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Cost and Performance Report

(WP-200936)

Electrodeposited Nanocrystalline Co-P Alloy Coatings as a Hard Chrome Alternative

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14. ABSTRACT
The replacement of hard chrome plating in aircraft manufacturing activities and maintenance depots is a high priority for the U.S. Department of Defense. Chromium plating baths contain chromic acid, in which the chromium is present in the hexavalent state (Cr^{+6}), a known carcinogen having a high level of toxicity. Nanocrystalline cobalt-phosphorus (nCoP) is an environmentally benign alternative to engineering hard chrome plating (EHC). The nCoP coatings are applied to both line-of-sight and non-line-of-sight surfaces using similar electroplating processes. This program aimed to qualify to the technology for use in applications currently specified for EHC. Overall, nCoP met the majority of acceptance criteria as defined in the Joint Test Protocol and showed excellent performance in demonstration and validation field testing. The results showed that nCoP exceeded EHC performance in corrosion and sliding wear tests. The abrasive wear performance of nCoP by Taber wear was poor as previously determined, while gravelometry testing showed equivalent performance relative to hard chrome. Fatigue and hydrogen embrittlement testing showed nCoP met or exceeded performance of EHC. While further testing may be required to support "General" authorization, it is anticipated that nCoP may be widely specified per *MIL-DTL-32502 - "Coating, Cobalt-Phosphorus Alloy, Nanocrystalline (Electrodeposited)"* as a hard chrome alternative on the basis of testing completed to date.

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ACRONYMS AND ABBREVIATIONS

µg	microgram(s)
ACE	Armored Combat Earthmover
AIMD	Aircraft Intermediate Maintenance Department
AMS	Aerospace Material Standards
ASTM	American Society for Testing and Materials
CBA	cost-benefit analysis
CCPE	Corrosion Control and Prevention Executive
COMNAVAIRANT	Commander, Naval Air Force Atlantic
Cr^{6+}	hexavalent chromium
CVN 75	USS Harry S. Truman
Dem/Val	demonstration and validation
DoD	Department of Defense
EHC	engineering hard chrome
EPA	U.S. Environmental Protection Agency
ESTCP	Environmental Security Technology Certification Program
FRCE	Fleet Readiness Center East
FRCSE	Fleet Readiness Center Southeast
FRCSW	Fleet Readiness Center Southwest
HCAT	Hard Chrome Alternatives Team
HCP	hexagonal close-packed
HVOF	high velocity oxygen fuel
Hz	hertz
Integran	Integran Technologies, Inc.
IRR	internal rate of return
JTP	Joint Test Protocol
JTR	Joint Test Report
Ksi	kilopound(s) per square inch
LVS	Logistic Vehicle System
m	meter(s)
mm	millimeter(s)
NAVAIR	Naval Air Systems Command
NAVSEA	Naval Sea Systems Command

NAWCAD	Naval Air Warfare Center Aircraft Division
nCoP	nanocrystalline cobalt-phosphorus
NDI	non-destructive inspection
NESDI	Navy's Environmental Sustainability Development to Integration
NFS	Notch Fracture Strength
Ni	nickel
Nm	Newton-meter
nm	nanometer(s)
NPV	net present value
NSWCCD	Naval Surface Warfare Center, Carderock Division
NSWCPCD	Naval Surface Warfare Center, Panama City Division
OEM	original equipment manufacturer
OSD	Office of the Secretary of Defense
OSHA	Occupational Safety and Health Administration
PAX	Patuxent River
PEL	permissible exposure limit(s)
PEO	Program Executive Office
PPA	Production Plant Albany
psi	pound(s) per square inch
ROI	return on investment
S-N	stress versus cycles-to-failure (related to fatigue data)
SERDP	Strategic Environmental Research and Development Program
SRC	Scheduled Removal Component
T45TS	T-45 Training System
USMC	U.S. Marine Corps
VHN	Vickers Hardness Number
WC	Tungsten Carbide

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EXECUTIVE SUMMARY

OBJECTIVES OF THE DEMONSTRATION

The replacement of hard chrome plating in aircraft manufacturing activities and maintenance depots is a high priority for the U.S. Department of Defense (DoD). Chromium plating baths contain chromic acid, in which the chromium is present in the hexavalent state (Cr^{+6}), a known carcinogen having a high level of toxicity. During operation, chrome plating tanks emit a Cr^{+6} mist into the air, which must be ducted away and removed by scrubbers or mist eliminators. Wastes generated from plating operations must be disposed of as hazardous waste and plating operations must abide by U.S. Environmental Protection Agency (EPA) emissions standards and Occupational Safety and Health Administration (OSHA) permissible exposure limits (PEL).

TECHNOLOGY DESCRIPTION

Nanocrystalline cobalt-phosphorus (nCoP) is an environmentally benign alternative to engineering hard chrome (EHC) plating. The nCoP coatings are applied to both line-of-sight and non-line-of-sight surfaces using similar electroplating processes. Previous Strategic Environmental Research and Development Program (SERDP) and Environmental Security Technology Certification Program (ESTCP) projects demonstrated nCoP possessed excellent corrosion and sliding wear resistance. The process was demonstrated to readily integrate within existing electroplating infrastructure present in DoD repair and overhaul facilities. While the nCoP technology has been in use for industrial applications for several years, its use for aerospace applications necessitates further investigation prior to implementation for DoD manufacturing repair and overhaul.

DEMONSTRATION RESULTS

This report presents the results of the demonstration and validation (Dem/Val) program to evaluate nCoP, which included extensive material characterization and testing. The primary objective of the work was to evaluate coatings through coupon testing, functional rig testing, and field testing selected components on relevant platforms. The nCoP process was operated primarily at Naval Air Systems Command (NAVAIR) Fleet Readiness Center Southeast (FRCSE) in Jacksonville, FL to prepare test coupons and for manufacturing of Dem/Val components.

A Joint Test Protocol (JTP) was devised by stakeholders to validate nCoP coatings for use in relevant applications such as landing gear, arresting gear, hydraulic cylinders, actuators, and dimensional restoration of damaged components. The stakeholders included aerospace original equipment manufacturers (OEM; Pratt & Whitney Canada, Heroux-Devtek, Messier-Bugatti-Dowty, and Boeing), the DoD maintenance depots (FRCSE, NAVAIR Fleet Readiness Center East [FRCE]), DoD engineering authority (NAVAIR Patuxent River [PAX], NAVAIR Lakehurst), and Naval Sea Systems Command (NAVSEA; through leveraged support funded by Navy's Environmental Sustainability Development to Integration [NESDI]), as well as the Integran Technologies, Inc. and Rowan Technology Group.

Performance testing conducted per the JTP included: coating quality, adhesion, fatigue, corrosion, hydrogen embrittlement, fluid compatibility, wear, and impact testing. The test matrix aimed to provide engineers and scientists with sufficient information for evaluation of the coating as a suitable alternative for hard chrome. The results showed that nCoP exceeded EHC performance in corrosion and sliding wear tests. The abrasive wear performance of nCoP by Taber wear was less than EHC, as previously determined, while gravelometry testing showed equivalent performance relative to hard chrome. Fatigue and hydrogen embrittlement testing showed nCoP met or exceeded performance of EHC.

Naval Surface Warfare Center, Carderock Division (NSWCCD)/NAVSEA, with leveraged support from NESDI, conducted an evaluation of nCoP and other hard chrome replacement candidate coatings for use on hydraulic cylinders for U.S. Marine Corps (USMC) vehicles. The testing demonstrated nCoP performed equivalent to EHC in adhesion, impact resistance, and wear resistance testing, and significantly outperformed EHC in corrosion testing. A hydraulic cycling test resulted in nCoP samples lasting over three times longer than any other candidate coating including EHC. On the basis of these results, NAVSEA conducted a Dem/Val on a hydraulic actuator on the M9 Armored Combat Earthmover (ACE) at Panama City, Florida.

Successful field testing on demonstration components coated with nCoP were completed:

- An arresting tail hook pivot assembly was installed with nCoP applied to the cam surface on the T-45 Goshawk aircraft. Following the most recent update from T-45 engineering, the nCoP component reached 116 arrestments showing no significant signs of wear (>825 ± 15 flight hours). Component level approval is pending review of Joint Test Report (JTR) results.
- A lifting arm pin was installed with nCoP on-board USS Harry S. Truman (CVN 75) on the A/S32A-32 Aircraft Towing Tractor also known as Spotting Dolly. Over the duration of the demonstration, 672 aircraft movements were completed. A non-destructive inspection (NDI) was completed successfully following 91 days. A fleet saving of approximately 2.5 man-hours/pin to clean and prepare for NDI inspection was observed. The nCoP lifting arm pins showed improved performance over the incumbent plating configuration applied pins (i.e., Cadmium plated). An approval memo was written by the field activity.
- A hydraulic cylinder was installed with nCoP on M9 ACE located at Panama City. The component was assembled and pressured tested at the USMC Depot in Albany, GA prior to field testing. Field testing was ended following only two months due to operational need for the vehicle. No signs of damage were found following inspection.

A cost benefit analysis was performed to determine the expected payback period if nCoP replaces the current FRCSE workload. A payback period of 4.7 years was determined. A substantial improvement in payback is expected by increasing workload as a result of the increased throughput obtained due to the high plating rate and lower energy consumption. Furthermore, a reduced plating shop infrastructure is possible to support an equivalent workload of EHC plating and elimination of hazardous materials (e.g., chromic acid volume reduction, lead anodes, etc.)

IMPLEMENTATION ISSUES

Further work is required to address implementation issues in order to facilitate widespread adoption of the nCoP technology, including: identification of appropriate masking materials that can survive the elevated operating temperature of the process; modification of the process to allow use of conventional direct current rectifiers; improvement of Taber wear performance; and additional characterization data on fatigue performance to study the variability present in the data set.

Overall, nCoP met the majority of acceptance criteria, as defined in the JTP, and showed excellent performance in Dem/Val field testing. While further testing may be required to support “General” authorization, it is anticipated that nCoP may be widely specified per *MIL-DTL-32502, “Coating, Cobalt-Phosphorus Alloy, Nanocrystalline (Electrodeposited)”*, as a hard chrome alternative on the basis of testing completed to date.

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1.0 INTRODUCTION

1.1 BACKGROUND

The replacement of engineering hard chrome (EHC) plating in aircraft manufacturing activities and maintenance depots is a high priority for the U.S. Department of Defense (DoD). Hard chrome plating is a technique that has been in commercial production for over 50 years and is a critical process that is used both for applying hard coatings to a variety of aircraft components in manufacturing operations and for general re-build of worn or corroded components that have been removed from aircraft during overhaul. Chromium plating baths contain chromic acid, in which the chromium is in the hexavalent state(Cr^{6+}), which is a known carcinogen with a level of toxicity greater than arsenic or cadmium. During operation, chrome plating tanks emit a Cr^{6+} mist into the air, which must be ducted away and removed by scrubbers. Wastes generated from plating operations must be disposed of as hazardous waste and plating operations must abide by U.S. Environmental Protection Agency (EPA) emissions standards and Occupational Safety and Health Administration (OSHA) permissible exposure limits (PEL).

Based on the projections of the metal finishing industry and the study conducted by Naval Sea Systems Command (NAVSEA) in 1995, it was clear that a reduction of the Cr^{6+} PEL would greatly increase the cost and processing times associated with EHC plating within DoD. As discussed in Section 1.3, OSHA issued a new Cr^{6+} PEL standard of 5 micrograms per meter cubed ($\mu g/m^3$) on 28 February 2006. The actual costs for compliance for Naval Air Systems Command (NAVAIR) Fleet Readiness Center Southeast (FRCSE) include an initial capital cost of $1,454,749 with annual costs of $1,153,697 per year.

1.2 OBJECTIVE OF THE DEMONSTRATION

The goals of this project were to demonstrate and validate (Dem/Val) electrodeposited nanocrystalline cobalt-phosphorus (nCoP) alloy coatings as a technically feasible and commercially viable replacement for EHC on multiple military systems. The nCoP will be viewed as part of an overall strategy to replace currently utilized EHC electroplating processes on all geometries and minimize environmental and worker safety issues associated with EHC electroplating, while significantly improving performance and reducing life-cycle costs. This technology has demonstrated pollution and cost/waste reduction from actual depot operations performed by maintenance personnel. There are several task areas within the project which will be addressed to achieve the above goals:

- Establishing a large-scale electroplating process line at the FRCSE that is capable of processing selected demonstration components and other workloads currently overhauled at the facility;
- Reducing environmental impacts by minimizing hazardous materials, Cr^{6+} and hazardous waste generation;
- Performing a technology transition cost/benefit analysis and waste reduction assessment of nCoP technology from actual depot maintenance operations;
- Validating field performance of nCoP electrodeposits applied to air and ground vehicle demonstration components; and

- Developing standards, specifications and technical documentation providing procedures and process controls for application and removal of nCoP electroplated coatings

1.3 REGULATORY DRIVERS

On 9 April 2009, a memo from the Office of the Secretary of Defense (OSD) stated that they would more aggressively mitigate the unique risks to DoD operations now posed by Cr^{6+} as a result of increased international and national restrictions. The memo instructs DoD Military Departments to restrict the use of Cr^{6+} unless no cost-effective alternatives are identified. Furthermore, this would force adoption of Cr^{6+}-free coatings and production methods unless otherwise approved directly by Program Executive Office (PEO) or equivalent level, in coordination with the Military Department's Corrosion Control and Prevention Executive (CCPE), to certify there is no acceptable alternative to the use of Cr^{6+} on a new system.

2.0 DEMONSTRATION TECHNOLOGY

2.1 TECHNOLOGY DESCRIPTION

Electrodeposited nanocrystalline materials have advanced rapidly to commercial application as a result of: (1) an established industrial infrastructure (i.e., electroplating and electroforming industries); (2) a relatively low cost of application whereby nanocrystalline materials can be produced by simple modification of bath chemistries and electrical parameters used in current plating and electroforming operations; (3) the capability in a single-step process to produce metals, alloys and metal-matrix composites in various forms (i.e., coatings and free-standing complex shapes); and (4) the ability to produce fully dense nanostructures, free of extraneous porosity. The fully dense nanocrystalline materials have displayed predictable material properties based upon their increased content of intercrystalline defects. This "predictability" in ultimate material performance has accelerated the adoption of these materials by industry, whereby such extreme grain refinement simply represents another metallurgical tool for microstructural optimization.

Numerous practical applications for nanocrystalline materials are based upon opportunities for high-strength coatings and freestanding structural components. The superior mechanical properties led to one of the first large scale industrial application of nanomaterials for in-situ repair of nuclear steam generator tubing using Electrosleeve® technology [i]. In this application, a nanocrystalline nickel (Ni)-phosphorous microalloy with a grain size of approximately 100 nanometers (nm) is electrodeposited on the inside surface of steam generator tubes to a thickness that ranges from 0.5 to 1 millimeter (mm) to structurally repair those sites in the tubes where the original structural integrity has been compromised. This technology has met all expectations to date and has led to its continued use in the nuclear power industry today. Several members of the technical materials team at Ontario Hydro Technologies, Inc., where this technology was developed, later formed Integran Technologies, Inc. (Integran).

Integran has successfully developed Nanovate™ products for a variety of commercial applications. The Nanovate product platform refers to materials with a nanostructured grain size built from a "bottom-up" approach, synthesized by electrodeposition techniques. Nanovate alloys achieve high strength, toughness, and hardness by reducing grain size to nm scale. They are applied as fully dense coatings or freestanding forms.

Nanovate CoP is the commercial trade name for the nCoP hard chrome alternative presented herein. Nanovate CoP achieves superior wear and corrosion resistance without any of the environmental hazards inherent to conventional hard Cr^{+6} solutions [ii,iii,iv]. Nanovate CoP is currently commercially available through Enduro Industries (Hannibal, MO) as an alternative to hard chromium for hydraulic bars for the fluid power industry. Pratt & Whitney Canada has also setup a Dem/Val process line for repair/overhaul of engine components.

2.1.1 Technology Development

The nCoP coatings to be evaluated in this project were originally developed under Strategic Environmental Research and Development Program (SERDP) Project WP-1152. The main objective of the SERDP project was to "engineer" the microstructure of electrodeposited cobalt-based coatings to optimize the properties to meet or exceed the performance of EHC.

At the conclusion of the SERDP program, the nCoP coating was shown to exhibit good wear and corrosion resistance, with no hydrogen embrittlement (even without heat treating) and a fatigue debit similar to that of EHC. As a result, an Environmental Security Technology Certification Program (ESTCP) Dem/Val project (WP-200411) was approved for a 2004 start.

In WP-200411, a chrome plating line was successfully converted/retrofitted to accommodate nCoP plating solution at FRCSE, including the installation of a pulse power supply. Integran issued a process specification document to FRCSE to provide instruction on the system operation. Early deposit quality issues included adhesion to low alloy steel substrates and pitting. After a considerable amount of development and testing both at Integran and at FRCSE, the team had resolved most of the early issues. However, the hydrogen embrittlement testing raised concern as to whether the process window was wide enough to use nCoP on typical DoD components without causing embrittlement.

In light of this significant technical risk, supplemental work was approved to define an operating window for a non-embrittling coating without detrimentally affecting the other beneficial properties of nCoP, and to regenerate performance data using optimized operating conditions. After optimizing plating deposition parameters using a design of experiment approach, hydrogen embrittlement was found to be independent of deposition conditions, and in many cases, nCoP passed testing without an embrittlement relief bake. Optimum deposition parameters were found to be non-embrittling with improved fatigue and neutral salt fog corrosion performance as compared to EHC. Producibility evaluations were performed utilizing a J52 Shaft section and J52 Coupling components. Details of the above supplemental work may be found in WP-200411 final report. Table 2-1 and Table 2-2 summarize the process and properties, respectively, of nCoP in comparison to those of EHC at the conclusion of the supplemental work.

Table 2-1. Process Summary for nCoP Compared with that of EHC.

	PROCESS DATA SUMMARY	
	Nanocrystalline Co-P Alloy	**EHC**
Bath Chemistry	Co 1-2wt%P (Co^{2+})	Cr ($CrO3$ / SO_4^{2-})
Efficiency	85-95%	15-35%
Deposition Rate	Up to 200 μm (0.008”) per hour	Up to 40 μm (0.0016”) per hour
Thickness	Demonstrated up to 1000 μm (0.040”)	Typically <500 μm (0.020”)
As-Deposited Appearance	Pit / Pore Free / Crack free	Microcracked
Microstructure	Nanocrystalline (avg. g.s.=8-15 nm)	-
Relative Process Cost	Comparable	-
Emission Analysis	Below OSHA limits	Cr^{+6}

Table 2-2. Property summary for nCoP compared with that of EHC.

PROPERTY DATA SUMMARY			
		Nanocrystalline Co-P	**EHC**
Hardness	*As-Deposited*	550-600 Vickers Hardness Number (VHN)	>800 VHN
	Heat treated (570°F for 5 hours)	700-750 VHN	-
Ductility		2-7% Elongation	<0.1%
Thermal Stability		660°F	-
Wear	*Abrasive (Taber)*	17 mg/1000 cycles (CS-17)	4 mg/1000 cycles (CS-17)
		11 mg/1000 cycles (CS-10)	1.0 mg/1000 cycles (CS-10)
	Adhesive (Pin-on-disk)	6-7 x 10^{-6} mm^3/Newton-meter (Nm) (Al_2O_3 ball on nCoP disk)	9-11 x 10^{-6} mm^3/Nm (Al_2O_3 ball on Cr plated disk)
Corrosion	*Salt Spray*	Protection Rating 8 @ 1000 hours (0.002” thickness)	Protection Rating 2 @ 1000 hours (0.004” thickness)
Internal Stress		10-15 kilopounds per square inch (ksi; Tensile)	Cracked – Exceeds cohesive strength
Hydrogen Embrittlement		None after bake 24 hours	None after bake 24 hours

2.2 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

Electrodeposited nCoP, in addition to being fully compatible with current EHC plating infrastructure, have been demonstrated to provide material performance superiority. Table 2-3 lists the advantages and disadvantages associated with the new technology.

Table 2-3. Advantages and limitations for nCoP.

Advantages/strengths	Disadvantages/weaknesses
Technical • Improved fatigue life, corrosion protection, sliding wear and embrittlement compared to EHC. • Thin uniform coatings down to 0.0002" thick can be applied in contrast with EHC which must be at least 0.001" thick. • Coatings up to 0.040" thick can be produced compared to EHC which is limited to less than 0.020" thick. • The plating rate of nCoP is five times faster than in EHC. • Coating microstructure is fully dense, free of pores and cracks, owing to low residual stress in contrast to EHC, which is microcracked. • nCoP can be ground effectively using current grinding procedures. • Stable electrolyte can be controlled with periodic maintenance and addition of additives. • Application to line-of-sight and non-line-of-sight geometries. *Depot and Original Equipment Manufacturer (OEM)* • Direct drop-in technology for existing EHC infrastructure. • Energy savings as a result of higher efficiency. • Higher throughput resulting from increased plating rate. *Environmental* • Emissions of Cobalt are below PEL, no Cr^{+6}. • Process is worker-friendly. • Anodic material is safe. Typically, chromium plating is accomplished with the use of lead anodes which degrade to form lead chromate sludge necessitating filtration and hazardous waste disposal.	*Technical* • Reduced performance in abrasive wear testing. • Similar to EHC, a post-plating bake-out is necessary to ensure coating is non-embrittling. • Maskant material compatible with plating solution, pH and temperature must be identified although a commercial product has been tested and performed well to date. • Susceptible to contaminants otherwise benign in EHC plating. • Ferromagnetic nature of the coating prevents the use of conventional non-destructive thickness testing. *Depot and OEM* • Pulse power supplies required. Low cost units are currently being developed as part of ESTCP project WP-200934 • Refurbishment of tank is necessary due to elevated operating temperature and lower pH. • Unproven technology in a full production setting although has been demonstrated on a pilot scale.

3.0 PERFORMANCE OBJECTIVES

The functional performance of the nCoP coating was evaluated in accordance with the tests as outlined in the Joint Test Protocol (JTP). Table 3-1 summarizes the performance objectives, success criteria, and results of testing.

Table 3-1. Performance Objectives.

Performance Objective	Data requirements	Success Criteria	Results
Quantitative Performance Objectives			
Deposit Testing			
Thickness uniformity/ deposition rate	Microscopic	Minimum 0.002”	Pass
Coating thickness uniformity	Microscopic	nCoP coating thickness shall be uniform within ±20% for deposition	Pass
Appearance	Visual examination	Smooth, fine grained, adherent, uniform in appearance, free from blisters, pits, nodules, excessive edge build-up, and other defects	Pass
Microstructure	Microscopic	Fine grained, adherent, uniform	Pass
Composition	SEM-EDS	Deposit phosphorus: 1-2 wt%P	Pass
Microhardness	Vicker’s Microhardness	Hardness: nCoP ≥ EHC	Pass on selected heat treatment conditions[1]
		Target Hardness > 850 VHN (EHC requirement) nCoP maximum hardness obtained 763 VHN following heat treatment 550°F for 5 hours.	Fail
		Threshold Hardness > 530 VHN (process requirement)	Pass
Porosity	Ferroxyl	No pits > 1/32” diameter < 15 pits in 150 sq.in < 5 pits in 30 sq.in.	Pass
Grain Size	X-ray diffraction	Grain size < 20 nm Crystal structure: hexagonal close-packed (HCP)	Pass
Ductility	Bend test American Society for Testing and Materials (ASTM) B489	As-deposited ductility > 2%	Pass
Internal Stress	Spiral contractometer	Internal stress 15 ksi maximum (tensile)	Fail[2]
Internal Stress	Copper stress strips	Internal stress 15 ksi maximum (tensile)	Fail[3]
Performance Testing			
Adhesion	Bend/chisel	nCoP does not show separation from the basis metal at the common interface for alloys (4130, 1018, 4130/Ni Sulfamate, 15-5PH, IN718, Al7075-T6, Aermet100)	Pass, Marginal Pass for Hy-tuff

Table 3-1. Performance Objectives. (Continued)

Performance Objective	Data requirements	Success Criteria	Results
Fatigue	Axial fatigue	Stress versus cycles-to-failure (S-N) curve fitted data: nCoP ≥ EHC at 0.003”	Pass
		S-N curve fitted data: nCoP ≥ EHC at 0.010”	Pass
		S-N curve fitted data: nCoP at 0.010” ≥ Ni+EHC at 0.005” ≥ Ni+nCoP at 0.005”	Marginal Pass, at one-load level Ni+nCoP fatigue debit compared to Ni+EHC
Coating integrity	Axial fatigue	The nCoP coatings must not spall or delaminate	Pass
Corrosion	Neutral salt fog	Average appearance/Protection ranking vs time curve: nCoP ≥ EHC per ASTM B537	Pass
Corrosion	SO_2 salt fog	Average appearance/Protection ranking vs time curve: nCoP ≥ EHC per ASTM B537	Pass
Corrosion	Beach exposure	Average appearance/Protection ranking vs time curve: nCoP ≥ EHC per ASTM B537	Pass
Corrosion	Open circuit potential	No acceptance criteria – for information purposes only	nCoP: Voc=-0.47V/SCE EHC: Voc=-0.60V/SCE
Hydrogen embrittlement – relief bake	Hydrogen embrittlement	1a1: four bars > 200h	Pass[4]
Hydrogen embrittlement – no relief bake	Hydrogen embrittlement	Load to failure: nCoP ≥ EHC	Pass
Fluid compatibility	Visual observation and weight loss following immersion	nCoP must not exhibit chemical attack greater than that exhibited by EHC. Pass: MIL-PRF-83282, MIL-PRF-680, Fluorescent penetrant, Cimstar 40, Turco 4181L Alkaline Cleaner, MIL PRF 85570 type 2, Bioact 280	Pass on 8 fluids
		Fail: Nital Etch, Ammonium Persulfate, Chlorine Bleach	Fail on 3 fluids
Environmental embrittlement	Sustained load in saltwater environment	150 hours+ and 45% Notch Fracture Strength (NFS)+: nCoP ≥ EHC in deionized water nCoP ≥ EHC in salt water	Pass (deionized water)[5]
Wear	Taber abrasive (ASTM D4060)	Taber wear index: nCoP ≤ EHC	Fail
	Taber abrasive (ASTM F1978)	Taber wear index: nCoP ≤ EHC	Fail

Table 3-1. Performance Objectives. (Continued)

Performance Objective	Data requirements	Success Criteria	Results
Wear	Pin on disk	Coating wear volume loss, coefficient of friction, static partner wear volume loss: nCoP ≤ EHC Material combinations: nCoP/4130, EHC/4130, 4130, 13-8 stainless steel, Al7075-T6, Cupronickel 70-30 Coefficient of friction: Pass	Pass
		Static partner volume wear loss: Pass	Pass
		Coating wear volume wear loss: EHC ball= Pass, other static partners nCoP ≥ EHC. However, static partner volume wear for nCoP << EHC	Pass
Wear	Endurance rig test	< one drop of hydraulic fluid in 25 cycles and acceptable wear (i.e., not affecting leakage performance)	Pass
Wear	Falex block on ring	Coefficient of friction, average weight loss and average wear volume: nCoP ≤ EHC	Pass
Wear	Gravelometry	nCoP performance equal to EHC	Pass
Wear	SATEC oscillating load	Coefficient of friction, average bushing wear: nCoP ≤ EHC	Pass
Corrosion	Cobalt oxide characterization	No acceptance criteria – for information purposes only Conditions tested: As-deposited, Salt Spray and Beach Exposure	N/A – Oxidation product confirmed to be native Cobalt oxide (CoO and Co_3O_4) which is dense, compact and adherent unlike red rust
NAVSEA (M9 Armored Combat Earthmover [ACE])	Adhesion, impact, corrosion, wear	No acceptance criteria – for information purposes only	N/A – Performance exceeded other candidate materials evaluated
Reduction in hazardous waste generated	Raw materials usage, mass balance Analysis for hazardous materials by EPA standard test number during demo	Reduction in hazardous waste observed based on CBA	Pass
Qualitative Performance Objectives			
Ease of use	Feedback from field technician on usability of technology and time required during demonstration	No operator training required Maskant materials identified (green lacquer and specialty electroplating tape) however is not as easy to use as the current maskants specified for hard chrome	Pass Special to activation processes required to ensure adhesion Must improve masking methods to increase ease of use. Identified potential candidates for use.

Notes

1 See Figure 6-5 thru 6-8.

2 Internal stress 22-26 ksi. However, demonstrated ability to build coating thickness

3 Internal stress 15-20 ksi. However, demonstrated ability to build coating thickness

4 Hydrogen embrittlement relief baked nCoP failed hydrogen embrittlement testing (2 of 4 bars), however supplemental testing on repeat hydrogen embrittlement relief baked nCoP passed hydrogen embrittlement testing (8 of 8 bars)

5 nCoP and EHC both fail in salt water

4.0 SITE/PLATFORM DESCRIPTION

4.1 TEST PLATFORMS/FACILITIES

4.1.1 Industrial Plating Facility

The NAVAIR FRCSE in Jacksonville, FL was selected as the site for demonstration of the nCoP plating technology for tank batch plating. FRCSE has been the most proactive of the Navy depots in implementing high velocity oxygen fuel (HVOF) thermal spray coatings for hard chrome replacement. Through implementation of HVOF and other activities, the depot recently was able to shut down three 2000-gallon chrome plating tanks. The depot, however, still has a significant chrome plating facility with several tanks of size ranging from 200 to 2000 gallons. Because of their expertise, FRCSE has been the lead depot in developing standards and specifications for deposition and grinding of the HVOF coatings. These specifications have recently been issued by the Aerospace Materials Engineering Committee of the Society of Automotive and Aerospace Engineers and will be used by all military repair depots and by manufacturers of weapons systems.

4.1.2 Demo Systems

The following section describes the test platforms and components identified for the Dem/Val of the technology.

4.1.2.1 T-45 Goshawk

The T-45 Training System (T45TS) (see Figure 4-1) developed for and used by the U.S. Naval Air Training Command is the first totally integrated jet aircraft training system. It comprises the Boeing-built T-45 Goshawk, advanced flight and instrument simulators, computer-assisted instructional programs, and a computerized training integration system. The integration of all five elements produces a superior pilot in less time and at lower cost than previous training systems. The T45TS has enabled the U.S. Navy to reduce total student flight-hours by 28 percent and duration of training by 17 weeks.

Figure 4-1. T-45 Goshawk Seen on Aircraft Carrier (left) and In-flight (right).

As of December 31, 2008, the T-45 fleet has logged more than 850,000 flight-hours. Since entering service in 1992, T-45's have made more than 50,000 arrested landings aboard aircraft carriers [v,vi].

The pivot assembly on the arresting tail hook of the T-45 is manufactured from Hy-tuff alloy steel and has been selected as a component for Dem/Val testing of nCoP. Figure 4-2 shows a schematic drawing of the arresting tail hook assembly. Figure 4-3 is an excerpt from a technical manual showing a more detailed arresting tail hook assembly. The cam which comes in contact with Cr-plated roller will be plated with sulfamate Ni and capped with nCoP. It is currently plated with sulfamate Ni and capped with EHC, 0.002" and 0.003" in thickness, respectively. The cam face enables the tail hook to be re-centered when landing. The cam experiences two cycles per landing. This is considered a non-flight critical part. The part is located in an area that is easily accessible for inspections. While in-service, upon reaching 100 arrestments, the tail hook assembly is disassembled for inspection. Using a rig-test, the tail hook assembly is tested for performance in order to determine if it can be returned to service or de-commissioned. De-commissioned cam surfaces are sent for repair and overhaul.

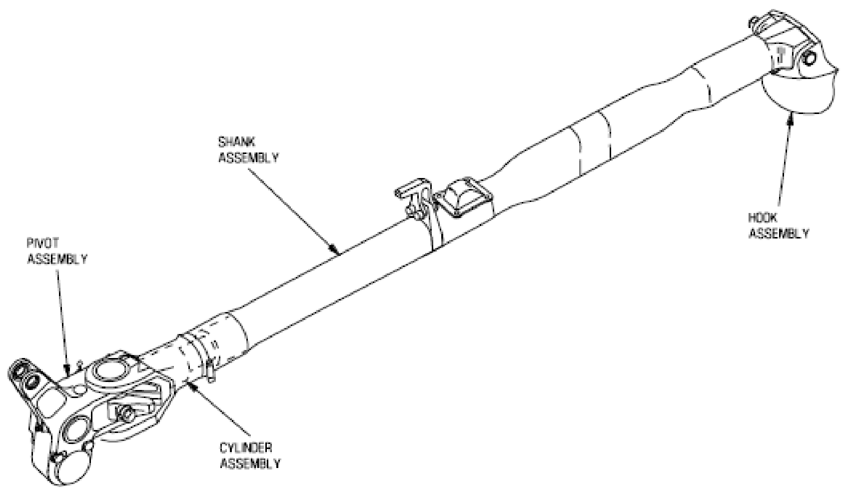


Figure 4-2. Schematic Drawing of the Arresting Tail Hook Assembly.

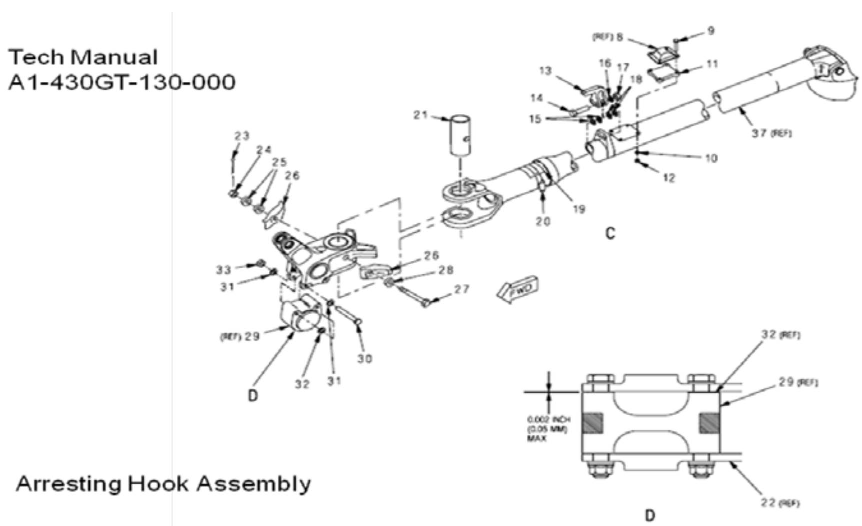


Figure 4-3. Technical Manual Excerpt with a Detailed Schematic of the Arresting Tail Hook Assembly.

4.1.2.2 Spotting Dolly

The A/S32A-32 Aircraft Towing Tractor (see Figure 4-4), also referred to as the “Spotting Dolly," serves as the ground support equipment nCoP demonstration component. Support equipment at sea has particularly demanding requirements due to the unusually harsh conditions the equipment might be exposed to such as: shock, vibration, corrosion and electromagnetic interference.

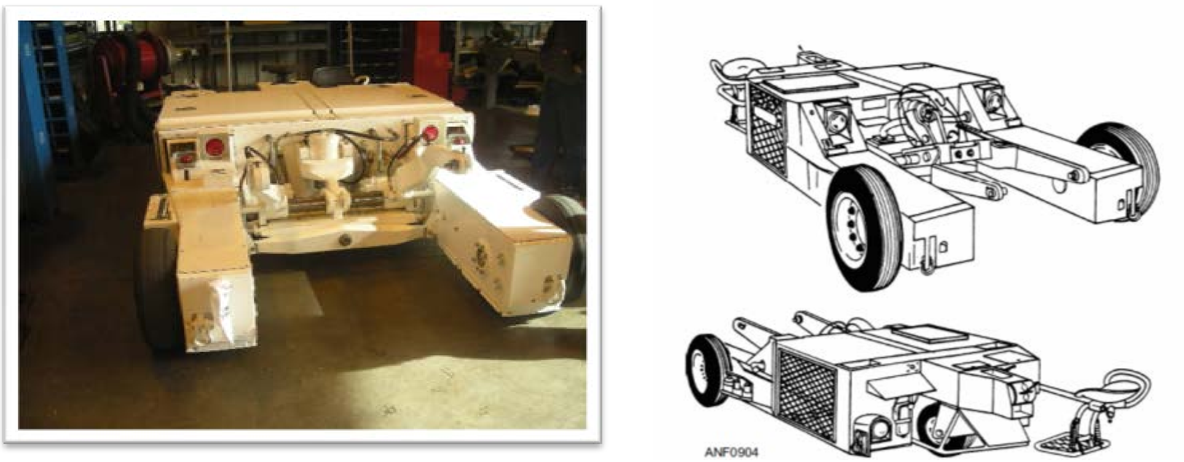


Figure 4-4. Image of the Spotting Dolly (left) and Schematic Drawing of the Spotting Dolly (right).

The spotting dolly lifting pin (P/N: 1117AS243-1) has been selected as the component for Dem/Val of nCoP electroplating. Figure 4-5 illustrates the location identified for application of the nCoP electroplate. This is the primary wear area resulting from aircraft spotting procedures. The Dem/Val component will be electroplated and then installed on a spotting dolly awaiting maintenance at Solomons, MD. This vehicle will then be tracked by NAVAIR Naval Air Warfare Center Aircraft Division (NAWCAD) Lakehurst during deployment by the vehicle’s unique identification number. In this manner, its location and service environment shall be known throughout its deployment. Field data will be obtained by regular correspondence with ship’s force. Final inspection will compare nCoP performance versus in-service baseline lifting pins.

Figure 4-5. Image of Lifting Arm Pin Installed on Spotting Dolly (left) and Close-up (right).

4.1.2.3 M9 ACE

The M9 ACE is a fully tracked armored combat engineer vehicle (see Figure 4-6), that provides combat engineering support to front-line USMC forces. Tasks of this vehicle include digging positions for armored vehicles and field artillery systems to increase their survivability. It also breaches berms, prepares anti-tank trenches, barriers, repairs roads, clears obstacles, and prepares riverbanks for vehicle crossing.

Figure 4-6. Image of M9 ACE Vehicle with an Inset Image of the Hydraulic Cylinder Coated with EHC Plating.

In collaboration with NAVSEA (OSD and Navy's Environmental Sustainability Development to Integration [NESDI] leveraged effort), a hydraulic cylinder on the M9 ACE was selected for the Dem/Val of nCoP. The substrate material is carburized steel. They are currently coated with EHC for corrosion and wear resistance with coating applied to a target thickness of 0.001". Naval Surface Warfare Center Panama City Division (NSWCPCD) procured a M9 ACE apron cylinder assembly that included an EHC cylinder rod. The apron cylinder was sent to NSWCCD for disassembly and then the rod only was shipped to Integran for EHC stripping and plating with nCoP. The nCoP coated rod was then sent back to NSWCCD, where the apron cylinder was reassembled and shipped to the Production Plant Albany (PPA) for 4500 pounds per square inch (psi) pressure testing. Once PPA validated that the cylinder assembly had no internal or external leakage, the Dem/Val prototype cylinder was shipped to NSWCPCD for M9 ACE installation.

In advance of demonstration testing, an evaluation in a cyclical wear and corrosion environment was conducted. The testing simulates field conditions for a different platform (MK48 Logistic Vehicle System [LVS]) with similar functional requirements. The coated cylinders were exposed for 240 hours in an ASTM B117 salt fog chamber and then inspected for corrosion damage. Test cylinders were then loaded into a piston manifold, operated for 1000 cycles, and then inspected for wear and evidence of seal leakage due to either corrosion or cylinder actuation.

Testing was repeated until cylinder failure occurred. Test results for the nCoP coated cylinders were ranked against the currently used EHC coating and other candidate coatings. Figure 4-7 shows disassembled hydraulic tube casings following service.

Figure 4-7. Representative Hydraulic Cylinder Tube Casings from MK48 LVS Disassembled Following Service.

4.1.3 Present Operations

Building 794 at FRCSE houses all cleaning, plating, plasma and HVOF spray, and other coating operations. The facility includes a closed-loop air circulation system that enables compliance with all EPA air emission regulations. EHC plating is conducted in a number of tanks of size ranging from 200 gallons to 2,000 gallons (see Figure 4-8). These tanks are located in a room separate from other plating operations with additional air exhaust capability to minimize worker exposure to plating chemical fumes. Power supplies are located in a separate rectifier room to ensure they are not exposed to the tank vapors which could induce corrosion of the electronic circuitry.

Figure 4-8. Image of a Chrome Plating Tank Currently in Use at FRCSE.

4.2 SITE-RELATED PERMITS AND REGULATIONS

This section identifies permits and potential regulations that were applicable to the demonstration and may be relevant to future implementation.

4.2.1 Environmental Checklist

- Permit for air emissions from scrubber
- Permit for release of rinse tank contents to on-site water treatment facility
- Permit for in-process neutralization of spent plating solution (hazardous waste)
- Permit for disposal of spent plating solution (hazardous waste)

The Environmental, Health and Safety Plan for the plating facility at FRCSE will be the overarching document that will apply to the nCoP process.

4.2.2 Other Regulatory Issues

Cobalt is not covered under the Clean Air Act regulations. The PEL for cobalt metal is 0.1 mg/m^3. From work conducted as part of SERDP WP-1152 and ESTCP WP-200411, the cobalt metal present in mist when the tank is in operation is below the PEL. A reasonable degree of care in waste disposal and grinding operations should be considered to ensure PEL requirements are met.

Cobalt salts were added to a candidate list for inclusion in Annex XIV, for consideration as a substance of very high concern under EU Registration, Evaluation, Authorization and Restriction of Chemicals legislation. They are being considered for alternative risk management options currently. Pre-authorization of cobalt salts by suppliers will enable use of the chemicals for manufacture within the EU jurisdiction. The addition of cobalt salts does not preclude importation of nCoP coated articles manufactured outside the EU. Major chemical suppliers have indicated that they intend to register for authorization for surface finishing users. To obtain more information refer to the Cobalt Development Institute.

4.3 END-USER/OEM ISSUES

A critical aspect associated with the validation of the nCoP plating technology for EHC replacement is the involvement of the stakeholder community throughout the project. Because of the success of the Hard Chrome Alternatives Team (HCAT) program on validation of HVOF thermal spray technology to replace hard chrome, the relevant stakeholders have already been identified and involved in that effort. For this project, the same stakeholders were consulted for development of the JTP and other requirements for qualification.

5.0 TEST DESIGN

5.1 JTP TESTING

This section details the work conducted under the Joint Test Report (JTR) as previously defined in the JTP. Table 5-1 and Table 5-2 summarize the tests, acceptance criteria, and references for common tests and extended tests, respectively. The tests are described in greater detail in subsequent sections.

The following section is intended to contain sufficient detail such that all stakeholders can review the results of testing conducted. However, to avoid excessive length of the document, several standards and specifications are referenced without providing details. The military standards, ASTM standards, and Aerospace Material Standards (AMS) that are referenced are considered to be readily available and no further information needs to be provided.

5.2 DEM/VAL TESTING

5.2.1 Conceptual Experimental Design

Dem/Val testing performed in this project involved fabricating nCoP coupons for JTP testing as well as Dem/Val articles for field testing. The nCoP plating line was installed in Building 794 at FRCSE. The facility includes a closed-loop system that enables compliance with all EPA regulations. The line consists of an activation tank, the nCoP plating tank, and associated rinse tanks (Figure 5-1). All prior processing steps, i.e. cleaning, blasting, masking & racking, are performed within the general area of plating facility.

Figure 5-1. nCoP Dem/Val Plating Line Location in Building 794 at FRCSE.

Figure 5-2. Photograph of 300 Gallon nCoP Plating Tank at FRCSE.

Table 5-1. Common Performance and Testing Requirements.

Engineering Requirement	Test	JTR Test Description - Section	Acceptance Criteria	References
Appearance	Visual examination	4.1	Smooth, fine grained, adherent, uniform in appearance, free from blisters, pits, nodules, excessive edge build-up and other defects	AMS 2460
Thickness uniformity/ deposition rate	Microscopic	4.1	Minimum 0.002”	ASTM B487 AMS 2460
Porosity	Ferroxyl	4.1	No pits > 1/32” diameter < 15 pits in 150 sq.in < 5 pits in 30 sq.in.	AMS 2460
Hardness	Vicker’s Microhardness	4.1	Hardness: nCoP ≥ EHC Target Hardness > 850 VHN Threshold Hardness > 530 VHN	ASTM B578 AMS 2460
Grain Size	X-ray diffraction	4.1	Grain size < 20 nm Crystal structure: HCP	N/A
Ductility	Bend test	4.1	As-deposited ductility > 2%	ASTM B489
Stress	Spiral contractometer or stress strips	4.1	Internal stress 15 ksi maximum (tensile)	ASTM B636
Fatigue	Axial fatigue	4.2	S-N curve: nCoP ≥ EHC at a given thickness nCoP or Ni+nCoP ≥ Ni+EHC	ASTM E466
Coating integrity	Axial fatigue	4.2	The nCoP coatings must not spall or delaminate.	N/A
Corrosion	Neutral salt fog	4.3	Average appearance/protection ranking vs time curve: nCoP ≥ EHC per ASTM B537	ASTM B117 ASTM B537
Corrosion	SO_2 salt fog	4.3	Average appearance/protection ranking vs time curve: nCoP ≥ EHC per ASTM B537	ASTM G85 A4 ASTM B537
Corrosion	Beach exposure	4.4	Average appearance/protection ranking vs time curve: nCoP ≥ EHC	ASTM B537
Corrosion	Open circuit potential	4.5	No acceptance criteria – characterization test.	ASTM G3
Adhesion	Bend/chisel	4.6	nCoP does not show separation from the basis metal at the common interface	ASTM B571
Hydrogen embrittlement – relief bake	Hydrogen embrittlement	4.7	1a1: four bars > 200 hours	ASTM F519
Hydrogen embrittlement – no relief bake	Hydrogen embrittlement	4.7	load to failure: nCoP ≥ EHC	ASTM F519
Fluid compatibility	Visual observation and weight loss following immersion	4.8	The nCoP must not exhibit chemical attack by required operating and maintenance fluids or reasonable substitutes.	N/A

Table 5-1. Common Performance and Testing Requirements. (Continued)

Engineering Requirement	Test	JTR Test Description - Section	Acceptance Criteria	References
Environmental embrittlement	Sustained load in saltwater environment	4.9	Time to failure and load to failure: nCoP ≥ EHC	ASTM F519
Wear	Taber abrasive	4.10	Taber wear index: nCoP ≤ EHC	ASTM F1978 ASTM D4060
Wear	Pin on disk	4.11	Coating wear volume loss, coefficient of friction, static partner wear volume loss: nCoP ≤ EHC	ASTM G99
Wear	Endurance rig test	4.12	< one drop of hydraulic fluid in 25 cycles; wear not affecting leakage performance.	N/A
Wear	Falex block on ring	4.13	Average weight loss and average wear volume: CoP ≤ EHC	ASTM G77
Wear	Gravelometry	4.14	CoP performance equal to that of EHC	ASTM D3170
Wear	SATEC oscillating load	4.15	Average friction and bushing wear: CoP ≤ EHC	N/A
Corrosion	Cobalt oxide characterization	4.16	No acceptance criteria – characterization test	N/A

Table 5-2. Extended Performance and Testing Requirements from NAVSEA.

Engineering Requirement[1]	Test	JTR Test Description - Section	Acceptance Criteria[1]	References
Adhesion	Chisel-knife grind saw	N/A	Per NSWCCD Test Protocol	ASTM B571
Corrosion	Accelerated corrosion	N/A	Per NSWCCD Test Protocol	ASTM B117
Adhesion/wear	Impact resistance	N/A	Per NSWCCD Test Protocol	ASTM B571
Wear	Taber abrasive	N/A	Per NSWCCD Test Protocol	ASTM D4060
Wear	Cyclic piston wear/corrosion	N/A	Per NSWCCD Test Protocol	ASTM B117 ASTM B610

Notes

1 Test methods and acceptance criteria detailed in Evaluation of Hard Chrome Replacements for Hydraulic Cylinders on USMC Vehicles funded through USMC and OSD, a NESDI leveraged effort.

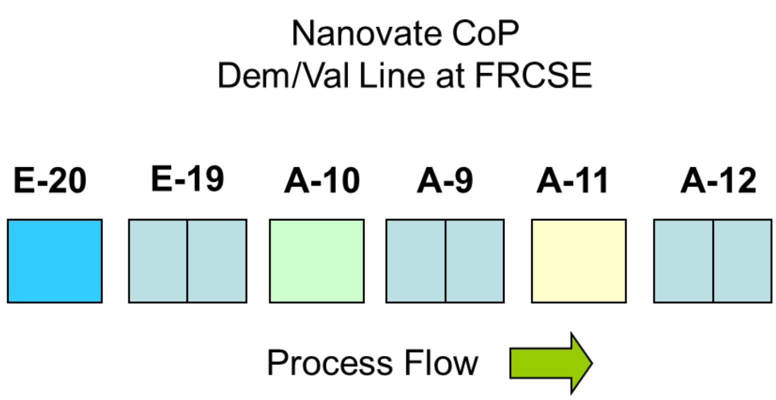
A technical process guide was provided by Integran to FRCSE to successfully operate the nCoP plating tank. Within the document, explicit operating parameters, maintenance schedules, and process specifications are contained. This document has been utilized to generate an internal military specification. The nCoP electroplating deposition process was performed by following the FRCSE Local Process Specification using the Process Plating Guidance provided by Integran. The document consists of various process steps which are described in more detail below.

An existing process tank within the chrome plating room at FRCSE, identified as Plating Tank A-11 (formerly used for EHC plating), was dedicated to the nCoP process. The tank has a capacity of ~ 300 gallons and was equipped with a drop-in liner made of a modified vinyl-based polymer, 0.125” thick. Other installed equipment includes a steam coil, steam line, regulator, in-tank pump, and filter using 5 µm nominal size polypro cartridges. A sparger equipped with eductors, Ni plated copper buss bars and a level sensor for maintaining the solution level was also installed. Titanium anode mesh baskets with anode bags were also installed to hold the 99.9% pure cobalt anode pieces. Figure 5-2 shows the installed nCoP process tank at FRCSE.

5.2.2 Deposition Process

Figure 5-3 presents a process flow chart for the current configuration for the Dem/Val nCoP process line at FRCSE.

Table 5-3 describes the typical processing flow for common carbon steel work pieces. Some additional steps are omitted for selected carbon steel, stainless steels and non-ferrous alloys. All samples and Dem/Val articles produced in this project followed this deposition protocol.



Tank#:	Process Step	Temp (°F)
E-20	Alkaline Cleaner	120 – 130°
E-19	Rinse	RT
A-10	Acid Activation	RT
A-9	Rinse	RT
A-11	Nanovate CoP Plate	180-190°
A-12	Rinse	RT

Figure 5-3. Process Flow Chart.

Table 5-3. Process Sequence for Selected Metals and Alloys.

General Plating Process Steps
nCoP electrodeposition on 4130 Steel

Title	Solution
nCoP Plate	Proprietary Alloy Plating Bath (nCoP)
Electrocleaner	P-C-535 (Alkaline Soak) or equivalent
Acid Activator	H_2SO_4/NH_4HF_2 (Acid Bath)

Step No.	Description	Time	Remarks	Temp (°F)
1	Degrease as necessary		Solvent clean/ wipe	
2	Inspect surface for defects			
3	Abrasive blast		Glass or AlOx grit (size 220)	
4	Remove excess blast media			
5	Verify pre-plate dimensions			
6	Tape, rack & mask (as necessary)		Enthone Stop-Off No. 1, or other suitable maskant	
7	Alconox/pumice scrub area to be plated if necessary	As req.	1 part Alconox to 100 parts pumice	
8	Rinse			Room
9	Electroclean (anodic)	1-3 minutes	4 to 6 volts anodic or (45 – 55 ASF)	125-135
10	Rinse			Room
11	Reverse anodic etch (activation)	40 seconds for 4130	250 ASF for 4130	Room
12	Rinse			Room
13	nCoP plate	As req.	116 ASF *(50% duty cycle)*	185
14	Rinse			Room
15	Inspect plating		Inspect for conformance	
16	De-mask/de-rack			
17	Bake for hydrogen embrittlement relief/deposit hardening	23 hours		375 ± 25

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6.0 PERFORMANCE ASSESSMENT

6.1 JTR RESULTS

The following section summarizes tests performed to validate the nCoP electroplating process prior to preparing samples and conducting material testing as defined in the JTP.

6.1.1 Coating Quality

Prior to application of the nCoP coating to samples used for JTP testing, coating quality tests were successfully performed to validate the process met specifications as defined in previous work, namely the composition, microstructure, thickness uniformity, and porosity. The adhesion of the coating to relevant metals and alloys used within aerospace was also demonstrated successfully.

6.1.1.1 Hardness

All nCoP samples met the threshold hardness of 550-600 VHN in the as-deposited condition. The nCoP samples met the target hardness of 600 VHN at selected heat treatment conditions: the hydrogen embrittlement bake out condition (375ºF for 23 hours); T-45 bake out condition (475ºF for 23 hours); and max hardening condition (550ºF for 4 hours). The hardness of nCoP samples was equal to or greater than EHC in the T-45 bake out condition and max hardening condition. The microhardness of nCoP and EHC at various heat treatments is summarized in Figure 6-1.

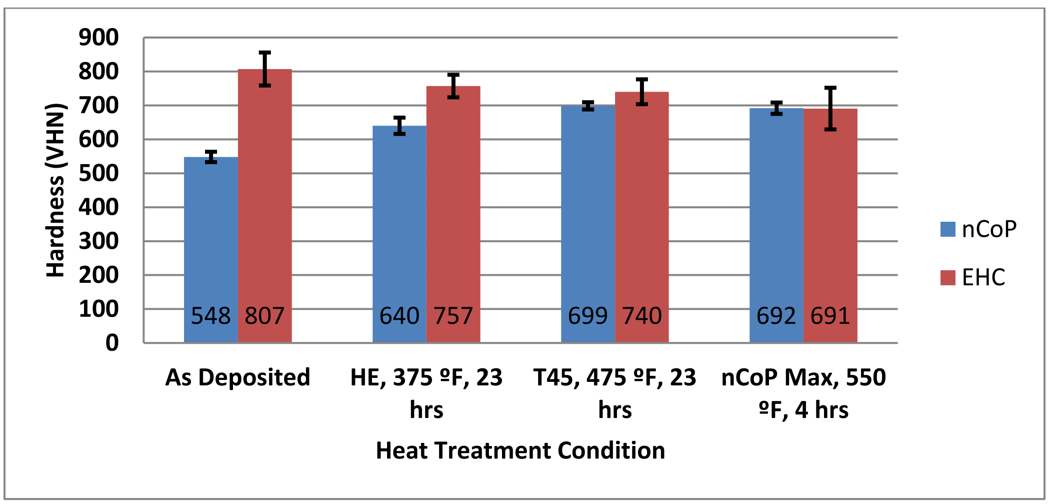

Figure 6-1. Microhardness of nCoP and EHC at Various Heat Treatment Conditions.

6.1.2 Fatigue

The axial fatigue performance of the nCoP was evaluated on 4340 steel specimens at varying coating thicknesses: 0.003”; 0.005”; 0.010”; and 0.020”. The testing was performed in ambient air using load control in a uniaxial configuration with a stress ratio of R = -1 and frequency of 20 hertz (Hz; sine wave). Based on consultation with Boeing, an hourglass round bar geometry was selected. Smooth curve fits were applied to S-N curves in order to compare fatigue life with bare steel and hard chrome coated on steel. At most load conditions, nCoP samples performed equivalent or in some cases provided a fatigue credit relative to hard chrome.

Notable exceptions were nCoP combined with a sulfamate Ni underlayer, where there was a fatigue debit observed at low loads relative to EHC and nCoP coatings tested at 130 ksi load levels for 0.003” coating thickness. Note that this fatigue debit was assessed qualitatively. No statistical correlation was observed.

6.1.3 Corrosion

Corrosion performance was evaluated in salt spray testing. Testing demonstrated equivalent or improved performance of nCoP relative to EHC in neutral media, SO_2 media, and in beach exposure testing conducted at Kennedy Space Center. A thin cobalt oxide layer formed on panels when exposed to air or select simulated environments such as salt fog. This oxide layer has been previously misinterpreted as red rust during visual inspection. The composition of the oxide was confirmed by x-ray photoelectron spectroscopy analysis to contain no ferrous compounds. The oxide composition consisted of cobalt’s natural forming oxides: CoO and Co_3O_4. Through corrosion testing, it has been established that the cobalt oxide is dense, compact and adherent. No substrate corrosion was observed through nCoP coatings.

The open circuit potential for nCoP was compared to hard chrome in simulated salt water and showed a slight ennoblement towards passive potentials. The measured potential for nCoP and EHC on 4130 steel substrates was -0.47V_{SCE} and -0.60V_{SCE}, respectively, after 7 days of monitoring. For reference, the measured potential for 4130 steel substrates was -0.77V_{SCE} after 1 day of monitoring. The impact of substrate exposure through the micro-cracked microstructure of EHC on 4130 may explain discrepancies between reported data and those found in the literature. Testing performed by two independent laboratories obtained similar results.

6.1.4 Hydrogen Embrittlement

Hydrogen embrittlement testing on nCoP showed equivalent performance to hard chrome. Following no hydrogen embrittlement relief bake-out, nCoP samples passed sustained load testing for 200 hours. Unexpectedly, the hydrogen embrittlement relief baked nCoP samples failed for 2 of 4 samples tested. An additional 8 samples were coated and baked in order to repeat testing. The repeat testing revealed that 8 of 8 samples passed sustained load testing for 200 hours. Application of thick builds (i.e., >0.003”) of nCoP and EHC resulted in several hydrogen embrittlement failures. The high variability in test results obtained for thick builds may be related to compromised geometry of the bar notch due to uneven coating thickness distribution, resulting in excess stress concentration.

Environmental embrittlement testing was performed at two coating thicknesses (0.003” and 0.010”) in deionized water and synthetic seawater. Testing showed equivalent performance between nCoP and EHC. For a coating thickness of 0.003” in deionized water, samples passed 150 hours for nCoP and EHC. For coating thicknesses of 0.010” in synthetic seawater environments, all consistently failed in testing for nCoP and EHC. This test is most commonly specified for sacrificial coatings and when evaluating compatibility of cleaners used by maintainers, therefore the significance of this result is not well understood at this time.

6.1.5 Fluid Compatibility

Fluid compatibility testing revealed no attack of nCoP samples exposed to several common maintenance fluids encountered in the field. Chlorine bleach, nital, and ammonium persulfate were found to attack nCoP and therefore exposure should be limited. Fluid compatibility of hard chrome with the same three fluids revealed no attack. The results obtained for nCoP shows very similar results to those found for HVOF Tungsten Carbide (WC)-Cobalt, which is currently approved and widely used for chrome replacement on aircraft.

6.1.6 Wear Testing

Several wear tests were performed to compare nCoP with EHC. The nCoP samples were tested in the as-deposited and heat-treated condition (i.e., hydrogen embrittlement relief bake-out and maximum hardness heat treatments were applied to increase microhardness via precipitation hardening). No significant differences in wear performance were observed for nCoP at varying levels of microhardness. While the microhardness of nCoP is lower than EHC, no decrease in wear performance compared to EHC is noted with exception of Taber wear.

- Pin-on-disc sliding wear testing revealed an improvement in coefficient of friction and wear loss for nCoP-coated pins for all material combinations evaluated. Some hard chrome coated samples exhibited less wear loss for discs. However, this was accompanied by significant wear loss on mating pins. Overall, the sliding wear performance of nCoP exceeds EHC.
- Taber abrasive wear revealed nCoP performed poorly relative to EHC.
- Relevant application-oriented abrasive and impact wear by gravelometry testing was performed. Despite poor Taber abrasive wear performance, nCoP performed equivalent to EHC in this test. Inspection of the microstructure following testing revealed EHC samples were significantly porous due to expansion of micro-cracking following impact with gravel media while nCoP remained fully dense.
- Falex and SATEC oscillating load wear testing showed a minor reduction in coefficient of friction for nCoP relative to EHC for all material combinations. Significant variability was noted in wear loss measurements. Flaking and cracking of both nCoP and EHC coatings were observed following testing, which may be an indication of poor grinding or sample preparation.
- Rod-seal testing was performed by Messier-Bugatti-Dowty. Despite performance issues with the testing apparatus, two seal material configurations were compared for nCoP and EHC samples. The measured leakage performance for nCoP and EHC met the acceptance criteria and performed equivalently.

6.1.7 NAVSEA – Adhesion, Impact, Corrosion, and Wear Testing

NAVSEA conducted a project to evaluate several alternative surface treatments for use on USMC hydraulic cylinders. Adhesion, impact, corrosion, and wear tests were completed on laboratory samples and the performances of the alternative surface treatments were compared to the performance of hard chrome plated hydraulic cylinders currently used on USMC vehicles.

This section focuses on results obtained on nCoP samples and omits results on alternative samples. A full report is available titled “Evaluation of Hard Chrome Replacements for Hydraulic Cylinders on USMC Vehicles” (NSWCCD-TR-61-2012/63, October 2012).

Based on the results, the nCoP plating system provided superior protection to hydraulic cylinders compared to EHC. The nCoP plating performed similarly to or better than hard chrome in adhesion, impact resistance, and wear resistance testing, and significantly outperformed hard chrome in corrosion testing. The nCoP samples lasted over three times longer than any other sample, including hard chrome, in the hydraulic cycling test with corrosion events. Financial and production data showed that while nCoP plating is more expensive than hard chrome, a net savings is achieved due to the extended life of the asset. It was recommended that field testing of nCoP plated hydraulic cylinders be conducted to validate these laboratory results.

A hydraulic cylinder was selected for a Dem/Val on the M9 ACE as a result of the outcome of this work.

6.2 DEM/VAL RESULTS

6.2.1 T-45 Pivot Assembly

Due to the amount of wear during service, rework of the T-45 Pivot Assembly requires the cam surface to be plated with a minimum of 2 mils (0.002”) Ni followed by a minimum of 3 mils (0.003”) chromium in the as-plated condition. In this study, two ea. T-45 Arresting Hook Pivot Assemblies were selected for field demonstration through operational flight testing of U.S. Navy T-45 aircraft located at the Naval Air Station Meridian for evaluating field performance between nCoP and the baseline EHC coating. One ea. pivot (P/N: DA327A5213-17, S/N: 0002PG-19DP) was selected to be plated with nCoP and one ea. pivot (P/N: DA327A5213-17, S/N: 006PG-204DR) was selected to be plated with EHC as the baseline component.

6.2.1.1 Plating Process of T-45 Pivot

Figure 6-2 visually illustrates the general plating process steps that were taken for processing the T-45 Dem/Val component. Activation and rinse steps are not shown.

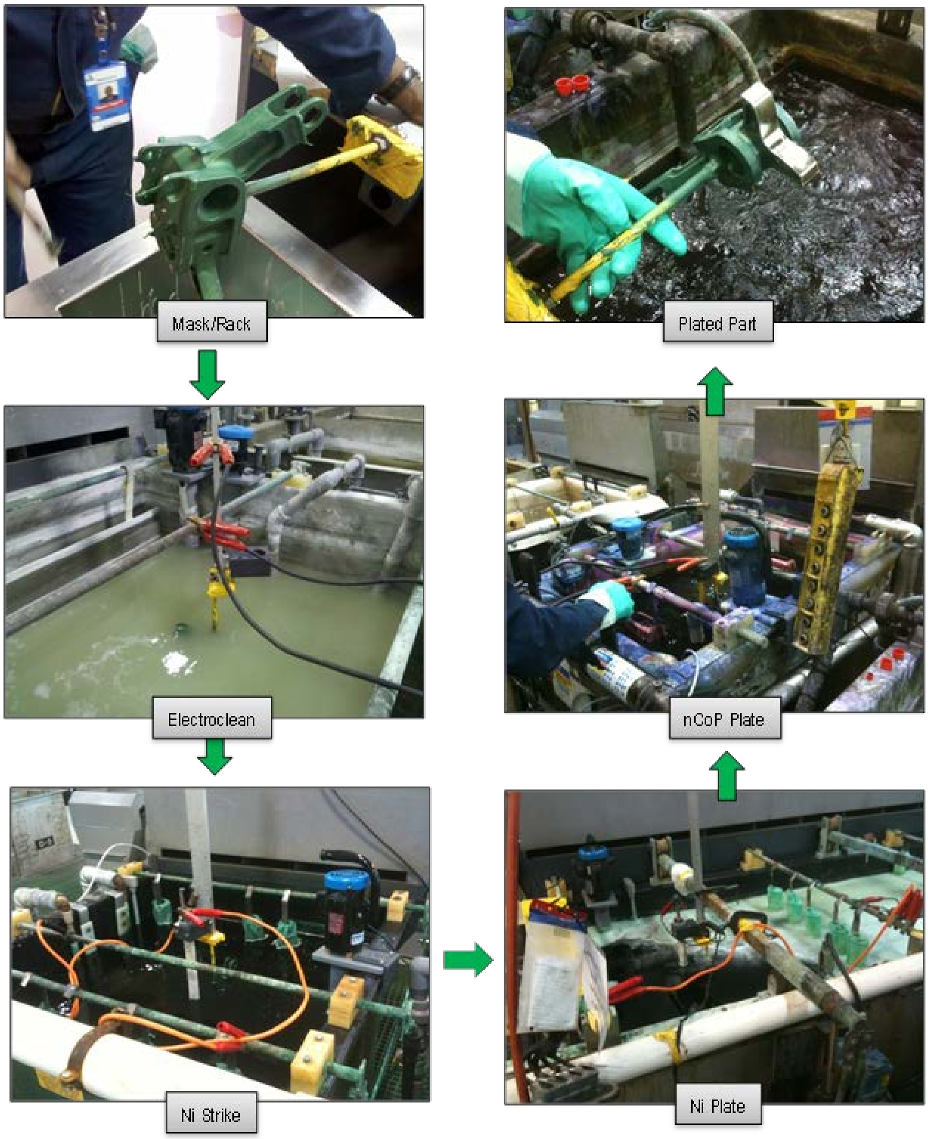

Figure 6-2. Visual Illustration of the General Plating Process Flow.

6.2.1.2 Installation and Tracking of Field Components

The nCoP plated Dem/Val pivot was successfully installed on aircraft as shown in Figure 6-3. Components processed were tracked by serial number and identified within the applicable Scheduled Removal Component (SRC) card as a T-45 engineering study. A mandatory off aircraft inspection is currently established at every 100 carrier arrestments. During this inspection the arresting hook is removed from aircraft, cleaned, disassembled, and prepared for visual and ultrasonic NDI evaluation. At the time of this report, only the nCoP plated pivot assembly had reached a sufficient amount of required arrestments for a full off aircraft inspection of the plated cam surface. A record of field history data was extracted from the SRC cards for both nCoP and EHC plated pivots.

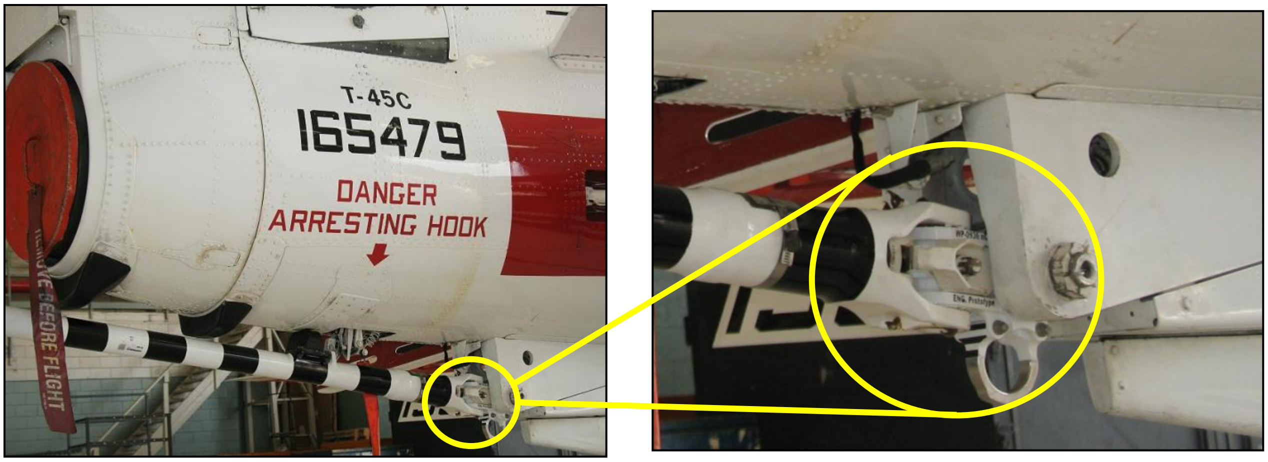

Figure 6-3. nCoP Plated Pivot Installed on A/C BUNO 165479.

6.2.1.3 Field Performance Summary

To date, field performance of the nCoP plated pivot assembly has performed equivalent or better than the baseline coating. Figure 6-4 shows the condition of the cam surface during field inspection following 97 arrestments. Based on visual inspections of the nCoP coated pivot (-19DP), no indication of corrosion or wear damage has been reported. Due to the number of arrestments still pending, the EHC plated pivot (-204DR) has yet to be inspected off aircraft but will continue to be tracked until such time. At present, the nCoP plated pivot is reported to have a total of 116 arrestments with 900 ± 15 flight hours. The EHC plated pivot is reported to have a total of 63 arrestments with 795 ± 5 flight hours. Both nCoP and EHC plated field components are still in the field with no reported issues and will remain on aircraft to generate additional performance validation data in support of technology transition. Components will be routed to FRCSE for refurbishment if plating damage is observed beyond the scope of repair by the operating site.

Figure 6-4. Images of Cam Surface of nCoP plated Pivot Following 97 Arrestments.

6.2.2 Spotting Dolly Lifting Pins

A/S32A-32 Spotting Dolly lifting pin demonstration consisted of field testing within a carrier based operational environment aboard the CVN 75. In preparation for demonstration, lifting pins were procured from the national supply system via NSN: 5315-01-081-6481, P/N: 1117AS243-1 during May 2011. Upon receipt, pre-existing electroplated coatings were removed via chemical striping and mechanical blasting. Items were then prepared for application of nCoP electroplate.

6.2.2.1 Fielding and Inspection

Following completion of electroplating operations by Integran and Ion Vapor Deposition-Al post processing by FRCSE, demonstration components were transferred to Commander, Naval Air Force Atlantic (COMNAVAIRLANT), Norfolk, VA for installation upon the next CVN scheduled for sea duty January 2013. March 2013 demonstration components were installed within the A/S32A Spotting Dolly (S/N: QCF137) aboard the CVN 75. While onboard, the CVN 75 deployed to the Mediterranean Sea and Persian Gulf regions supporting Operation Enduring Freedom.

Demonstration components were evaluated during regularly scheduled NDI inspections established at a 91-day interval. Feedback reports were provided to NAWCAD Lakehurst via email by the ASCS Charles VanSteinburg of the Aircraft Intermediate Maintenance Department (AIMD) for the CVN 75. Most reports were received as qualitative assessment to minimize impact upon fleet operations. Notable comments included: i) nCoP lifting pins exhibited no defects; ii) Several legacy lifting pins were replaced due to failed NDI inspection; and iii) nCoP lifting pins were easier to prepare for NDI as compared to legacy components, thus reducing inspection man-hours by eliminating the need for bead blast.

Field photos were obtained during September 2013 and January 2014 and are shown in Figure 6.5. As of January 2014, an estimated 672 aircraft moves were performed without failure or defect. Figure 6-6 summarizes the demonstration background timeline.

Figure 6-5. CVN 75 Reported that Approximately 672 Aircraft Moves Have Been Performed at Time of Photo.

Figure 6-6. Lifting Pin Demonstration Background Timeline.

6.2.2.2 Field Performance Summary

The nCoP electroplated lifting pins performed better than legacy components onboard the CVN 75. The general area of operation was the Mediterranean and Persian Gulf regions. As of January 19^{th}, the spotting dolly has operated for 672 distinct events since installation of the nCoP coated lifting pins.

According to ASCS VanSteinburg, IM4 LCPO, on board the CVN 75, "Not having to clean (bead blast) prior to 91-day PMS [inspection] has been a real time saver and reduces TAT [turnaround time] on these mission essential items. These pins are a dream to work with; considering no prop work is required for NDI. Hopefully this project leads to all pins, including the adapter pins, having this coating."

Non-nCoP coated pins require 2–2.5 hours of preparation prior to an NDI to remove a corrosion preventative coating, and then reapplication, after the NDI has been complete. The elimination of corrosion preventative coating is likely a result of the superior corrosion protection afforded by nCoP. As of the installation date, there have been no issues with these pins during service, nor has the 91-day NDI revealed any structural weakness in the pins themselves. Recently, the cognizant engineer for the pins, Douglas Kilgore, Code 4.8.6.9, provided this office and the program with a letter of endorsement, citing the feedback from the fleet concerning ease of inspection and the robustness of the nCoP coating's anti-corrosion robustness. Currently, non-nCoP coated pins are drawn from the stock system at 22.5 parts/quarter, over the last 24-month period.

6.2.3 M9 ACE HYDRAULIC CYLINDER

The M9 ACE hydraulic apron cylinder Dem/Val consisted of pressure testing, assembly and installation on vehicle followed by field testing at NSWCPCD. Technical points of contact for initiating, organizing and performing field demonstrations are presented as Table 6-1.

Table 6-1. Demonstration Points of Contact.

POC	Organization	Location
Denise Aylor	Corrosion Engineering	NSWCCD
Jeff Dinges	M9ACE	NSWCPCD

6.2.3.1 Processing

Electroplating was performed at Integran in December 2013 in accordance with the Nanovate R3010 process guide. The component provided by FRCSE was stripped of EHC. Maskant was applied to areas where no coating was desired. Coating was applied to the outside diameter surface selectively as shown in Figure 6-7.

Figure 6-7. Images of the nCoP Coated component at Integran.

6.2.3.2 Fielding & Inspection

The original test plan for the apron cylinder Dem/Val was a one year exposure on the M9 ACE, which included an interim visual inspection at six months and a final evaluation at 12 months.

The apron cylinder was installed in March 2014 and was removed from the test vehicle in May 2014, after approximately two months of exposure. The M9 ACE vehicle selected for the Dem/Val was NSWCPCD's Program Management vehicle used for local operations, tests and instructional purposes. During the field trial, an operational need for the M9 ACE presented itself and resulted in the prototype Dem/Val cylinder being removed from the vehicle and a production cylinder installed in its place for verified product assurance and operation. The Dem/Val cylinder was then shipped back to NSWCCD for final inspection. Post exposure visual examination of the nCoP coated cylinder showed no indication of any coating degradation or damage. Figure 6-8 shows the prototype Dem/Val nCoP coated apron cylinder after installation on the M9 ACE vehicle, and Figure 6-9 shows the unassembled hydraulic cylinder after the field test exposure on the M9 ACE.

Figure 6-8. Image of Installed Prototype nCoP Dem/Val Cylinder Assembly on the M9 ACE.

Figure 6-9. Image of Unassembled Prototype Dem/Val Cylinder Assembly Following Two Months of Field Exposure on the M9 ACE.

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7.0 COST ASSESSMENT

7.1 COST ANALYSIS AND COMPARISON

The cost breakdown for EHC compared to nCoP related to production/labor, materials and supplies, utilities and environmental costs is shown in Table 7-1. Although the cost for nCoP supplies is higher, the utility and environmental costs for nCoP are lower, resulting in a total cost per unit area for nCoP of about a third that of hard chrome.

The cost breakdown for the two coatings is compared in Figure 7-1. The major differences are found in Utilities (because of the plating power requirements), and in Environmental costs (because of the cost of running the scrubber for Cr^{6+}). If OSHA compliance costs were included, the cost of chrome plating would be completely overwhelmed by them. OSHA compliance costs were therefore not included because the savings would only be realized when almost all the Cr^{6+}-producing materials and processes are eliminated (not just EHC plating).

Table 7-1. Breakdown of Cost per sq ft.

Summary of Production Cost	EHC total cost/ sq ft	nCoP total cost/ sq ft
Production labor	$596	$629
Materials and supplies	$100	$162
Utilities	$1,157	$174
Environmental	$1,476	$135
Total cost	**$3,329**	**$1,100**

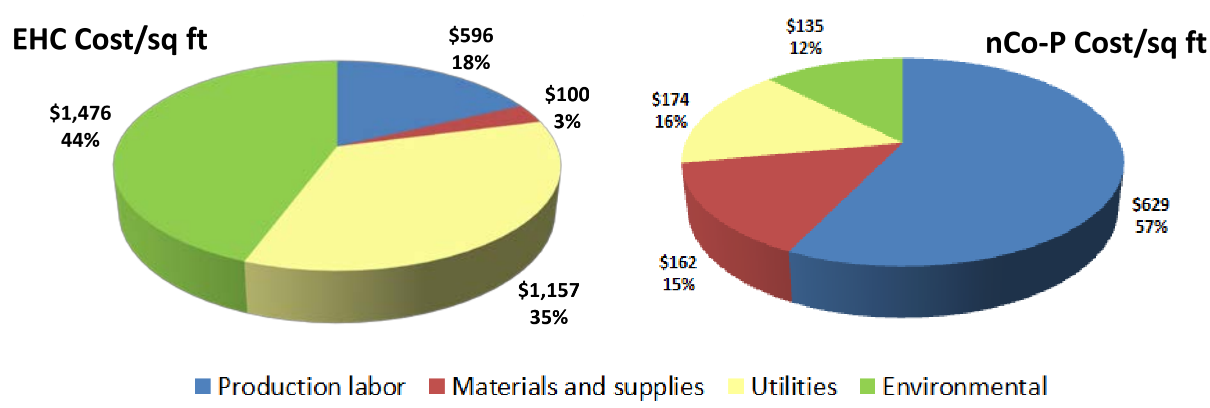

Figure 7-1. Cost Breakdown for EHC and nCoP.

7.1.1 Cost-benefit for replacing hard chrome plating with nCoP

The cost of continuing to use Cr^{6+} processes has not been included in our cost analysis, since the depot must have eliminated almost all Cr^{6+} materials and processes before the OSHA regulatory savings can be realized.

The cost-benefit was evaluated using the C-MAT software, which is designed specifically for the evaluation of material and coating alternatives, especially for depot level sustainment. It compares the cost of continuing to use the current technology with adopting and using the new technology. The above data, except Table 7-1, were assembled into the C-MAT model. The CBA results are shown in Table 7-2 assuming an accuracy of 20% in most of the inputs. Table 7-2 shows that the payback period is about 5 years. The 15-year net present value (NPV), internal rate of return (IRR) and return on investment (ROI) are all reasonable, even for the small chrome plating workload at FRCSE.

Table 7-2. CBA 15-year Value Measures, Assuming 20% Accuracy.

	-2 sigma	Value	+2 sigma
NPV	$1,304,750	$1,549,073	$1,793,397
IRR	37%	25%	19%
Annualized ROI	18%	26%	34%
Total ROI	189%	220%	250%
Payback period	6.1	4.7	3.3

The effect of halving or changing the workload is shown in Table 7-3. As expected, doubling the workload significantly improves the payback, and even halving it still produces a positive payback. We can obtain a rough idea of how this dependence on workload will affect different FRCs. For example, the chrome plating workload at FRCSW (North Island) is approximately 5 times the FRCSE workload. Simply substituting the higher workload into the model produces the right-hand column of Table 7-3 (i.e., we take that same cost structure as FRCSE but substitute a workload comparable with FRCSW). This doubles the IRR but does not make a large difference in NPV or ROI. However, it produces such as a large annual cost change that the payback occurs within the first year. This is shown in Figure 7-2, where the cumulative cost is shown for workloads typical of FRCSE and FRCSW.

Table 7-3. CBA 15 Year Value Measures for Different Plating Workloads.

	48 sq ft/yr	95 sq ft/yr	190 sq ft/yr	530 sq ft/yr (e.g. FRCSW)
NPV	$1,160,672	$1,549,073	$1,937,475	$2,263,185
IRR	22%	25%	29%	59%
Annualized ROI	23%	26%	30%	26%
Total ROI	206%	220%	233%	308%
Payback period	5.6	4.7	4.0	0

Note that this comparison only takes into account differences in workload in the same depot (FRCSE); the numbers do not reflect the cost structure at FRCSW or any need for additional tanks. However, because nCoP is so much more efficient than hard chrome plating, plating times are typically four hours for nCoP versus 24-30 hours for hard chrome. This makes it possible in principle for a single nCoP tank in a 3-shift operation to process up to five or six times the throughput of an EHC tank (or equivalently the throughput of 5 or 6 EHC tanks), depending upon setup time, etc.

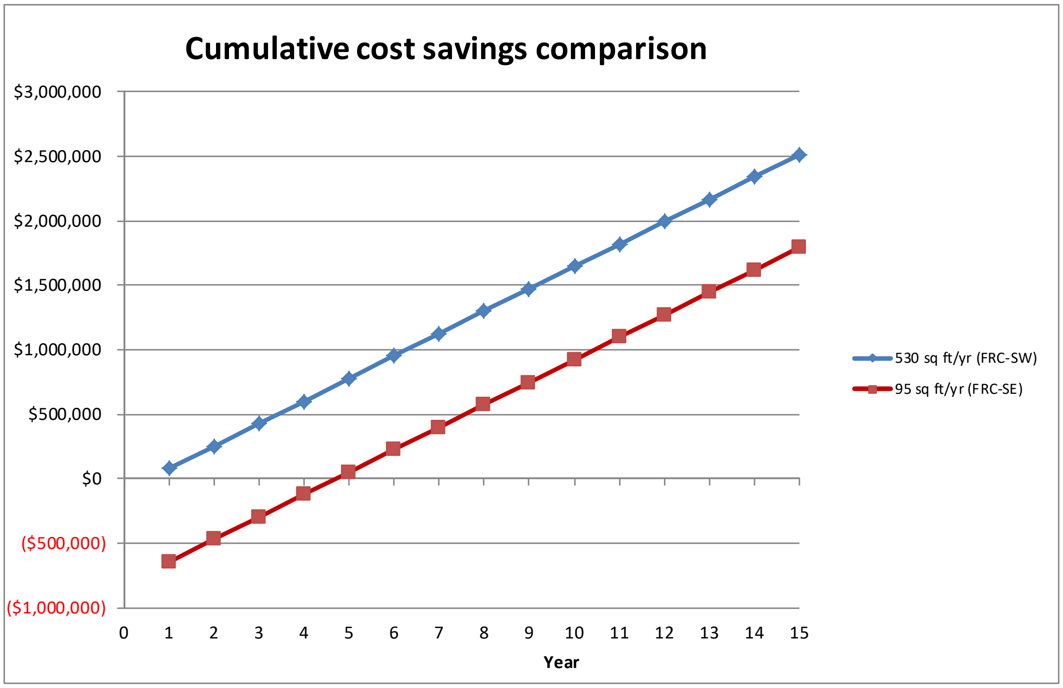

Figure 7-2. Comparison of Cumulative Cost Savings for nCoP Implementation at a Low-volume Depot (such as FRCSE) and High-volume Depot (such as FRCSW).

7.1.2 Optimum Method of Adoption

Since there is no performance saving from a switch to nCoP, there is nothing to be gained from stripping hard chrome and replacing it with nCoP outside normal depot overhaul cycles. There is also no environmental saving in stripping hard chrome to replace it with nCoP. Therefore, the optimum method of adoption is to use nCoP in place of hard chrome only on components that would otherwise be chrome plated during normal depot overhaul cycles.

Should FRCSE put into effect an overall plan to eliminate all Cr^{6+} from the depot in order to eliminate the million-dollar annual cost of OSHA Cr^{+6} compliance, replacement of hard chrome plating will be an essential component of Cr^{+6} elimination.

7.1.3 Summary

A cost benefit analysis was performed to determine the expected payback period if nCoP replaces the current FRCSE workload. A payback period of 4.7 years was determined. A substantial improvement in payback is expected by increasing workload as a result of the increased throughput obtained due to the high plating rate and lower energy consumption. Furthermore, a reduced plating shop infrastructure is possible to support an equivalent workload of EHC plating and elimination of hazardous materials (chromic acid volume reduction, lead anodes, etc.)

8.0 IMPLEMENTATION ISSUES

The following section aims to address implementations issues in order to facilitate future installations of nCoP at DoD facilities.

8.1 OPERATING TEMPERATURE & MASKANT

The Nanovate nCoP electroplating process operates at a nominal temperature of 185°F (85°C) as compared to chromium electroplating solutions that typically operate at temperatures of 140°F (60°C). This relatively higher operating temperature affects the ability to utilize conventional electroplating wax formulations for selective electroplating. Due to the high temperature, the use of thermoplastic masking materials that cure via solvent evaporation is required. These materials are difficult to work with due to thinning, contain high levels of VOCs, and are difficult to remove following electroplating. This issue was further confirmed during Dem/Val processing of the T-45 Arresting Gear Pivot during which production electroplaters commented on the need for ease-of-use improvements to selectively electroplate.

High temperature waxes are currently available as an alternative to thermoplastic maskants. However, these materials will require additional evaluation for suitability of use as an electroplate maskant. Evaluations should consider ease-of-use as primary criteria for acceptable implementation. At the time of writing this report, one product was identified for preliminary investigation: Protecto Wax HM, manufactured by The Darent Wax Company.

8.2 PULSE WAVEFORM

The nCoP process requires pulse waveform engineering in order to obtain the nanocrystalline microstructure and resultant mechanical properties. A regulated pulse power supply is required to produce a low frequency square waveform with maximum ripple of 5% at 75% output.

This process control technology is not common within most industrial electroplating facilities and thus requires retrofitting existing process lines with new pulse waveform rectifier technology. Currently, pulsed waveform generation is produced using power supplies manufactured by Dynatronix, Inc.

8.3 HARDNESS & ABRASIVE WEAR

The nCoP is able to achieve relatively high hardness through development of a nanostructured grain size (<20 nm) along with the ability to perform precipitation hardening heat treatments following electroplating. Hardness values produced from standard heat treatment protocols associated with hydrogen embrittlement baking are less than values typically obtained from hard chromium electroplating.

While hardness is not a formal design requirement, it is often used to correlate wear performance of a material. The nCoP has demonstrated equal or improved wear performance as compared to EHC with exception of Taber wear. Although the coating has been used in abrasive environments, further tribological studies are recommended for evaluation of specific applications that may not be represented within the JTP for WP-200936.

Another approach to improve abrasive wear performance is by incorporation of hard ceramic particles. Integran has developed the Nanovate nCoP-X coating process, which behaves similarly to Nanovate nCoP in terms of sliding wear and corrosion performance, while obtaining Taber wear resistance equivalent to EHC. Hard particles are co-deposited from an electrolyte with suspended particles. While other composite electroplating systems are industrially available, the Nanovate nCoP-X has been designed to obtain consistent hydrogen embrittlement and salt spray corrosion performance by controlling presence of micro-porosity. Another novel approach to obtaining nano sized particles within an electroplating matrix is use of a sol-type additive to produce particles in-situ during the electroplating process. The technology was developed by Cirrus Nano Coatings from University of Auckland, NZ.

8.4 FATIGUE PERFORMANCE

Fatigue testing was performed using a testing protocol developed between Boeing and NAVAIR. This protocol was developed to obtain material performance data as a comparison to EHC while considering cost and schedule as independent variables. Testing frequency and load reversals were fixed at 20 Hz and R = -1, respectively, for a sample set consisting of 16 shot peened coupons that were evaluated over four load conditions. While sufficient for evaluating relative technical performance, this dataset does not provide enough statistical information for NAVAIR General Authorization of all fatigue critical aerospace components. Additional mechanical testing may also be required. These requirements are under review by NAVAIR stakeholders.

8.5 MITIGATION

MIL-DTL-32502 electrodeposits produced using the nCoP process have demonstrated sufficient material performance for consideration as an alternative to hard chromium electroplating. While awaiting general authorization, implementation of nCoP should be performed on a part by part basis for components approved by the cognizant component or system design engineer. Additional testing will be considered in support of general authorization once supplemental testing requirements are identified. A transition path is also possible utilizing electroforming processing techniques developed under SERDP Project WP-2137. This project successfully developed nCoP electroformed bushings as a copper-beryllium replacement. Applications also exist for the manufacturing of repair bushings for aluminum aircraft structure. Follow-up galvanic corrosion evaluations are in progress for consideration as a substitute for cadmium electroplated stainless steel materials.

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