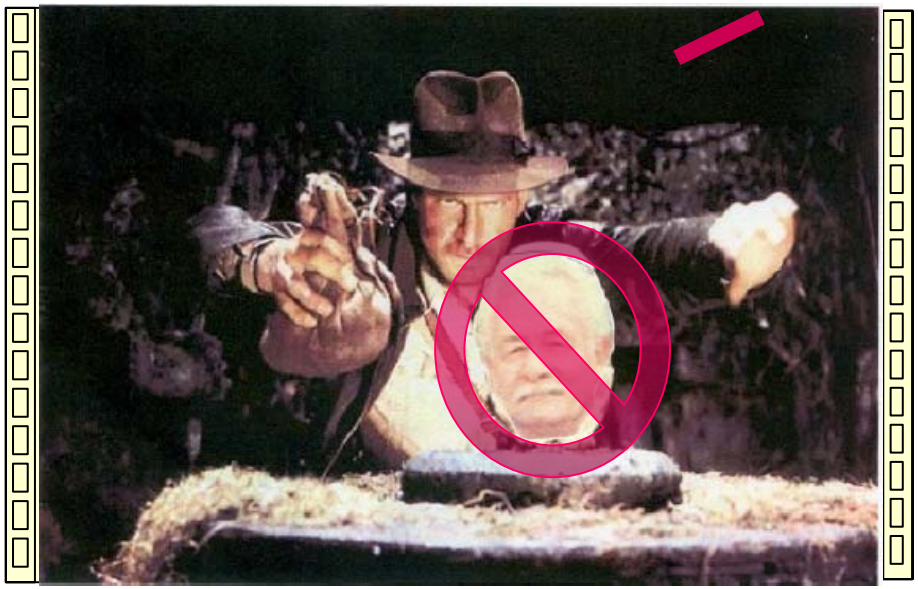
Joe Kolek, Eric Brooman, AFRL/MLSC & Nick Jacobs, AFRL/MLSA

In Search of the Holy Grail of EHC Alternatives (cont'd.)



HCAT Meeting
24-26 January, 2006
San Diego, CA

AFRL / AFMC Quests

- **Non-Line-of-Sight Hard Chrome Alternatives (NLOS)**
  - Completed in July, 2005
  - One Ni-based coating selected as best candidate
- **Advanced Non-Line-of-Sight Hard Chrome and Nickel Alternatives (ANLOS)**
  - Ends in December, 2006
  - Several non-Ni-based coatings being evaluated

Non-Line of Sight (NLOS) Hard Chrome Alternatives

NLOS Hard Chrome Alternatives

- **Down-selected Candidates**
  - **NiPlate 700 by Surface Technology**
    - Electroless Nickel (95%) – P(5%) with SiC particles
  - **UltraCem by Universal Chemical**
    - Electroless Nickel (95%) – B(5%)
      (forms crystalline clusters of nickel boride)
  - **Nanon 9 by Nanon Technologies**
    - Electrolytic Nickel (50-70%) – Cobalt (50-30%)
      (forms nano-crystalline microstructure)
  - **NiCom by US Chrome**
    - Electroplated Nickel with SiC particles

NLOS Hard Chrome Alternatives

- **Final Screening Testing**
  - **Corrosion:** B-117 salt fog test (1,000 hr) and electrochemical testing
  - **Fatigue:** 185 ksi, R = 0.1, 10 cycles/sec until failure
  - **Hydrogen Embrittlement:** ASTM F519 test
  - **Block-on-Ring Wear:** ASTM G77-98 test
  - **Grindability:** Qualitative determination
  - **Strippability:** Qualitative determination

NLOS Hard Chrome Alternatives

- **Final Screening Test Results**

Test/Criterion	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 Not as good as EHC	#4 Significantly Inferior
Fatigue EHC: 5,801 - 7,661 cycles to failure	Pass Fatigue Debit < EHC 42,000 - 93,000 cycles	Pass Fatigue Debit < EHC 7,951 - 13,871 cycles	Fail Fatigue Debit > EHC 1,020 - 1,738 cycles	Pass Fatigue Debit < EHC 19,587 - 124,367 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 Wear 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)
Coefficient of Friction: EHC 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	Most difficult to grind	Acceptable (coating on corrosion panels cracked)	Best surface finish	Acceptable
Strippability (3 mil coating): EHC 1hr with 50% HCl	Acceptable 3 - 4 hr Nistrip R501	Acceptable 3 - 4 hr Nistrip R501	Acceptable 3 - 4 hr Nistrip R501	Acceptable 3 - 4 hr Nistrip R501

NLOS Hard Chrome Alternatives

- **Summary of Screening Test Results**
  - **NiPlate 700: best alternative overall**
    - Hardness and Wear performance comparable to EHC
    - Best adhesion test performance
    - Best electrochemical 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 of NiPlate 700 by the NDCEE/*CTC***
  - **Established coating capability at NDCEE and barrel plated 66 test specimens**
  - Bath temperature 190˚F; deposition rate 0.6 - 0.8 mil/hr
    - NDCEE plating personnel comfortable with all processing activities
  - **NDCEE applied coating evaluated to confirm its properties**
    - Quality; profilometry; thickness; hardness; adhesion; block-on-ring wear; fatigue; hydrogen embrittlement
      - **All properties acceptable**
    - Limited grinding evaluation
      - **Slightly softer coating not difficult to grind**
  - **ALC Dem/Val and implementation plan developed**

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

ANLOS Hard Chrome Alternatives

- **Focus:**
  - **Non-chromium, non-nickel based coatings only**
  - **Processes should fit ALC production environment**
  - **Available and emerging technologies considered**
    - Nano-structured and nano-composite coatings included
  - **Technical approach similar to the NLOS project**
  - **Alternatives identification phase and initial down-selection of candidates completed**

- **Status:**
  - **Specimen preparation and screening testing is underway**
    - Some preliminary test data available at this time

ANLOS Hard Chrome Alternatives

- **Down-selected Candidates**
  - Integran Technologies **electroplated nano Co-P** (limited testing as benchmark)
  - Boeing Corp. **electroplated Co-P*****
    (for comparison with Integran's Co-P coating)
  - Integran **electroplated nano Co-P with boron carbide**
  - Surface Technology **electroless Co-P with diamond***
  - Surface Technology **electroless Co-B with diamond***
  - Zinex Corp. **electroplated Co-W****
  - Whyco Technologies **electroplated Co with SiC*****

* Could be deposited as an amorphous microstructure and converted to a nano-structure by heating.

** Subsequent analysis and discussions with vendor confirmed this was essentially a Co-P type coating with only trace amounts of W present from the bath additives to control properties.

*** Vendor withdrew this coating as not being robust enough yet for commercialization. Replaced with U.S. Chrome electroplated Co-P with SiC particles coating.

ANLOS Hard Chrome Alternatives

- **Phase IIA Testing**
  - **Composition:** various methods (to identify unacceptable constituents)
  - **Quality:** visual inspection per QQ-C-320B
  - **Coverage:** profilometry (measures smoothness, leveling power)
  - **Adhesion:** ASTM B 571 (mandrel and vise bend tests)
  - **Hardness:** HVN determination (with 75 gm indenter)
  - **Wear Resistance:** Taber Wear Index (weight loss per 1,000 cycles over 10,000 cycles)

ANLOS Hard Chrome Alternatives

- **Phase IIB Testing**
  - **Corrosion:** B 117 salt fog test (1,000 hr) and electrochemical (polarization and impedance) testing
  - **Fatigue:** smooth bars, 185 ksi, R = 0.1 (10 cycles/sec until failure)
  - **Hydrogen Embrittlement:** ASTM F519 test
  - **Wear Resistance:** ASTM G77-98 block-on-ring test
  - **Grindability:** qualitative determination
  - **Strippability:** qualitative determination

ANLOS Hard Chrome Alternatives

Details of Tests Conducted to Date

- **Quality**
  - Federal Specification QQ-C-320B
  - Specimens visually inspected after coatings applied: coating must:- cover all specimen surfaces; be free from beads, modules, jagged edges, and other irregularities; be smooth and uniform, dull, matte, or bright as required; contain minimal staining or discoloration
- **Surface Roughness**
  - ANSI B 46.1, *The American National Standard for Surface Texture*
  - Roughness of panels recorded both before and after the plating process by profilometry to determine the leveling power of the plating chemistry
- **Compositional Analysis**
  - ASTM E1508, *Standard Guide for Quantitative Analysis by Energy-Dispersive Spectroscopy*
  - Energy-dispersive x-ray spectroscopy (EDS) in conjunction with a scanning electron microscope (SEM); Rutherford Backscattering Spectrometry (RBS), Glow Discharge Spectrometry (GDS) utilized as needed
  - AFRL will conduct Inductively Coupled Plasma (ICP) - or other analytical techniques, as necessary - to determine elemental concentrations that are not measurable by the above techniques
- **Thickness & Hardness**
  - Averaged thickness on cross-sections in mils (0.001 inch units)
  - Microhardness - Knoop and Vickers Hardness Number, 75 gm indenter

ANLOS Hard Chrome Alternatives

Details of Tests Conducted to Date (cont'd.)

- **Adhesion**
  - ASTM B571 Section 3.1- Mandrel Bend Test
  - ASTM B571 Section 3.2- Vise Bend Test
- **Taber Wear Resistance**
  - ASTM D4060, 1000 gram, CS-10 wheels
  - TWI, weight loss per 1,000 cycles over 10,000 cycles
- **Block-on-Ring Wear Resistance**
  - ASTM G77, *Ranking Resistance of Materials to Sliding Wear Using the Block-on-Ring Wear Test*
  - No lubricant, 25-lb load, 120 revolutions per minute (rpm), 5,400 revolutions
- **Grindability**
  - Diamond or green silicon carbide wheels, 100 -120 grit, 3,600 rpm, 6,494 sfpm, 55 ipm traverse speed, 0.0003 inch depth of cut

ANLOS Hard Chrome Alternatives

Quality Inspection Results

Vendor	Coating Process	PASS/FAIL	Comments
Boeing	**Electroplated Co-P**	**PASS**	Beading along edges and small pits and jagged edges noted on several specimens; small amounts of red rust
Integran	**Electroplated nano Co-P**	**BT**	Currently being tested and evaluated
Zinex	**Electroplated Co-W***	**PASS**	Slight edge build-up, beading, and surface staining noted on many specimens; pitting noted on some specimens
STI	**Electroless Co-B + diamond particles**	**PASS**	Discoloration and watermarks noted on backside of some panels; small amounts of red rust noted
STI	**Electroless Co-P + diamond particles**	**PASS**	Coating appeared to be chipped along the edges of many specimens; staining and watermarks noted on many specimens; small amounts of red rust noted
Integran	**Electroplated nano Co-P + B_4C particles**	**PASS**	Small pitting, jagged edges, and edge staining noted on many specimens; red rust noted on side with ID on many specimens
U.S. Chrome	**Electroplated Co-P + SiC particles**	**PASS**	Some beading noted; numerous stains and fingerprints; small amounts of red rust

ANLOS Hard Chrome Alternatives

Surface Roughness Results

Vendor	Coating Process	Overall Profile Change	
		As-Plated	375°F for 24 hours
Boeing	Electroplated Co-P	Some surface leveling ability	
Integran	Electroplated nano Co-P	Currently being tested	
Zinex	Electroplated Co-W*	Roughening of surface	
STI	Electroless Co-B + diamond particles	Significant roughening of surface	
STI	Electroless Co-P + diamond particles	Roughening of surface	
Integran	Electroplated nano Co-P + B_4C particles	Roughening of surface	
U.S. Chrome	Electroplated Co-P + SiC particles	Roughening of surface	

* When questioned, vendor stated that W was a minor additive and that this was essentially a Co-P coating.

ANLOS Hard Chrome Alternatives

Composition Analytical Results

- **Integran – Electroplated nano-Co-P**
  - Co/P (at%/at%) = 25.3
  - Elemental concentrations: Co - 91 wt%, P - 2 wt%, free carbon - 4 wt%
- **Integran – Electroplated nano-Co-P-B_4C**
  - Coated test specimens not yet received from the vendor
- **Zinex – Electroplated Co-W***
  - Co/P (at%/at%) = 5.0
  - Elemental concentrations: Co - 84 wt%, P - 8 wt%, free carbon - 5 wt%, W - none detected*
- **STI – Electroless Co-B + diamond particles**
  - Concentration of boron not discernible via EDS, RBS, or GDS – additional analysis (e.g., ICP) is required
  - Average particle (diamond) size: 0.48 microns (480 nm)
- **STI – Electroless Co-P + diamond particles**
  - Co/P (at%/at%) = 9.3
  - Elemental concentrations: Co – 52 wt%, P – 2 wt%, Diamond + Free Carbon – 45 wt%
  - Average particle (diamond) size: 0.58 microns (580 nm)
- **U.S. Chrome – Electroplated Co-P + SiC particles**
  - Coated test specimens not yet received from the vendor

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* When questioned, vendor stated that W was a minor additive and that this was essentially a Co-P coating.

ANLOS Hard Chrome Alternatives

Thickness and Hardness Results

Vendor	Coating Process	Thickness inch	Hardness, Ave.			
			HK		HV	
			As-Plated	375°F for 24 hours	As-Plated	375°F for 24 hours
Boeing	Electroplated Co-P	0.004	511	614	669	625
Zinex	Electroplated Co-W*	0.003	461	576	659	703
STI	Electroless Co-B + diamond particles	0.003	442	640	601	682
STI	Electroless Co-P + diamond particles	0.004	666	981	727	822
Integran	Electroplated nano Co-P + B_4C particles	0.006	709	869	675	657
U.S. Chrome	Electroplated Co-P + SiC particles	0.005	624	737	625	713

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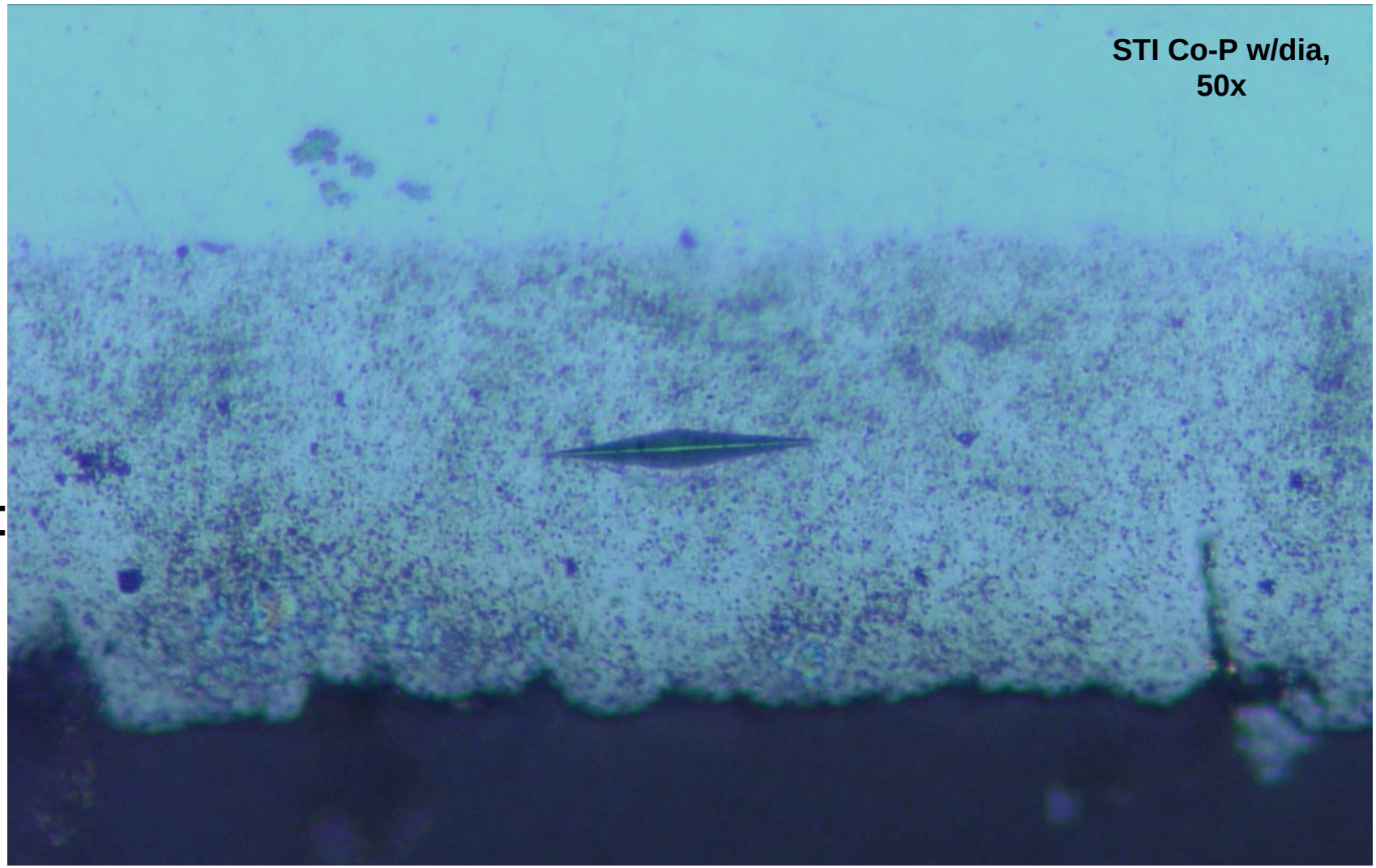
ANLOS Hard Chrome Alternatives



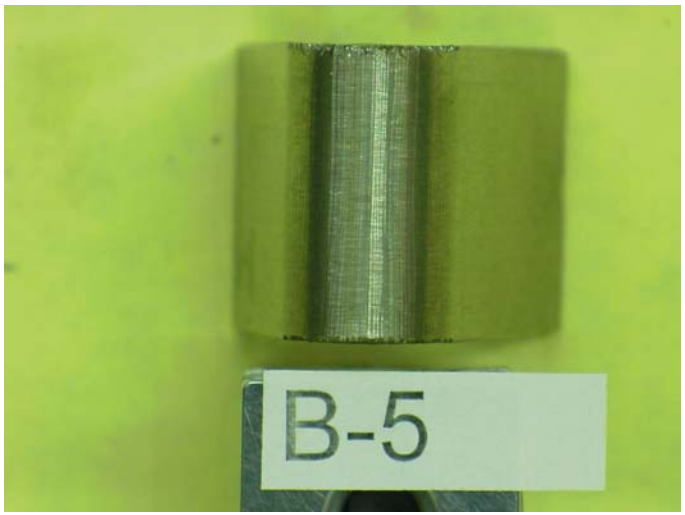
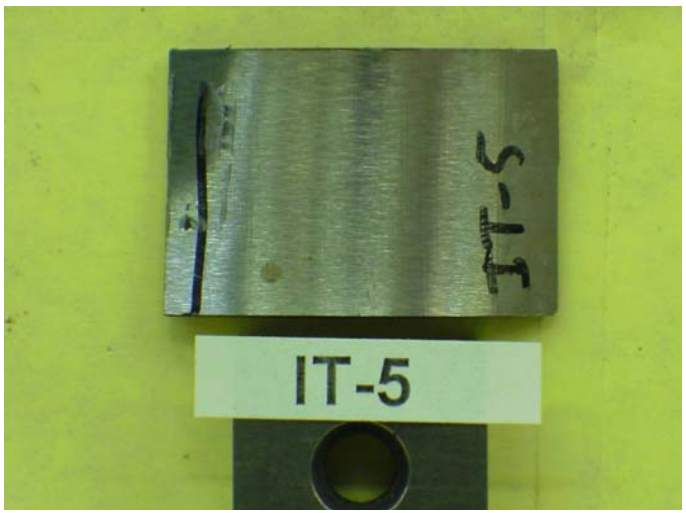
ANLOS Hard Chrome Alternatives

Adhesion Test Results

Vendor	Coating Process	Surface Condition	H2 Relief Baking	Adhesion		
				3.1 Mandrel	3.2 Vise	Overall
Boeing	Electroplated Co-P	As-plated	As-plated	Pass	Pass	**Pass**
		Ground	As-plated	Pass	Pass	**Pass**
		As-plated	375 F/24 hr	Pass	Marg. Pass	**Pass**
		Ground	375 F/24 hr	Pass	Pass	**Pass**
Zinex	Electroplated Co-W*	As-plated	As-plated	Fail	Marginal	**Fail**
		Ground	As-plated	Pass	Pass	**Pass**
		As-plated	375 F/24 hr	Fail	Marginal	**Fail**
		Ground	375 F/24 hr	Pass	Pass	**Pass**
STI	Electroless Co-B + diamond particles	As-plated	As-plated	Pass	Pass	**Pass**
		Ground	As-plated	Pass	Pass	**Pass**
		As-plated	375 F/24 hr	Pass	Pass	**Pass**
		Ground	375 F/24 hr	Pass	Pass	**Pass**
STI	Electroless Co-P + diamond particles	As-plated	As-plated	Pass	Marginal	**Marg. Pass**
		Ground	As-plated	Pass	Pass	**Pass**
		As-plated	375 F/24 hr	Pass	Fail	**Marg. Pass**
		Ground	375 F/24 hr	Pass	Pass	**Pass**
Integran	Electroplated nano Co-P + B_4C particles	As-plated	As-plated	Pass	Pass	**Pass**
		Ground	As-plated	Pass	Pass	**Pass**
		As-plated	375 F/24 hr	Pass	Pass	**Pass**
		Ground	375 F/24 hr	Pass	Pass	**Pass**
U.S. Chrome	Electroplated Co-P + SiC particles	As-plated	As-plated	Fail	Fail	**Fail**
		Ground	As-plated	- -	- -	**- -**
		As-plated	375 F/24 hr	Fail	Fail	**Fail**
		Ground	375 F/24 hr	Pass	Pass	**Pass**

***** When questioned, vendor stated that W was a minor additive and that this was essentially a Co-P coating.

ANLOS Hard Chrome Alternatives

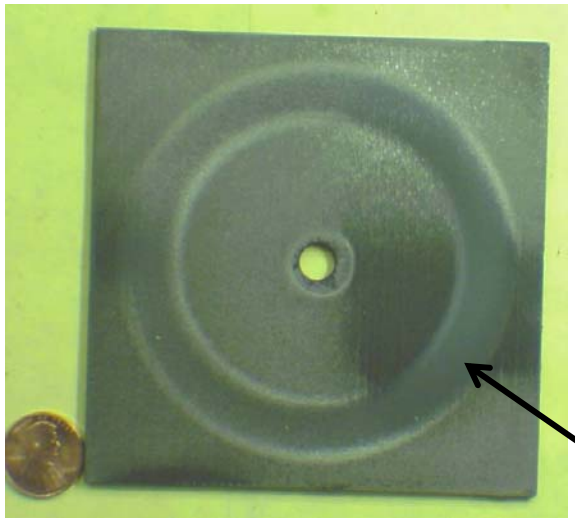
Pass		Fail
	**B571 3.1** **Mandrel Bend**	
	**B571 3.2** **Vise Bend**	

ANLOS Hard Chrome Alternatives

Taber Wear Index Results

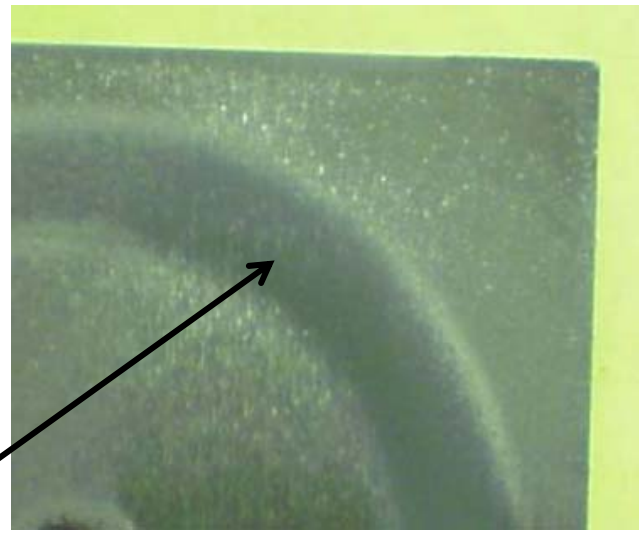
Vendor	Coating	Surface Condition	H2 Relief Baking	TWI Average (last 9,000 cycles)
Integran	Electroplated Nano Co-P	As-plated	As-plated	BT
		Ground	As-plated	BT
		As-plated	375 F/24 hr	**18.2**
		Ground	375 F/24 hr	BT
Boeing	Electroplated Co-P	As-plated	As-plated	**18.8**
		Ground	As-plated	**23.0**
		As-plated	375 F/24 hr	**18.2**
		Ground	375 F/24 hr	**18.5**
Zinex	Electroplated Co-W*	As-plated	As-plated	**20.3**
		Ground	As-plated	**21.2**
		As-plated	375 F/24 hr	**20.5**
		Ground	375 F/24 hr	**20.7**
STI	Electroless Co-B + diamond particles	As-plated	As-plated	**15.5**
		Ground	As-plated	**11.8**
		As-plated	375 F/24 hr	**8.2**
		Ground	375 F/24 hr	**2.1**
STI	Electroless Co-P + diamond particles	As-plated	As-plated	**3.9**
		Ground	As-plated	**3.9**
		As-plated	375 F/24 hr	**3.1**
		Ground	375 F/24 hr	**2.9**
Integran	Electroplated nano Co-P + B_4C particles	As-plated	As-plated	**4.8**
		Ground	As-plated	**5.1**
		As-plated	375 F/24 hr	**4.6**
		Ground	375 F/24 hr	**5.7**
U.S. Chrome	Electroplated Co-P + SiC particles	As-plated	As-plated	**5.4**
		Ground	As-plated	**15.2**
		As-plated	375 F/24 hr	**3.5**
		Ground	375 F/24 hr	**8.2**

ANLOS Hard Chrome Alternatives

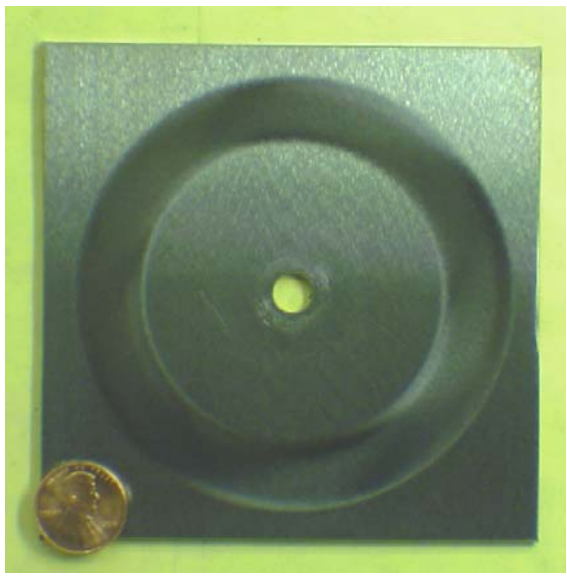


STI Co-P + diamond particles

TWI = 3

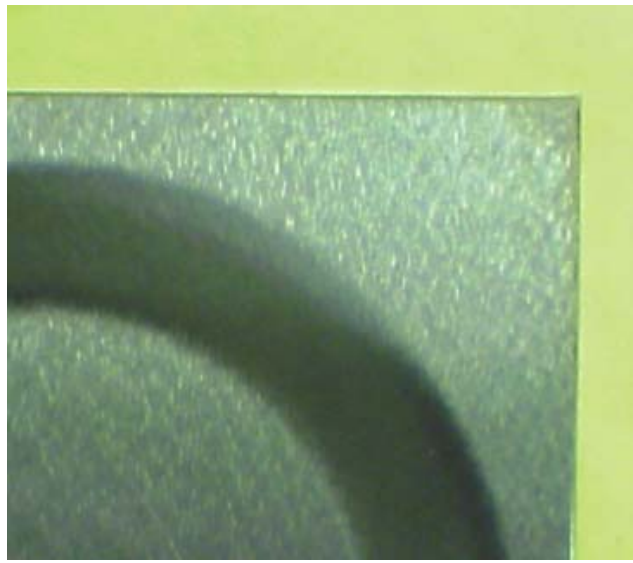


Wear Tracks



Zinex Co-W*

TWI = 20



ANLOS Hard Chrome Alternatives

Block on Ring Wear Results

Vendor	Coating Process	Coating Ground	Average Coefficient of Friction		Average Depth of Block Scar (mils)	
			As-Plated	375°F for 24 hours	As-Plated	375°F for 24 hours
Integran	Electroplated nano Co-P	Yes	Currently being tested			
		No		0.39		2.91
Zinex	Electroplated Co-W*	Currently being tested				
STI	Electroless Co-B + diamond particles	Currently being tested				
STI	Electroless Co-P + diamond particles	Yes	0.54	0.52	3.87	3.76
		No	0.52	0.57	2.55	2.82
Integran	Electroplated nano Co-P + B_4C particles	Coated test specimens not yet received from vendor				
U.S. Chrome	Electroplated Co-P + SiC particles	Coated test specimens not yet received from vendor				

* When questioned, vendor stated that W was a minor additive and that this was essentially a Co-P coating.

ANLOS Hard Chrome Alternatives

- **Grindability Results**
  - All coatings grindable via conventional techniques
  - Specific best practices not identified
  - Surface finish and material removal rates not optimized

	ANLOS Grinding Summary - Best Resulting Finish and Grindability					
Grind Parameter	Boeing B-10	US-Chrome US-4	STI ST-1	Zinex ZC-5	STI STB-8	Integran IT-12
Wheel Type	diamond	diamond	diamond	green SiC	diamond	diamond
Wheel Grit	100	100	100	120	100	100
Wheel Hardness	N	N	N	L	N	N
Wheel Diameter (in)	7	7	7	6.4	7	7
Wheel RPM	3600	3600	3600	3600	3600	3600
Wheel SFPM	6494	6494	6434	6029	6494	6494
Depth of Cut (in)	0.0003	0.0003	0.0003	0.0003	0.0003	0.0003
Table Traverse speed (IPM)	55	55	55	55	55	55
Final Surface Finish (Ra)						
Longitudinal	**15**	**19**	**24**	**7**	**30**	**35**
Transverse	**17**	**23**	**30**	**8**	**42**	**34**

ANLOS Hard Chrome Alternatives

- **Summary of Preliminary Findings**

Coating	Quality	Roughness	Thickness	Hardness	Adhesion	Abrasive Wear		Coeff. of Friction	Grindability
						TWI	BOR		
Electroplated Co-P	Pass	Some leveling	Fair	Softer than EHC	Pass	Fail	- -	- -	Marginal
Electroplated nano Co-P	BT*	BT	BT	BT	BT	Fail	Marginal	Pass	- -
Electroplated Co-W**	Pass	Rougher	Fair	Softer than EHC	Marginal pass	Fail	BT	BT	Pass
Electroless Co-B + diamond particles	Pass	Much rougher	Fair	Softer than EHC	Pass	Marginal pass	BT	BT	Marginal
Electroless Co-P + diamond particles	Pass	Rougher	Fair	Close to EHC	Marginal pass	Pass	Marginal	Pass	Marginal
Electroplated Co-P + B_4C particles	Pass	Rougher	Good	Softer than EHC	Pass	Marginal pass	BT	BT	Marginal
Electroplated Co-P + SiC particles	Pass	Rougher	Good	Softer than EHC	Fail	Marginal pass	BT	BT	Marginal

* BT = Yet to be tested.

**When questioned, vendor stated that W was a minor additive and that this was essentially a Co-P coating.

ANLOS Hard Chrome Alternatives

- **Acknowledgments**
  - AFRL/MLSC
    - Joe Kolek*, Nick Jacobs (NLOS & ANLOS)
    - Eric Brooman (ANLOS)
  - Concurrent Technologies Corporation
    - Milissa Pavlik (NLOS & ANLOS)
    - Brad Biagini (NLOS & ANLOS)
    - Melissa Klingenberg (ANLOS)

* Effective 1 April, 2006 Eric Brooman will execute the ANLOS project in place of Joe Kolek.