8 (As deposited) 2.6 (Baked)	9.8 (As deposited) 14.0 (Baked)	12.0 (As deposited) 10.8 (Baked)	9.7 (As deposited) 6.0 (Baked)



# NLOS Hard Chrome Alternatives



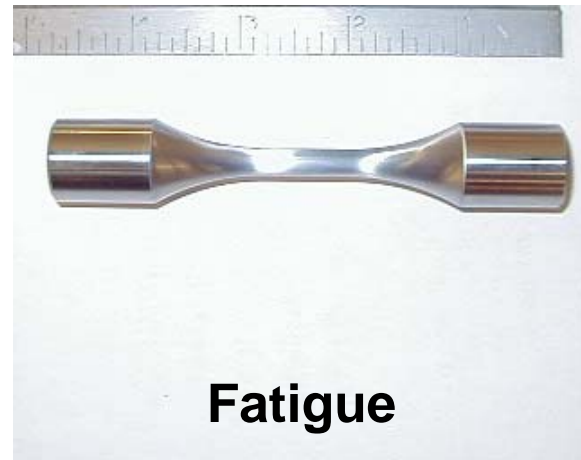
- **Final Screening Testing**
  - **Corrosion:** B-117 (1000hrs) and Electrochemistry Eval.
  - **Fatigue:** @185 ksi, R=.1, 10 cycles / sec until failure
  - **Hydrogen Embrittlement:** ASTM F519
  - **Block-on-Ring Wear:** ASTM G77-98
  - **Grindability determination**
  - **Strippability determination**



# NLOS Hard Chrome Alternatives Test Specimen



**Corrosion  
4130 steel**



**Fatigue  
4340 steel**



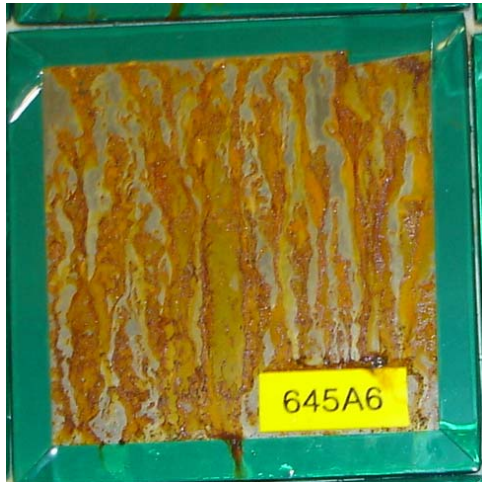
**Hydrogen Embrittlement  
4340 steel**



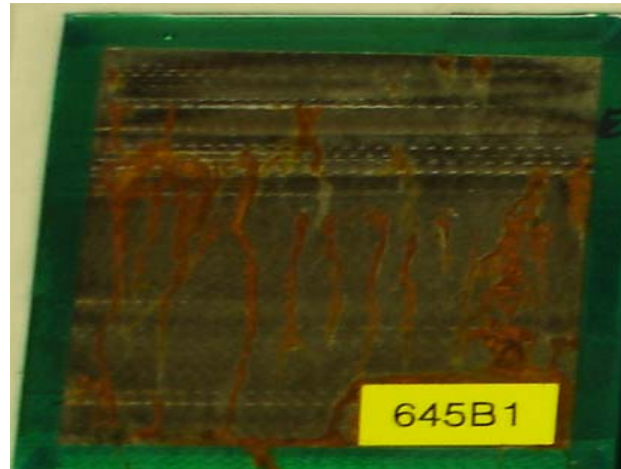
**Block-on-Ring Wear  
Both 4340 steel  
Only block is coated**



# NLOS Hard Chrome Alternatives After 20 hrs



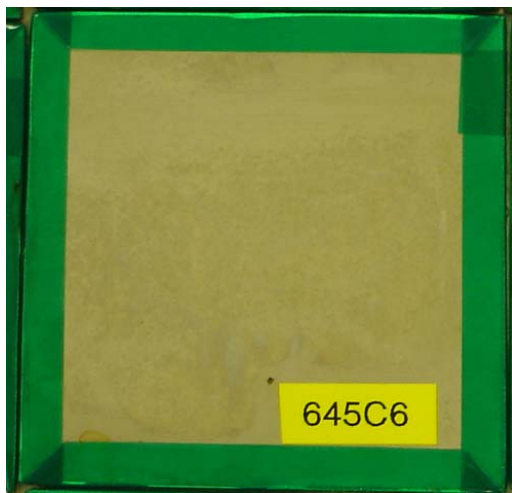
**UltraCem**



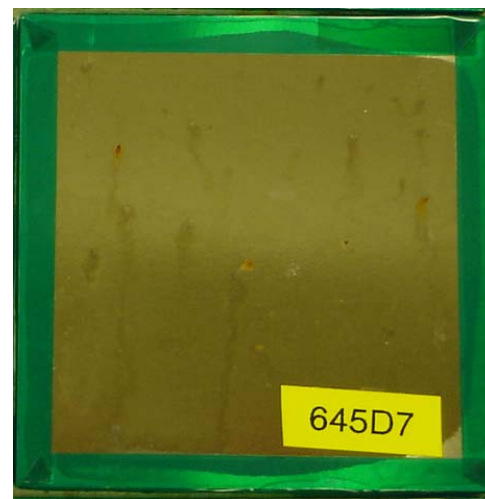
**Niplate 700 Ground**



**Niplate 700 Unground**



**NiCom**



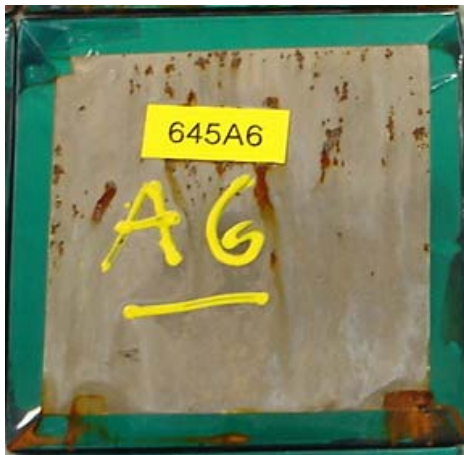
**Nanon 9**



**EHC**



# NLOS Hard Chrome Alternatives After 1000 hrs



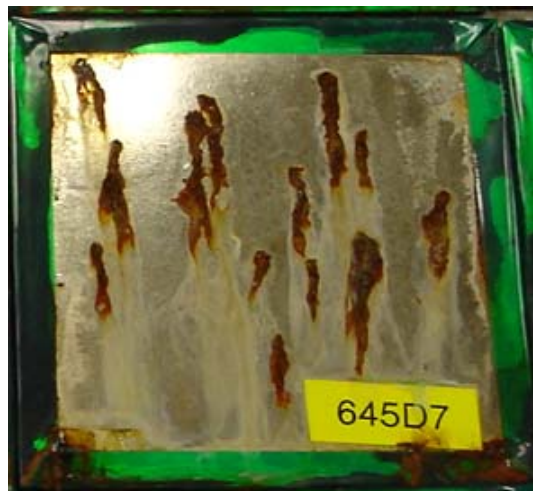
**UltraCem  
Unground**



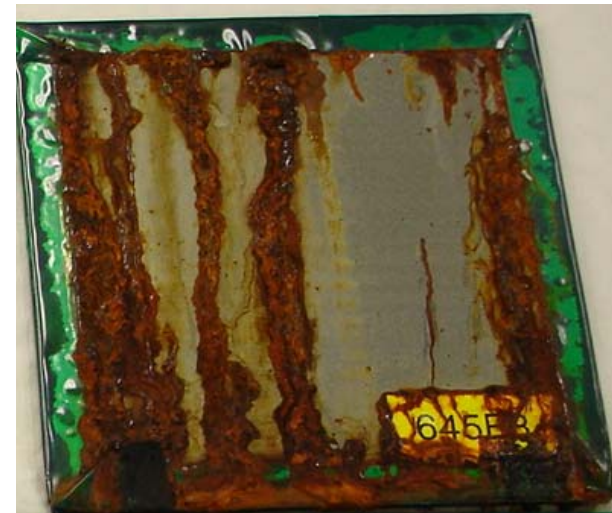
**Niplate 700  
Unground**



**NiCom**



**Nanon 9**



**EHC**



# Electrochemistry Evaluation



- To evaluate the electrochemical corrosion resistance of EHC and the alternative coatings in a mild (3.5%) NaCl solution.
- High Impedance ( $|Z|$ ) is desirable. Indicates coating is better barrier to the flow of electrons. This metric is the most indicative of the coating performance expected in a field application.
- Difference between  $E_{corr}$  (equivalent to Open Circuit Potential) and  $E_{pit}$  (Pitting Potential) indicates voltage required to induce pitting. Higher difference indicates greater corrosion resistance.
- Low Corrosion Current ( $I_{corr}$ ) is desirable. Indicates that a larger current density is required to cause coating failure.
- $|Z|$  primary metric. Use  $\Delta$  between  $E_{corr}$  and  $E_{pit}$  if coatings have same magnitude of  $|Z|$ . If needed (it wasn't), use  $I_{corr}$  to further distinguish between performances of two coatings. <sup>16</sup>



# Electrochemistry Evaluation



- $|Z|$ : EHC =  $3.66 \times 10^5$  ohm-cm ~ Niplate 700 =  $3.55 \times 10^5$  ohm-cm
- $|Z|$ : NANON 9 =  $1.01 \times 10^5$  ohm-cm
- $|Z|$ : UltraCem and NiCom values more than order of magnitude less
- Difference between  $E_{corr}$  &  $E_{pit}$ : EHC = **875**mV NiPlate 700 = **200**mV  
NANON 9 = **80** mV

Result of overall weighted average rankings

#1: EHC

#2: NiPlate 700 ( a close second to EHC )

#3: NANON 9

#4 / #5 : UltraCem and NiCom ( clearly inferior to the other alternatives )



# NLOS Hard Chrome Alternatives



## Final Screening Test Results

	Niplate 700	UltraCem	Nanon 9	NiCom
B117 Corrosion Testing	#3 As good as EHC	#2 As good as EHC	#4 As good as EHC	#1 As good as EHC
Electrochemistry Evaluation EHC is best	#1 Almost as good as EHC	#3 Significantly Inferior	#2	#4 Significantly Inferior
Fatigue EHC: 5801 - 7661 cycles to failure	Pass Fatigue Debit < EHC 42000 - 93000 cycles	Pass Fatigue Debit < EHC 7951 - 13871 cycles	Fail Fatigue Debit > EHC 1020 - 1738 cycles	Pass Fatigue Debit < EHC 19587 - 124367 cycles
Hydrogen Embrittlement EHC (baked): Pass	Pass (as deposited and baked)	Pass (as deposited and baked)	Pass (as deposited and baked)	Fail: as deposited Pass: baked
Block-on-Ring Testing EHC scar depth: 0.88 mil	0.90 mil (as deposited) 0.59 mil (baked)	2.49 mil (as deposited) 2.57 mil (baked)	2.62 mil (as deposited) 4.60 mil (baked)	3.21 mil (as deposited) 3.19 mil (baked)
EHC Coefficient of Friction: 0.7	0.7 (as deposited) 0.7 (baked)	0.8 (as deposited) 0.9 (baked)	0.7 (as deposited) 0.8 (baked)	0.6 (as deposited) 0.7 (baked)
Grindability	I Difficult to grind	Acceptable (coating on corrosion panels cracked)	Best surface finish	Acceptable
Strippability: 3 mil coating EHC: 1hr w/50% HCL	Acceptable 3 - 4 hrs w/ Nistrip R501	Acceptable 3 - 4 hrs w/ Nistrip R501	Acceptable 3 - 4 hrs w/ Nistrip R501	Acceptable 3 - 4 hrs w/ Nistrip R501



# NLOS Hard Chrome Alternatives



- **Niplate 700: Best Alternative**
- **Hardness and Wear performance comparable to EHC**
- **Best adhesion test performance**
- **Best electrochemistry evaluation results**
- **Corrosion test performance as good as EHC**
- **Passed hydrogen embrittlement and fatigue testing**
- **Optimum grinding technique needs to be determined**



# NLOS Hard Chrome Alternatives



## Validation Testing of NiPlate 700

- Establish coating capability at NDCEE
- Coat and test specimen to ensure that performance of coating is equivalent to that applied by Surface Technology
- Demonstrate NiPlate 700 can plate components with NLOS requirements

## ALC Implementation Plan

- Identify facility requirements and cost to establish plating capability
- Facilitate transition



# Advanced Non-Line of Sight (ANLOS) Hard Chrome Alternatives



# Advanced Non-Line of Sight (ANLOS) Hard Chrome Alternatives



- Non-chrome, non-nickel containing coatings
- Processes should fit ALC production environment
- Available and emerging technologies considered
  - Nano structured and nano material coatings are included
- Technical approach similar to the NLOS project
- Alternatives identification phase and initial down-selection completed
- STATUS: Preparation of Specimen For Screening Testing Is Underway



# Advanced Non-Line of Sight (ANLOS) Hard Chrome Alternatives



- Integran Technologies electroplated Co-P\*
  - Surface Technology electroless Co-P w/ diamond\*\*
  - Surface Technology electroless Co-B w/ diamond\*\*
  - Zinex Corp. NR 2000 electroplated Co-W
  - Whyco Technologies electroplated Co-SiC
  - Boeing Corp. electroplated Co-P
- \* Nano structured as deposited
- \*\* Could be deposited as an amorphous microstructure and converted to Nano-structure by heating



# Alternative Spray Process Coatings Evaluation



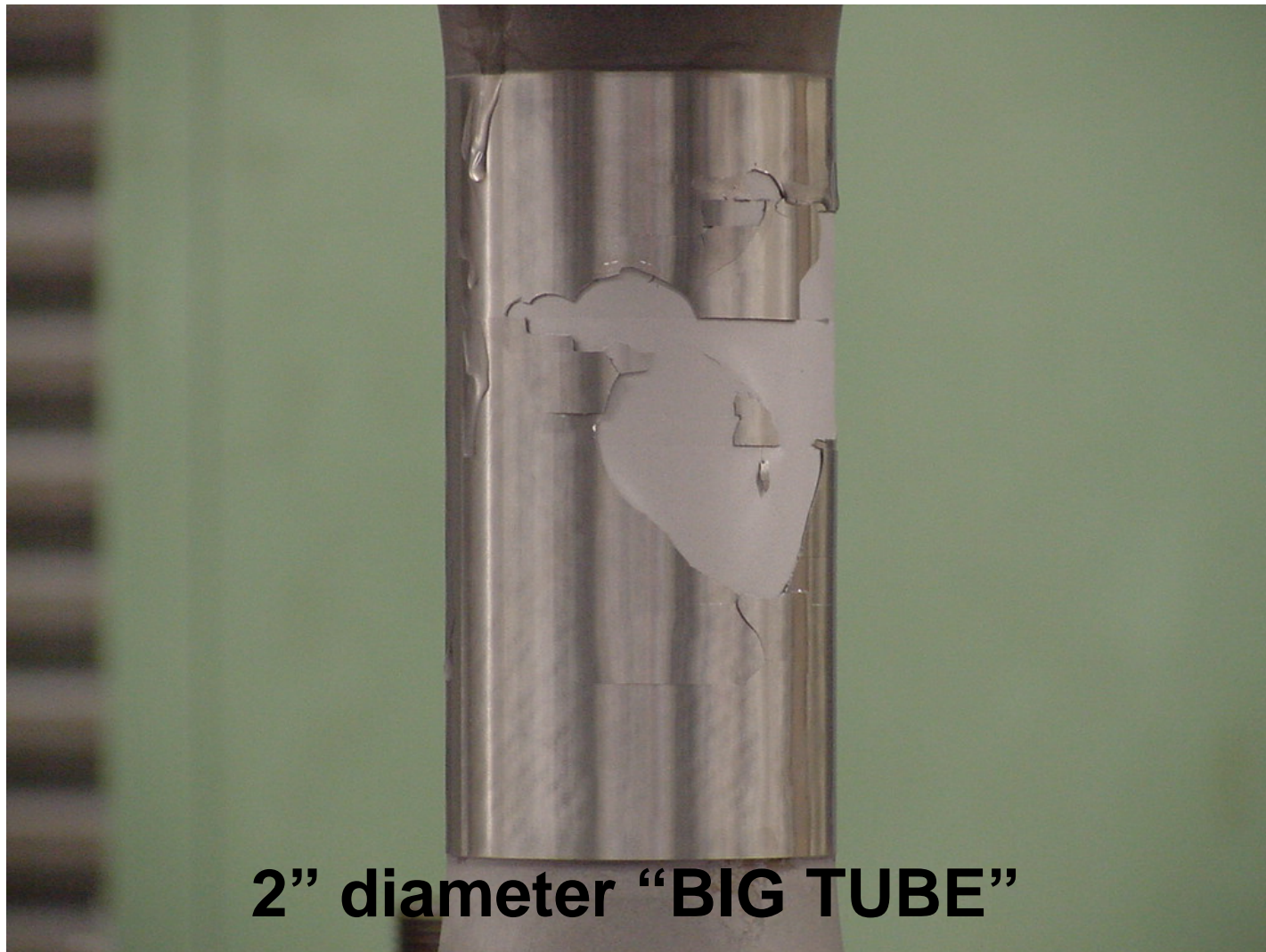
# Alternative Spray Process Coatings Evaluation



- HVOF WC-17Co coatings will crack, spall and delaminate when the 4340, Rc 53, High Strength Steel substrate is stressed greater than 180 ksi
- This “coating integrity” issue could limit the use of HVOF WC-17Co coatings for high stress applications
- Spray processes that operate at a lower temperature than HVOF may provide a more ductile WC-17Co coating



**.005" thick HVOF WC-17Co coating  
180 ksi @ R=-.33**



**2" diameter "BIG TUBE"  
After only 5 cycles**



# Alternative Spray Process Coatings Evaluation



- Previously conducted a limited evaluation of WC-17Co coatings applied by 5 alternative processes
- Only two coating processes showed promise
  - **Activated Combustion – High Velocity Air Fuel (AC-HVAF) developed by Unique Coat Technologies, Corp.**
    - Particles preheated and sprayed below their melting point
  - **Kinetic Metallization (KM) developed by Inovati, Corp.**
    - Particles sprayed at room temperature
    - Helium used for propellant

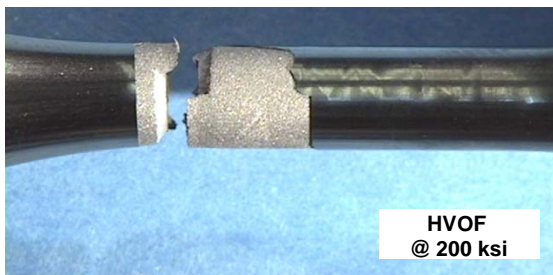
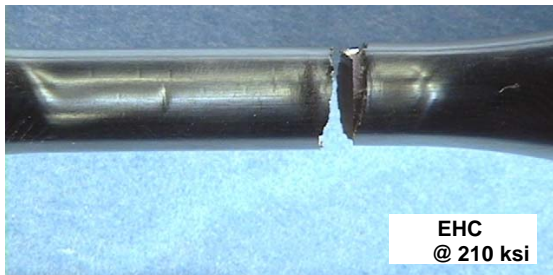


# Alternative Spray Process Coatings Evaluation



## Results of Limited Coating Integrity Evaluation

Unique Coat Technologies AC-HVAF coated specimen after fatigue testing  
(.1 stress ratio, 25 Hz)



AC-HVAF @ 185 ksi



AC-HVAF @ 220 ksi

**3 mil thick WC-17Co coating on 4340 steel**



# Alternative Spray Process Coatings Evaluation



WC-17Co applied by  
**Unique Coat (AC-HVAF) and Inovati (KM)**  
compared to  
**HVOF WC-17Co** applied by OO-ALC

- Coating Integrity Fatigue Testing W/ Big Tubes
- B117 Corrosion Testing
- Metallurgical Analysis



# Alternative Spray Process Coatings Evaluation



- **“BIG TUBE” Coating Integrity Fatigue Testing**
  - 2” diameter, 4340,  $R_c$  53, smooth gage specimen
  - 4” long coating patch, ground to .003” and .010”
  - Stress Ratio (R) = -.33 & -1 @ .125 Hz
  - Start @ 140 ksi, increase 10 ksi every 20 cycles
  - Photo will be taken every cycle; NDI as required
  - Acoustic emission used to determine acoustic event onset, frequency, magnitude and stress state of the specimen during these events



# Alternative Spray Process Coatings Evaluation



- **B117 Corrosion Testing**
  - 1” diameter X 6” long 4340, R<sub>c</sub> 53 Bars
  - 5” long coating patch, ground to .003” and .010”
  - Test for 1000 hrs
  - Photographs & visual evaluation at least every 100 hrs
  
- **Metallurgical Analysis**
  - Carbide distribution must be uniform
  - Interface contamination not > 10%
  - Cracks or delaminates should not exist
  - Voids/oxides not > 1%, no voids > .002”
  - Unmelted particles are not acceptable

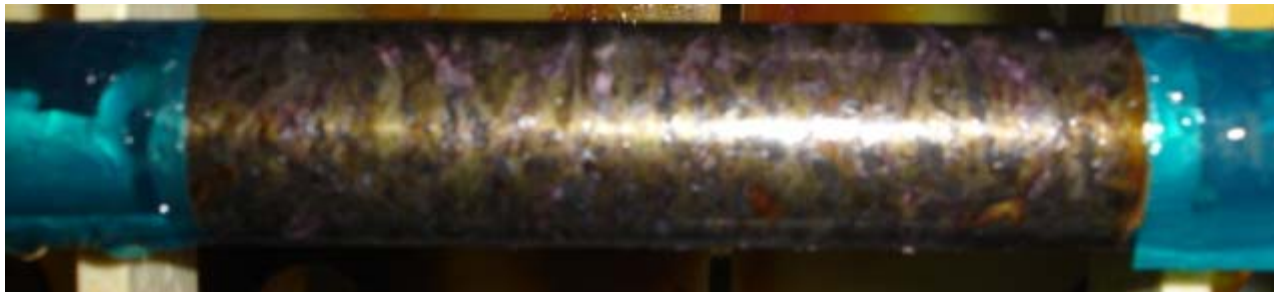


# Alternative Spray Process Coatings Evaluation



## 1000hrs of Corrosion Testing

- No appreciable difference in performance between KM, AC-HVAF and HVOF WC-17Co coatings





# Alternative Spray Process Coatings Evaluation



## Preliminary Integrity Test Data

Type	Thickness	R	Accoustic Emission	Max Stress (ksi)	Remarks
OO-ALC HVOF (2)	.003"	-1	Events began at 150 ksi and continued for every cycle above 160 ksi	220	cracked / spalled at end of coated region @ 170 ksi. Remainder of coating intact
				220	coating intact
HVOF (3)	.010"	-1	Events began with initial cycling and continued to coating failure	160	small spalled area that became large by 180 ksi
				160	coating cracked / spalled
				180	coating cracked / spalled
UniqueCoat AC-HVAF	.003"	-1	Few events with initial cycling and very few afterwards	220	coating intact
AC-HVAF (3)	.010"	-1	Few events with initial cycling and very few until coating failure or test completion	200	coating cracked / spalled
				220	coating intact
				220	coating intact
Inovati KM	.003"	-1	Multiple events with initial cycling and continued to test completion	220	coating cracked / spalled at ends of coated region @ 160 ksi. remainder of coating intact
KM (2)	.010"	-1	Multiple events with initial cycling	160	immediate cracking / spalling (started @ 160 ksi)
				140	immediate cracking / spalling
EHC	.003"	-1	Singular event upon initial cycling, No events until the big bang	220	coating intact specimen failed 16th cycle @ 220 ksi
EHC	.010"	-1	Multiple events starting @ 180 ksi and continuing to test completion	220	coating intact



## Coating Integrity Test Setup



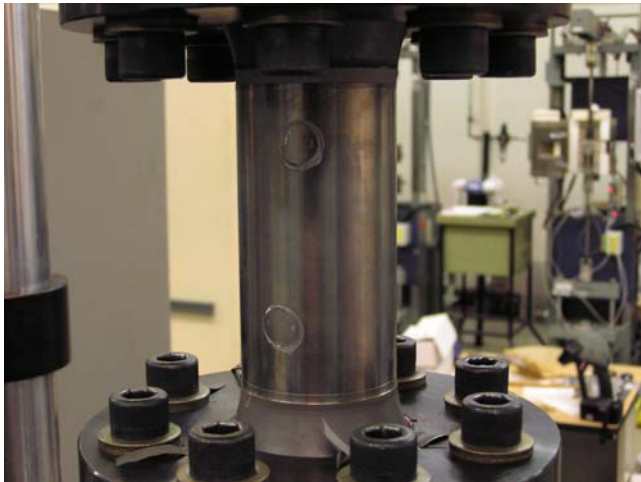


# Alternative Spray Process Coatings Evaluation



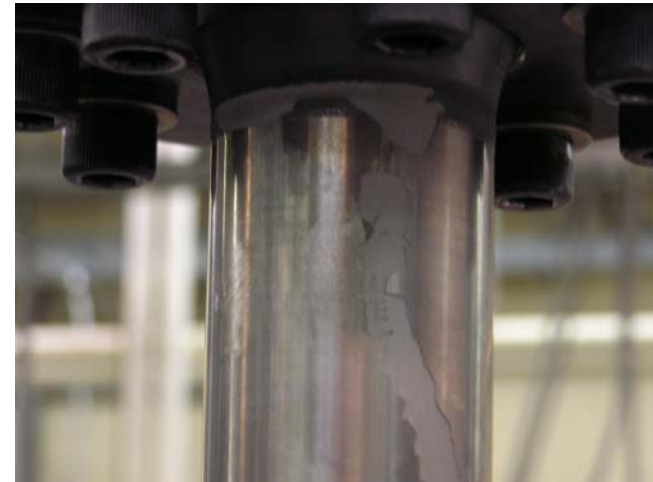
## OO-ALC HVOF

**.003"**



**20 cycles @ 220 ksi**

**.010"**



**4 cycles @ 160 ksi**



# Alternative Spray Process Coatings Evaluation



## UniqueCoat AC-HVAF

**.003"**



**20 cycles @ 220 ksi**

**.010"**



**3 cycles @ 200 ksi**



# Alternative Spray Process Coatings Evaluation



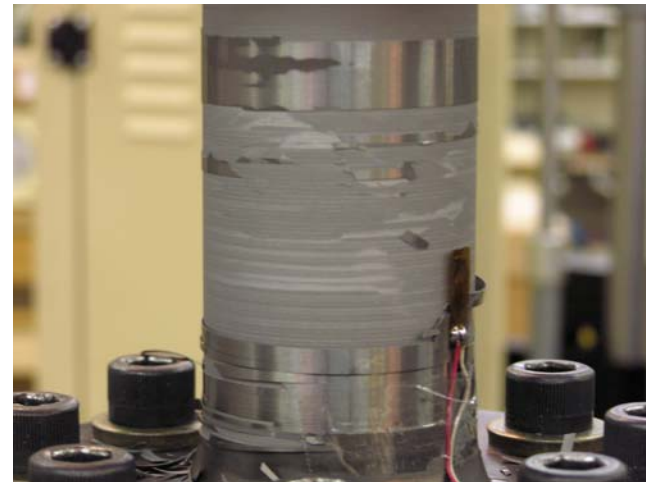
## Inovati KM

**.003"**



**20 cycles @ 220 ksi**

**.010"**



**3 cycles @ 140 ksi**

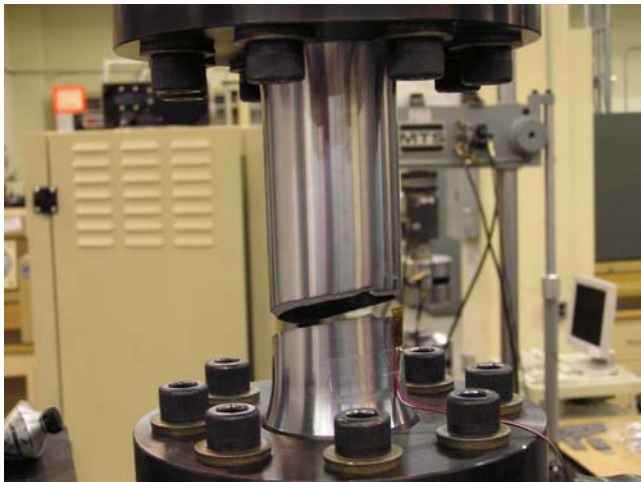


# Alternative Spray Process Coatings Evaluation



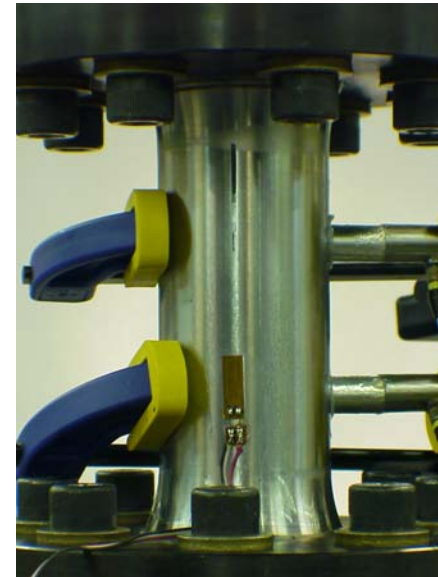
## Hohman EHC

**.003"**



**16 cycles @ 220 ksi**

**.010"**



**20 cycles @ 220 ksi**



# **Metal-Coated Particles**

## **Proof of Concept Evaluation**



# Metal-Coated Particles Evaluation



## **Plating WC particles of HVOF WC-17Co powder, may improve coating ductility and enhance integrity at higher stresses**

- Federal Technology Group nickel plated WC particles as small as 5 microns in diameter
- Cincinnati Thermal Spray is presently coating the specimen using Ogden ALC HVOF spray parameters.
- AFRL/MLSC provided ¼” diameter, 4340 steel, Rc53 fatigue specimen and will evaluate coating microstructure and integrity
- Coating with cobalt preferred, but too costly for this evaluation
- If warranted, a follow-on project is planned to evaluate the integrity of WC-Co coatings with cobalt plated WC particles



# Effects of Chemicals on HVOF Coatings

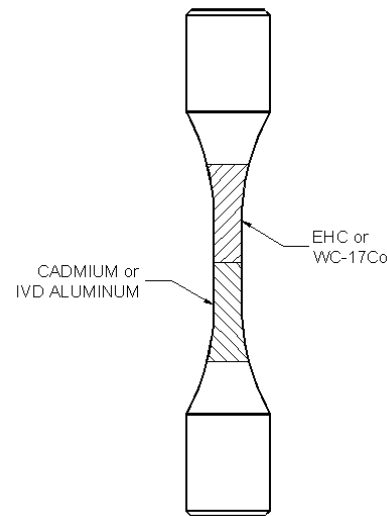
## Follow-up



# Testing Approach



1/4" diameter, 4340 steel, Rc53, smooth gage specimen



Duplex coated specimen

- 1/2 coated with **WC-17Co**, ground to .003"
  - **Other** 1/2 coated with **IVD aluminum** or **cadmium**
- or**
- 1/2 coated with **EHC**, ground to .003"
  - **Other** 1/2 coated with **IVD aluminum** or **cadmium**



# Testing Approach



- Specimen exposed to chemicals while subjected to a constant stress of 180 ksi
- Specimen wetted with chemical every 24 hrs
- 150 hrs without failure was considered passing
- Testing stopped after 196 hours



# Testing Approach



Test Set-up

- **6 chemicals**

- Type III cleaner
- Type IV cleaner

- Aircraft deicer
- Runway deicer

- Paint stripper
- Decontaminant



# Testing Results



- **100%** of **55** EHC / IVD alum specimen passed
- **96%** of **28** EHC / cadmium specimen passed
- **92%** of **12** uncoated specimen passed
- **50%** of **48** WC-17Co / IVD alum specimen passed
- **19%** of **16** WC-17Co / cadmium specimen passed
  
- WC-17Co coatings cracked when stressed to 180 ksi
- Specimen **failed in the WC-17Co region**



# Follow-on Testing



- WC-17Co / cadmium coated specimen  
Distilled water (pH 5.26): 1 out of 4 passed  
Rain water (pH 6.02): 3 out of 4 passed
- EHC / cadmium coated specimen  
Distilled water: 3 out of 3 passed  
Rain water: 3 out of 4 passed
- STATUS: Project Completed