



FINAL REPORT

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

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**Ruben A. Prado and John E. Benfer
Naval Air Systems Command**

**Neil Mahalanobis
Integran Technologies, Inc.**

**Keith Legg
Rowan Technology Group**

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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 LOS and NLOS 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 Dem/Val 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 gravimetry 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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LIST OF ACRONYMS AND SYMBOLS

AMEC	Aerospace Materials Engineering Committee
ASETS	Advanced Surface Engineering Technologies for a Sustainable Defense
ASTM	American Society for Testing and Materials
CMAT	Calculation for Material Alternative Technologies
CTC	Concurrent Technologies Corporation
CTE	Coefficient of Thermal Expansion
DC	Direct Current
DEM/VAL	Demonstration and Validation
DoD	Department of Defense
DOE	Design of Experiment
ECAM	Environmental Cost Analysis Methodology
EHC	Engineering Hard Chrome
EPA	Environmental Protection Agency
ESOH	Environment, Safety and Occupational Health
ESTCP	Environmental Security Technology Certification Program
FRCSE	Fleet Readiness Center Southeast
GSE	Ground Support Equipment
GTE	Gas Turbine Engine
HASP	Health and Safety Plan
HCAT	Hard Chrome Alternatives Team
HCP	Hexagonal Close-Packed
HT	Heat Treatment
hex-Cr	Hexavalent Chromium
HVOF	High Velocity Oxygen Fuel
ICP-MS	Inductively Coupled Plasma Mass Spectrometer

ID	Internal Diameter
IDLH	Immediately Dangerous to Life and Health
JG-PP	Joint Group on Pollution Prevention
JIS	Japanese Industrial Standards
JTP	Joint Test Protocol
LOS	Line-Of-Sight
LVS	Logistic Vehicle System
MSDS	Materials Safety Data Sheet
NADEP JAX	Naval Air Depot Jacksonville
NAS	Naval Air Station
NAVAIR	Naval Air Systems Command
nCoP	Nanocrystalline Cobalt-Phosphorus
NDI	Non-Destructive Inspection
NESDI	Navy's Environmental Sustainability Development to Integration
NESHAPS	National Emissions Standards for Hazardous Air Pollutants
NFS	Notch Fracture Strength
NIOSH	National Institute of Occupational Safety and Health
NLOS	Non-Line-Of-Sight
OCP	Open Circuit Potential
OD	Outside Diameter
OEM	Original Equipment Manufacturer
ONR	Office of Naval Research
OP	Optimization Procedure
OSD	Office of the Secretary of Defense
OSHA	Occupational Safety and Health Administration
PEL	Permissible Exposure Limit
PEWG	Propulsion Environmental Working Group

QA	Quality Assurance
RBU	Rear Body Unit
RCRA	Resource Conservation and Recovery Act
REL	Recommended Exposure Limit
SAE	Society of Automotive and Aerospace Engineers
SERDP	Strategic Environmental Research and Development Program
SRC	Service Removal Component Card
S/N	Stress Versus Cycles-To-Failure (Related to Fatigue Data)
TO	Technical Order
TS	Thermal Spray
TWA	Time Weighted Average
UTS	Ultimate Tensile Strength
VHN	Vickers Hardness Number
WC	Tungsten Carbide
XPS	X-ray Photoelectron Spectroscopy
XRD	X-Ray Diffraction

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Mr. Richard Polinsky, Ms. Luzmarie Youngers, Mr. Peter Sheridan, Ms. Mikaleen Morrell, NAVAIR Jacksonville

Mr. Steve Brown and Mr. Craig Matzdorf, NAVAIR Patuxent River

Ms. Denise M. Aylor, NSWCCD

Mr. Michael Firth, NAVAIR Lakehurst

Mr. Robert Kestler, NAVAIR Cherry Point

Dr. Keith Legg, Rowan Technologies, Inc.

Mr. Neil Mahalanobis, Integran Technologies, Inc.

Principal Investigators:

Mr. Ruben A. Prado
NAVAIR Jacksonville

Mr. John E. Benfer
NAVAIR Jacksonville

1.0 EXECUTIVE SUMMARY

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. During operation, chrome plating tanks emit a hexavalent chromium 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 EPA emissions standards and OSHA permissible exposure limits (PEL).

Nanocrystalline cobalt-phosphorus (nCoP) is an environmentally benign alternative to engineering hard chrome plating (EHC). The nCoP coatings are applied to both LOS and NLOS surfaces using similar electroplating processes. Previous SERDP and ESTCP programs demonstrated nCoP possessed excellent corrosion and sliding wear resistance. The process was demonstrated to readily integrate within existing electroplating infrastructure present in US 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.

This report presents the results of the demonstration and validation program to evaluate nCoP including 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 NAVAIR 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 OEM (Pratt & Whitney Canada, Heroux-Devtek, Messier-Bugatti-Dowty and Boeing), the DoD maintenance depots (NAVAIR FRCSE, NAVAIR FRC-E), DoD engineering authority (NAVAIR PAX, NAVAIR Lakehurst), NAVSEA (through leveraged support funded by NESDI), as well as the Integran Technologies, Inc. and Rowan Technology Group.

This program involved studies on the following substrate materials:

- Aerospace steel (4130, 4340, Hy-tuff, 15-5PH, IN718, Aermet100)
- Generic steel (Low and Medium Carbon Steel)
- Copper alloys (Copper Beryllium, Aluminum Nickel Bronze, 70-30 Copper Nickel)
- Aluminum alloys (AA7075-T6)

All of the testing was conducted in accordance with the JTP approved by ESTCP. Where applicable, minor deviations are noted with rationale. The type of testing conducted 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.

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

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 20Hz (sine wave). Based on consultation with The Boeing Company, 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 nickel underlayer, where there was a fatigue debit observed at low loads relative to EHC and nCoP coatings tested at 130ksi load levels for 0.003" coating thickness. Note that this fatigue debit was assessed qualitatively. No statistical correlation was observed.

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 (KSC). 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 XPS 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 (OCP) 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 $-0.47\text{V}_{\text{SCE}}$ and $-0.60\text{V}_{\text{SCE}}$ respectively, after 7 days of monitoring. For reference, the measured potential for 4130 steel substrates was $-0.77\text{V}_{\text{SCE}}$ 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.

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 200hrs. 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 200hrs. 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 (i.e., 0.003” and 0.010”) in DI water and synthetic seawater. Testing showed equivalent performance between nCoP and EHC. For a coating thickness of 0.003” in DI water, samples passed 150 hrs 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.

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 WC-Co, which is currently approved and widely used for chrome replacement on aircraft.

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.

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 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 M9ACE Armored Combat Earthmover 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 coating applied to the cam surface on the T-45 Goshawk aircraft. An inspection was completed following 72 and 97 arrestments (825 ± 15 flight hrs.), showing no significant signs of wear. The component was therefore re-installed on aircraft. Following the most recent update from T-45 engineering, the nCoP component reached 116 arrestments. Component level approval is pending review of JTR results.
- A lifting arm pin was installed with nCoP coating on-board CVN-75 USS Harry S Truman on the A/S32A-32 Aircraft Towing Tractor also known as Spotting Dolly. Over the duration of the demonstration, 672 aircraft movements were completed. An NDI inspection was completed successfully following 91 days. A fleet saving of approximately 2.5 man hrs/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., Cd plated). An approval memo was written by the field activity.
- A hydraulic cylinder was installed with nCoP coating on M9 ACE located at Panama City. The component was assembled and pressured tested at the Marine Corps Depot, 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 yrs 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.)

The successful implementation of nCoP will require some further work to address implementation issues, which include identification of appropriate masking materials that can survive the elevated operating temperature of the process. Although a commercially available peelable maskant has been used successfully, traditional wax-based masking materials, which are employed heavily within the DoD, must be identified. Additionally, the use of pulse waveform necessitates the procurement of specialized rectifiers. Based on poor Taber wear performance, abrasive wear applications may require additional field tests by cognizant engineering groups to determine performance on a component level. Additional material testing and characterization data on fatigue performance is recommended 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. 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.

2.0 INTRODUCTION

2.1 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, with hexavalent chromium (Cr^{6+}) being a known carcinogen having 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 EPA emissions standards and OSHA permissible exposure limits (PEL).

A significant lowering of the Cr^{6+} PEL would likely have the greatest cost impact on military and commercial repair facilities. Such a change had been expected since the mid 1990's. In anticipation of this change, in 1995 a Navy/Industry task group [1], under the coordination of the Naval Sea Systems Command, studied the technical and economic impact of a reduction in the Cr^{6+} PEL. At the time, a reduction in the 8-hour time-weighted average (TWA) from the existing $52 \mu\text{g}/\text{m}^3$ to between 0.5 and $5.0 \mu\text{g}/\text{m}^3$ was being considered. The Navy/Industry task group performed the following tasks:

- Identified the manufacturing and repair operations, materials and processes that are used in Navy ships, aircraft, other weapons systems and facilities where worker exposure to Cr^{6+} would be expected
- Developed data on current worker exposure levels to Cr^{6+} using OSHA Method 215
- Estimated the technical and economic impact of the anticipated reductions in Cr^{6+} exposure on Navy ships, aircraft, other weapons systems and facilities
- Identified future actions required to comply with the anticipated PEL reductions

The following operations within the Navy were identified as having the potential for exposing workers to Cr^{6+} :

- Metal cleaning (including abrasive blasting and grinding) of chromate-coated materials
- Electroplating of chromium
- Painting and application of chromate paints and coatings
- Welding, thermal spraying and thermal cutting

The following conclusions were reached by the task group:

- Regulated areas for Cr^{6+} would need to be created in much greater numbers than have been required for cadmium or lead exposure.

- Local exhaust ventilation, which is the presently available engineering control, is not completely effective in reducing exposure to below $0.5 \mu\text{g}/\text{m}^3$ for many operations, or even below $5 \mu\text{g}/\text{m}^3$ in some cases.
- The inability of engineering controls to consistently reduce worker exposure below the anticipated PEL levels would significantly increase the use of respirators.
- The costs of reducing the Cr^{6+} PEL would include costs for training, exposure monitoring, medical surveillance, engineering controls, personal protective equipment, regulated areas, hygiene facilities, housekeeping and maintenance of equipment. There would also be costs due to reduced efficiency of not only the operations involving Cr^{6+} , but adjacent operations and personnel as well.
- The estimated costs for compliance with a PEL of $0.5 \mu\text{g}/\text{m}^3$ at Navy facilities include an initial capital cost of about \$22,000,000, with annual costs of about \$46,000,000 per year.
- The estimated costs for compliance with a PEL of $5.0 \mu\text{g}/\text{m}^3$ at Navy facilities include an initial capital cost of about \$3,000,000, with annual costs of about \$5,000,000 per year.
- In addition to the greatly increased cost that would be associated with chrome plating, turnaround times for processing of components would be significantly increased as well, impacting mission readiness.

Based on the projections of the metal finishing industry and the study conducted by 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 $5 \mu\text{g}/\text{m}^3$ on 28 February 2006. The actual costs for compliance for FRCSE Jacksonville, include an initial capital cost of \$1,454,749, with annual costs of \$1,153,697 per year.

On April 8, 2009, the Office of the Secretary of Defense (OSD) released a memo [2] restricting the use of Cr^{6+} unless no cost-effective alternatives with satisfactory performance were available. The effect of this will be to force adoption of Cr^{6+} free coatings and production methods which would go above and beyond established hazardous materials management protocols.

Previous research and development efforts [3,4] had established that high-velocity oxygen-fuel (HVOF) thermal spray coatings are the leading candidates for replacement of EHC. Using commercially available thermal spray systems, HVOF thermal spraying can be used to deposit both metal alloy and ceramic/metal (e.g., WC/Co) coatings that are dense and highly adherent to the base material. These coatings may also be applied to thicknesses in the same range as EHC plating.

In order to conduct the advanced development work required for qualification of the HVOF coatings, the Hard Chrome Alternatives Team (HCAT), principally sponsored by the Environmental Security Technology Certification Program (ESTCP), was established in 1996. Since EHC was being used on a wide variety of aircraft components, it was realized that it would be impossible to develop one test plan or conduct one series of tests that would address all materials and component qualification requirements. It was therefore decided to develop separate projects related to categories of aircraft components onto which EHC was being used. At the same time, the DoD Joint Group on Pollution Prevention (JG-PP) decided to partner with the HCAT on development and execution of the various projects. JG-PP is chartered by the Joint

Logistics Commanders to coordinate joint service pollution prevention activities during the acquisition and sustainment of weapons systems. It was jointly determined by the HCAT and JG-PP that projects would be executed for landing gear, propeller hubs, hydraulic actuators and helicopter dynamic components. In addition, the HCAT partnered with the Propulsion Environmental Working Group (PEWG) to execute a project to qualify HVOF coatings as an EHC replacement on gas turbine engine (GTE) components. The landing gear, propeller hub, and GTE projects have now been completed with extensive materials testing generally showing that HVOF coatings such as WC/17Co demonstrate performance superior in fatigue, wear and corrosion to EHC coatings. Rig and flight tests on WC/17Co-coated components showed acceptable performance for the HVOF coatings and, in many cases, superior performance to what would be expected had the components been coated with EHC. As a result of these projects, HVOF is being implemented at a number of Air Force and Navy repair facilities for processing of landing gear, propeller hub, and GTE components.

However, HVOF is a line-of-sight process and the angle of deposition is limited to 45 degrees. Therefore, on the internal diameters (ID) of cylinders, for example, coatings can only be applied to a depth equivalent to the diameter of the cylinder (e.g., for a 6" ID cylinder, coatings can be applied up to a depth of 6" insides the cylinder). Evaluations of the application of EHC on many different types of aircraft components at DoD repair depots showed that between 20-25% of all EHC applications were not amenable to HVOF. With the ultimate goal of eliminating all EHC plating from DoD operations, SERDP sponsored three different projects to investigate alternative technologies that would be capable of replacing EHC on all types of geometries including internal surfaces. These were (1) electrospark deposition, (2) miniature gun plasma spray, and (3) electroplating of nanocrystalline Co alloys. The projects were completed in late 2003 and an assessment of the results from the projects indicated that electroplated nanocrystalline cobalt phosphorus (nCoP) had the broadest capability for replacing EHC on all types of components and that it was the most ready for demonstration/validation [5]. The nCoP alloys were produced using an electrodeposition process similar to EHC. This allows for the retention of numerous benefits associated with hard chrome coating technology (i.e., non- line-of-sight application, excellent coating adhesion, dimensional consistency and superior surface finish) and leverages existing hard chrome plating infrastructure. The nCoP alloys exhibited material properties in many cases superior to EHC. As a result, a proposal was submitted to ESTCP for a demonstration/validation program and the project (WP-0411) was approved for a 2004 start. At the conclusion of the project in 2007, several technical risks were identified. In 2009, supplemental work was successfully completed in order to optimize process parameters and address technical risks. In 2009, a demonstration/validation program (WP-0936) was approved by ESTCP for a 2009 start.

2.2 OBJECTIVE OF THE DEMONSTRATION

The goals of this project were to demonstrate and validate electrodeposited nCoP alloy coatings as a technically feasible and commercially viable replacement for EHC on multiple military systems. Table 2-1 lists the target hazardous materials across the DoD. 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 Fleet Readiness Center South East Jacksonville (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
- Developing standards, specifications and technical documentation providing procedures and process controls for application and removal of nCoP electroplated coatings

The environmental and worker safety advantages of the nCoP technology lie in the fact that it does not utilize Cr^{6+} , nor does it use nickel, which is undergoing increasing regulatory scrutiny. From a technological standpoint, all present-day depot (and most commercial) electroplating processes use direct current between cathode and anode. The nCoP technology utilizes pulse plating to control the intercrystalline content of the coating material and thus create a defined nanocrystalline grain structure. The microstructural control is enabled by controlling a combination of chemical and electrical parameters, to enable tailoring of certain material properties. More specifically, pulse control allows the optimal ratio of grain nucleation and growth, which determines the final grain size of the material. Grain size controls dislocation motion, and with it the mechanical properties of the material such as tensile strength, hardness and ductility. The Hall-Petch strengthening in nCoP makes this nanocrystalline material harder and more wear-resistant.

Additionally, the technology is based upon traditional electroplating and is therefore a direct drop-in for the existing EHC process and is able to be incorporated into existing plating lines, although it will require replacement of plating power supplies and inert tank liners. It is anticipated that this will facilitate the implementation of the technology.

Table 2-1. Target Hazardous Material (HazMat) Summary.

Target HazMat	Current Process	Applications	Current Specifications	Affected Programs	Candidate Parts and Substrates
Hexavalent Chromium	Hard Chromium Electroplating	▪ Landing Gear ▪ Arresting Gear ▪ Hydraulic Cylinders ▪ Actuators ▪ Dimensional Restoration	AMS-QQ-C-320 AMS-2460 DoD-STD-2182 MIL-STD-1501 MIL-C-20218	T-45 GSE M9ACE	▪ Arresting Tail Hook, T-45 Pivot Assembly ▪ Lifting Pins, GSE Spotting Dolly ▪ Hydraulic Cylinder, MarineCorps M9 ACE

2.3 REGULATORY DRIVERS

Gaseous emissions from EHC plating operations must conform to the National Emissions Standards for Hazardous Air Pollutants (NESHAPS) and any solid or liquid waste generated from EHC (such as plating sludge) must be disposed of as hazardous waste in accordance with Resource Conservation and Recovery Act (RCRA) regulations. The costs associated with compliance with these regulations are minor compared to the overall costs of the plating process.

By far the largest regulatory cost driver for EHC plating is the Cr^{6+} PEL as established by OSHA. For many years a lowering of the PEL was expected. But it was only in 2004 that the agency began the process to issue a new PEL as a result of a lawsuit filed in 2002 by a citizens group and union that petitioned OSHA to issue a lower PEL, and a subsequent ruling by a Federal District Court upholding the petition. The court ruling required OSHA to publish a new draft Cr^{6+} PEL in the Federal Register no later than October 2004. On October 4, OSHA proposed a new PEL of $1 \mu\text{g}/\text{m}^3$ with a $0.5 \mu\text{g}/\text{m}^3$ action level, which represented a significant reduction from the then PEL of $52 \mu\text{g}/\text{m}^3$. In addition to the reduction in the Cr^{6+} PEL, the rule also included provisions for employee protection such as preferred methods for controlling exposure, respiratory protection, protective work clothing and equipment, hygiene areas and practices, medical surveillance, hazard communication, and record-keeping. The expected one-time compliance costs, as determined by OSHA, in all industries including electroplating, welding, painting and chromate production, was \$226 million, although the surface finishing industry expected that the costs would be substantially higher. There would also be increased annual recurring costs associated with health monitoring, record-keeping, etc. On 28 February 2006 the final rule was promulgated at $5 \mu\text{g}/\text{m}^3$ [6]. Using the cost estimates performed by the Navy in 1995, it is clear that the additional annual costs to DoD will be at least several million dollars.

On 9 April 2009, a memo from the 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.

2.4 DEMONSTRATION TECHNOLOGY

2.4.1 Technology Description

The first systematic studies on the synthesis of nanocrystalline materials by electrodeposition, in an attempt to optimize certain properties by deliberately controlling the volume fraction of grain boundaries in the material, were published in the late 1980's [7,8]. The general conditions for producing nanocrystalline metals and alloys by electrodeposition are documented in US Patent No. 5,352,266 Oct. 4, 1994 and US Patent No. 5,433,797 July 18, 1995. The synthesis of nanocrystalline materials with grain size control during the electrodeposition process can be considered a distinct form of grain boundary engineering, in which the grain boundary content of

a material is controlled during material processing to achieve certain physical, chemical and mechanical properties. The result is thus a bulk interfacial material that does not require any further processing of precursor powder material. In this respect, electrodeposited nanocrystalline materials are quite different from other nanostructures that are based on consolidated particles.

A very large number of pure metals, alloys, composites and ceramics can be electrodeposited or co-electrodeposited with grain sizes less than 100 nm. These include pure metals (Ni, Co, Pd, and Cu), binary alloys (Ni-P, Ni-Fe, Zn-Ni, Pd-Fe, Co-W) and ternary alloys (Ni-Fe-Cr). Even multi-layered structures or compositionally modulated alloys (Cu-Pb, Cu-Ni, Ag-Pd), metal matrix composites (Ni-SiC), and ceramics (ZnO) have been successfully produced by electrodeposition methods.

Electrocrystallization occurs by either the buildup of existing crystals or the formation of new ones. These two processes are in competition with each other and are influenced by different factors. The two key mechanisms that have been identified as the major rate determining steps for nanocrystal formation are charge transfer at the electrode surface and surface diffusion of adions on the crystal surface. One of the key factors in the microstructural evolution of electrodeposits in terms of grain size and shape is inhibition. The effectiveness of grain refiners depends on surface adsorption characteristics, compatibility with the electrolyte, and temperature stability.

The second important factor in nanocrystal formation during electrocrystallization is overpotential. Grain growth is favored at low overpotential and high surface diffusion rates. On the other hand, high overpotential and low diffusion rates promote the formation of new nuclei. These conditions can be experimentally achieved when using pulse plating, where the peak current density can be considerably higher than the limiting current density attained for the same electrolyte during direct current (DC) plating. Figure 2-1 illustrates this, with the top illustration showing grain growth during conventional DC plating and the bottom illustration showing nucleation of new grains resulting in nanocrystallites [9].

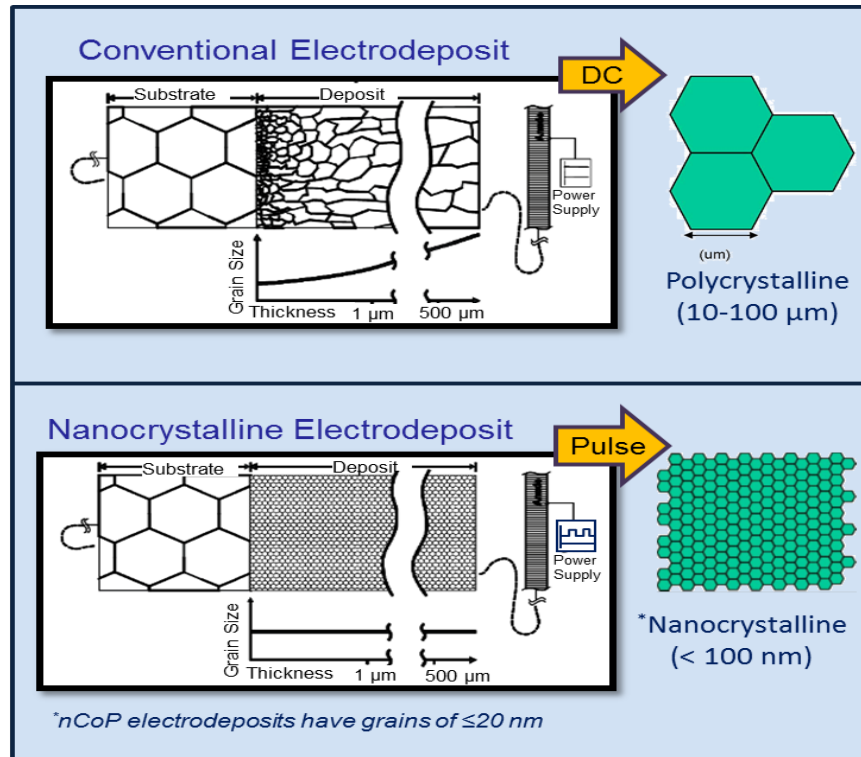


Figure 2-1. Illustration of grain growth (top) during conventional DC plating and nucleation of new grains (bottom) during pulse plating.

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 [10]. In this application, a nanocrystalline Ni-P microalloy with a grain size of approximately 100 nm is electrodeposited on the inside surface of steam generator tubes to a thickness that ranges from 0.5 to 1 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 Technologies, Inc has successfully developed Nanovate™ and GBE® products for a variety of commercial applications. The GBE® (grain boundary engineering) platform refers to materials with a similar nanostructure grain size built from a “top-down approach” using thermo-mechanical processing techniques. GBE® is a patent protected thermomechanical process (forming + heat treatment), where the internal structure of conventional metals and alloys is optimized locally on a nanometer scale to yield breakthrough improvements in material reliability, durability and longevity [11,12]. The aim is to create many more “special grain boundaries” which are more stable and can resist “creep cracking”, one of the main failure modes for the material. The most pronounced improvement in properties occurs for high temperature alloys, such as exotic nickel based alloys that are used for high temperature portions of turbines. Other applications that take advantage of this effect include lead-acid battery grids and shaped charge liners for military and industrial 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 nanometer scale. They are applied as fully dense coatings or freestanding forms.

The Nanovate™ product platform includes:

- Nanovate™ NS is an extremely strong and resilient nanocrystalline coating that protects carbon composites from impact and erosion [13]. Nanovate™ NS technology is featured in several commercially available products including: Colt Hockey Stick, Epic™ Golf Shaft, PING iN & JAS Series Putter inserts, Anderson Bat Company’s Nanotek Bat Series, HEAD Metallix line of raquets. Nanovate™ NV is an award winning, low CTE (coefficient of thermal expansion), hard nanocrystalline coating designed to increase the durability of Carbon Composite/Tooling Molds. [14,15,16,17,18].
- Nanovate™ EM is a nanocrystalline ferromagnetic coating that provides low frequency magnetic shielding for sensors and other sensitive electronics. Unlike other EM coatings, electrodeposited coatings can be applied to various geometries. When applied to polymer based substrates, the high strength nanometal imparts improved strength and stiffness to the part [19]. Integran’s Nanovate™ EM coating for low frequency magnetic shielding has been used in numerous defense applications.
- Nanovate™ NP is a hybrid of high strength nanocrystalline metal coating and injection molded polymer that has the flexibility of injection molding, with the strength and rigidity of lightweight metals. Several polymer substrates including PEEK, ABS and polyamide can be coupled with nanometal to produce lightweight hybrid materials [20,21].
- 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 hexavalent hard chrome solutions [22,23,24]. 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 demonstration/validation process line for repair/overhaul of engine components.

Figure 2-2 shows some examples of the applications of the Nanovate™ product line. In addition to the commercially ready Nanovate™ technology platforms, Integran and its subsidiaries are engaged in product research and development activities, including SERDP project WP-2137 “Nanostructured copper alloys as an alternative to Copper Beryllium” and SERDP project WP-1616 “Ultra-High Efficiency/Low Hydrogen Embrittlement Nanostructured Zinc-Based Electrodeposits as Environmentally Benign Cadmium-Replacement Coatings for High-Strength Fasteners.”

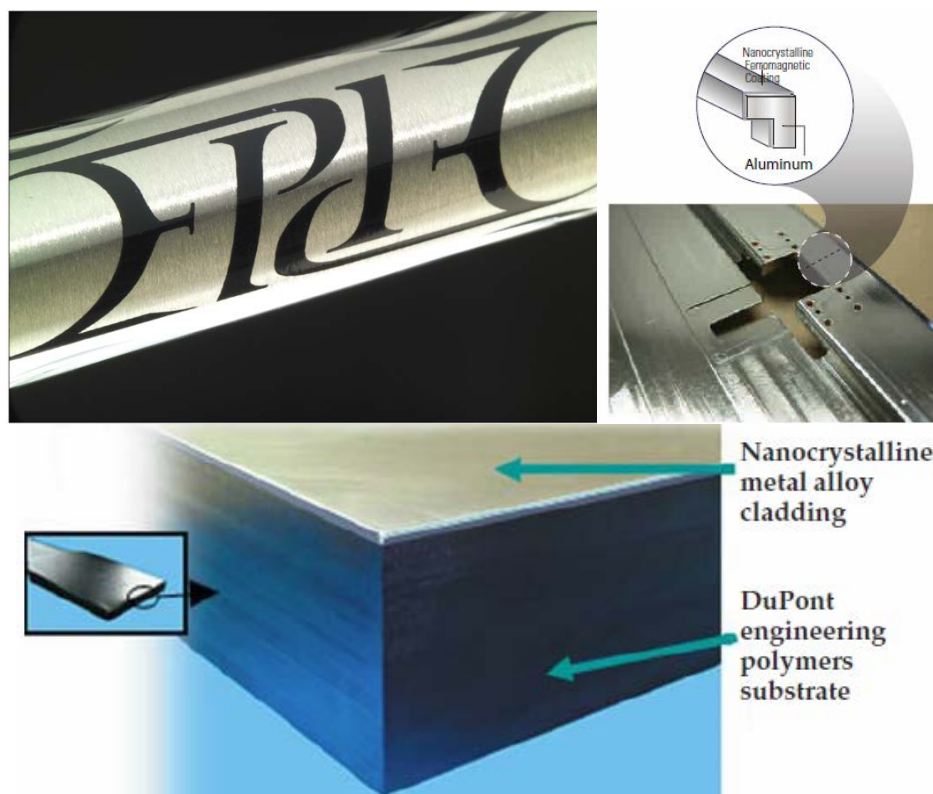


Figure 2-2. Nanovate™ NS applied to the Epic Golf Shaft (top left), Nanovate™ EM coated on an aluminum housing (top right), Nanovate™ NP demonstrating the application of a nanometal on an engineering polymer substrate (bottom).

2.4.2 Technology Development

2.4.2.1 SERDP Development Work

The nCoP coatings to be evaluated in this project were developed under SERDP Project PP-1152 [5]. With the structure-property relationships having been developed for many nanocrystalline materials, the main objective of the SERDP project was to “engineer” the microstructure of electrodeposited cobalt-based coatings to optimize the relevant properties such that the performance of the coating met or exceeded that of EHC.

The nCoP electrodeposits developed during the SERDP project were produced from a plating solution containing: Co^{2+} ions, Cl^- ions, a buffer and a source of P ions. Electrodeposition parameters (e.g., bath composition, pH, temperature, overpotential, bath additives, etc.) were

modified to optimize the microstructure of the coating for property enhancement (hardness, wear, corrosion resistance, etc.). It was determined that coatings could be deposited with phosphorus concentrations ranging from 1 to approximately 10 weight percent (wt%). The overall plating efficiency of the process was determined to be approximately 90%, with a deposition rate ranging from 50 μm (0.002") to 200 μm (0.008") per hour, depending on current density. Consumable (cobalt pieces in a titanium basket) anodes could be used for the deposition process, allowing for easy application to internal diameter surfaces. The resulting nanocrystalline material showed strengthening by virtue of its small grain size (via the Hall-Petch effect) as well as incorporation of phosphorus (i.e., solid solution strengthening). Subsequent heat treatment further strengthened deposited material by a precipitation hardening mechanism. Performance tests were conducted on coated samples as well as reference materials to establish properties and performance including: hardness (ASTM B578), ductility (ASTM B489), adhesion (ASTM B571), abrasive (Taber) wear (JIS H8503), sliding (pin-on-disk) wear (ASTM G99), electrochemical potentiodynamic polarization (ASTM G5), and salt spray (fog) corrosion (ASTM B117). Hydrogen embrittlement (ASTM F519) and constant amplitude axial fatigue (ASTM E466) tests were also performed. Upon completion of the program, electrodeposited nCoP demonstrated comparable and in some cases superior performance to EHC. Additionally, application to components with complex ID and OD geometries was successfully demonstrated. The final SERDP report provides a detailed account of the process and properties of the final optimized nanostructured coating selected as the most promising replacement for EHC. Please refer to the report for all relevant performance data obtained.

2.4.2.2 ESTCP DEVELOPMENT WORK

At the conclusion of the SERDP program to develop nCoP as an alternative to EHC plating for IDs, 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, a proposal was submitted to ESTCP for a demonstration/validation program and the project (WP-0411) was approved for a 2004 start.

At the beginning of the ESTCP program (WP-0411), a 1300 L Dem/Val plating tank and pulse power supply were installed at Integran. nCoP deposits were successfully reproduced under the operating conditions defined by the SERDP work. In the course of the program, process optimization was conducted to define windows of operation, sensitivity to impurities, activation processes for different substrate materials, masking materials, ID plating processes and coating removal processes. Quality assurance procedures to monitor deposit quality were implemented and are still in use today. Test samples were produced to support Joint Test Protocol (JTP) testing.

In addition to the plating line installed at Integran, a chrome plating line was successfully converted/retrofitted to accommodate nCoP plating solution at NAVAIR Jacksonville. A pulse power supply similar to that used at Integran was first qualified by Integran, and subsequently installed at NAVAIR Jacksonville. Integran issued a process specification document to NAVAIR Jacksonville to provide instruction on the system operation. Early deposit quality issues included adhesion to low alloy steel substrates and pitting. Adhesion issues were addressed through the installation of an alternate activation solution on the plating line. Pitting was found to be caused by organic contamination from the maskant material and was resolved through carbon treatment.

However, this did not represent a permanent solution, highlighting the need for a new maskant material that was compatible with the plating solution. Both OD and ID plating capability were demonstrated on test coupons as well as demo components.

Performance testing was conducted in accordance with the Joint Test Protocol for the project. Grinding studies showed that nCoP could be ground, polished and superfinished using processes similar to those currently in use for EHC. In fluid immersion testing, nCoP was found to be compatible with most service and overhaul fluids. Rod-seal wear testing suggested that nCoP exhibited comparable wear and fluid leakage to EHC. Hardening heat treatment and low surface roughness were found to minimize leakage. Axial fatigue data produced a fatigue debit larger than EHC. However, analysis of the test specimens showed that most of the failures were at the end of the plated section which was not properly terminated (i.e. it had no coating runout), invalidating the dataset. Hydrogen embrittlement testing suggested that nCoP was embrittling to high strength steels with or without a relief bake. This was in stark contrast to prior SERDP testing which suggested that nCoP was non-embrittling and did not require the typical bakeout. Reverting to the deposition conditions (current density, pulse frequency and duty cycle) developed in the SERDP program appeared to resolve the embrittlement problem.

2.4.2.3 SUPPLEMENTAL WORK TO WP-0411

After a considerable amount of development and testing both at Integran Technologies Inc. and at NAVAIR Jacksonville, 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, a whitepaper was issued to extend the deadline of the program in order 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. The whitepaper was accepted and this supplemental work commenced in 2008.

The supplemental work focused on optimizing plating deposition parameters using a Design of Experiment (DOE) approach with hydrogen embrittlement as the primary response variable and hardness, appearance, composition, microstructure, internal stress, thickness distribution, current efficiency and adhesion as secondary response variables. Hydrogen embrittlement was found to be independent of deposition conditions, and in many cases nCoP passed testing without an embrittlement relief bake. Secondary response variables were used to down-select optimum deposition parameters for subsequent performance testing. These parameters were found to be non-embrittling with improved fatigue and neutral salt fog corrosion performance as compared to EHC. The nCoP deposit did exhibit lower hardness (560 VHN) and abrasive wear performance as compared to EHC, which is in good agreement with previously obtained data. Producibility evaluations were performed utilizing a J52 Shaft section and J52 Coupling components. A commercially available maskant was utilized and performed well. Coating deposition using optimized plating parameters was successfully demonstrated on both ID & OD areas of these parts. Please refer to Appendix for a complete embedded version of the final report (Supplemental work to WP-0411).

Table 2-3 and Table 2-3 summarize the process and properties, respectively, of nCoP in comparison to those of EHC.

Table 2-2. 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 200µm (0.008”) per hour	Up to 40µm (0.0016”) per hr
Thickness	Demonstrated up to 1000µm (0.040”)	Typically <500µm (0.020”)
As-Deposited Appearance	Pit / Pore Free / Crack free	Microcracked
Microstructure	Nanocrystalline (avg. g.s.=8-15nm)	-
Relative Process Cost	Comparable	-
Emission Analysis	Below OSHA limits	Cr ⁺⁶

Table 2-3. Property summary for nCoP compared with that of EHC.

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

2.5 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 2-4 lists the advantages and disadvantages associated with the new technology.

Table 2-4. Advantages and limitations for nCoP.

Advantages/strengths	Disadvantages/weaknesses
<i>Technical</i>	
Improved fatigue life, corrosion protection, sliding wear and embrittlement compared to EHC.	Reduced performance in abrasive wear testing.
Application of thin uniform coatings down to 0.0002" thick can be applied in contrast with EHC which must be at least 0.001" thick.	Similar to EHC, a post-plating bake-out is necessary to ensure coating is non-embrittling.
Coatings up to 0.040" thick can be produced compared to EHC which is limited to less than 0.020" thick.	Maskant material compatible with plating solution, pH and temperature must be identified although a commercial product has been tested and performed well to date.
The plating rate of nCoP is 5x faster than in EHC.	Susceptible to contaminants otherwise benign in EHC plating.
Coating microstructure is fully dense, free of pores, cracks owing to low residual stress, in contrast to EHC which is microcracked.	Ferromagnetic nature of the coating prevents the use of conventional non-destructive thickness testing.
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.	
<i>Depot and OEM</i>	
Direct drop-in technology for existing EHC infrastructure.	Necessitates the use of pulse power supplies although low cost units are currently being developed as part of the ESTCP project WP-0934 "Industrial Implementation of Environmentally Friendly Nanometal Electroplating Process for Chromium and Cadmium Replacement using Low Cost Pulse Current Power Supplies."

Energy savings as a result of higher efficiency.	Refurbishment of tank is necessary due to elevated operating temperature and lower pH.
Higher throughput resulting from increased plating rate.	Unproven technology in a full production setting although has been demonstrated on a pilot scale.
<i>Environmental</i>	
Emissions of Co are below PEL, no hexavalent chrome.	
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.	

Several alternative technologies have been identified, tested and in some cases validated for use as EHC alternative technologies [25]. Some prominent technologies which exist include: Thermal Spray (HVOF), electrodeposited Trivalent Chromium, nickel alloys and electroless nickel. Other niche technologies not discussed here include PVD, plasma spray, electrocomposites, laser cladding, electrospark deposition and explosive cladding.

The following is a brief commentary of the alternative technologies and challenges they face for implementation as EHC alternatives:

As mentioned earlier, HVOF is limited to line-of-sight applications. Several commercial trivalent chromium systems are available. They are typically unsuitable as functional coatings as a result of poor corrosion resistance necessitating pre-plate/post-plate coatings. The production of thick deposits is also a challenge. Several Nickel-based alloy coatings exist, however there are some concerns as a result of its inclusion as a hazardous material. Nickel-Tungsten alloys possess upon heat treating hardness comparable to hard chrome. However, technological maturity is required before it can be implemented. The use of electroless Ni is generally unsuitable as a result of its poor tribological performance. Maximum wear resistance can only be achieved through heat treatments in excess of the allowable temperatures for most structural steels. Issues are also surrounding deposition rate, thickness limitations, frequent dilution and replenishment of plating solution as well as adhesion reliability problems. A comparison of all alternative technologies indicates nCoP is a viable alternative with several advantages to its widespread usage.

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. 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			
<i>Deposit Testing</i>			
Thickness uniformity/ deposition rate	Microscopic	Minimum 0.002"	Pass
Coating thickness uniformity	Microscopic	nCoP coating thickness shall be uniform within $\pm 20\%$ 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: 1-2wt%P	Pass
Microhardness	Vicker's Microhardness	Hardness: nCoP $\geq$ EHC <u>1/</u> See Figure 6-5 thru 6-8. <u>Target Hardness</u> > 850 VHN (EHC requirement)	Pass on selected heat treatment conditions ^{1/}
		nCoP maximum hardness obtained 763 VHN following heat treatment 550°F for 5 hrs.	Fail
		<u>Threshold Hardness</u> > 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: HCP	Pass
Ductility	Bend test ASTM B489	As-deposited ductility > 2%	Pass
Internal Stress	Spiral contractometer	Internal stress 15 ksi maximum (tensile) <u>2/</u> Internal stress 22-26ksi. However, demonstrated ability to build coating thickness	Fail ^{2/}
Internal Stress	Copper stress strips	Internal stress 15 ksi maximum	Fail ^{3/}

		(tensile) 3/ Internal stress 15-20ksi. However demonstrated ability to build coating thickness	
<i>Performance Testing</i>			
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) (Aermet100 leveraged effort supported by Northrop Grumman Corporation)	Pass, Marginal Pass for Hy- tuff
Fatigue	Axial fatigue	S-N curve fitted data: nCoP $\geq$ EHC at 0.003" ANOVA test (For bare, nCoP- 0.003", EHC-0.003") 187 ksi: p=0.00 168 ksi: p=0.21 149 ksi: p=0.75 130 ksi: p=0.06 Two sample t-test (nCoP-0.003", EHC-0.003") 187 ksi: p=0.010 168 ksi: p=0.228 149 ksi: p=0.609 130 ksi: p=0.197	Pass
		S-N curve fitted data: nCoP $\geq$ EHC at 0.010" ANOVA test (For bare, nCoP- 0.010", EHC-0.010") 187 ksi: p=0.00 168 ksi: p=0.81 149 ksi: p=0.43 130 ksi: p=0.03 Two sample t-test (nCoP-0.010", EHC-0.010") 187 ksi: p=0.002 168 ksi: p=0.665 149 ksi: p=0.387 130 ksi: p=0.149	Pass

		S-N curve data fitted: nCoP at 0.010" $\geq$ Ni+EHC at 0.005" $\geq$ Ni+nCoP at 0.005" ANOVA test (For bare, Ni-0.002" + nCoP-0.003", Ni-0.002" + EHC-0.003") 187 ksi: p=0.31 168 ksi: p=0.00 149 ksi: p=0.00 130 ksi: p=0.00 Two sample t-test (Ni-0.002" + nCoP-0.003", Ni-0.002" + EHC-0.003") 187 ksi: p=0.269 168 ksi: p=0.776 149 ksi: p=0.001 130 ksi: p=0.159	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 $\geq$ EHC per ASTM B537	Pass
Corrosion	SO2 salt fog	Average appearance/Protection ranking vs time curve: nCoP $\geq$ EHC per ASTM B537	Pass
Corrosion	Beach exposure	Average appearance/Protection ranking vs time curve: nCoP $\geq$ 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 – HE relief bake	Hydrogen embrittlement	1a1: four bars > 200h <u>4/</u> HE relief baked nCoP failed HE testing (2 of 4 bars), however supplemental testing on repeat HE relief baked nCoP passed HE testing (8 of 8 bars)	Pass ^{4/}
Hydrogen embrittlement – no HE relief bake	Hydrogen embrittlement	load to failure: nCoP $\geq$ 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-	Pass on 8 fluids

		680, Fluorescent penetrant, Cimstar 40, Turco 4181L Alkaline Cleaner, MIL PRF 85570 type 2, Bioact 280	
		Fail: Nital Etch, Ammonium Persulfate, Chlorine Bleach	Fail on 3 fluids
Environmental embrittlement	Sustained load in saltwater environment	150 hrs+ and 45% NFS+: nCoP $\geq$ EHC in DI water nCoP $\geq$ EHC in Salt water 5/ nCoP and EHC both fail in salt water	Pass (DI water) ^{5/}
Wear	Taber abrasive (ASTM D4060)	Taber wear index: nCoP $\leq$ EHC	Fail
	Taber abrasive (ASTM F1978)	Taber wear index: nCoP $\leq$ EHC	Fail
Wear	Pin on disk	Coating wear volume loss, coefficient of friction, static partner wear volume loss: nCoP $\leq$ 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 $\geq$ EHC. However, static partner volume wear for nCoP $\ll$ 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: CoP $\leq$ EHC	Pass
Wear	Gravelometry	CoP performance equal to EHC	Pass
Wear	SATEC oscillating load	Coefficient of friction, average bushing wear: CoP $\leq$ 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 Co oxide (CoO and Co ₃ O ₄) which is dense, compact and adherent unlike red rust

NAVSEA (M9ACE)	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.

4.0 SITE/PLATFORM DESCRIPTION

4.1 TEST PLATFORMS/FACILITIES

4.1.1 Industrial Plating Facility

The Fleet Readiness Center Southeast (FRCSE) in Jacksonville, Florida 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 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 (AMEC) of the Society of Automotive and Aerospace Engineers (SAE) and will be used by all military repair depots and by manufacturers of weapons systems.

Another reason for the selection of FRCSE is that it is Lead Maintenance Technology Center for the Environment and has a prominent role in facilitating a continuous reduction of hazardous waste by changing materials and processes used in aircraft maintenance.

In 1939 the citizens of Jacksonville voted a \$1 million bond issue to purchase the site for the Naval Air Station (NAS). The Naval Air Depot began in a small way in 1940, operating as an Assembly and Repair Department of the NAS. The first N2S “Yellow Peril” was overhauled and rolled out for test in August 1941, followed closely by a newly reworked SNJ aircraft. During World War II there were 7300 people working at the NADEP, 3500 of them military.

On April 1, 1967, the Naval Air Rework Facility was commissioned as a separate command under the Commander, Naval Air Systems Command. In 1987, the name was changed to Naval Aviation Depot Jacksonville and in 2001 was renamed Naval Air Depot Jacksonville. Since 1940 the production shops have turned out almost every type of Navy aircraft—fighter and attack planes, patrol, antisubmarine, reconnaissance, transport, trainer, special configuration and helicopters. The overall workload has also expanded to include the rework of engines, components, and ground support equipment, plus other support functions vital to the Fleet. In November 2001 FRCSE received ISO 9001:2000 certification, making it the first command in NAVAIR and the Department of Defense to receive such a certification.

FRCSE currently occupies 53 buildings on over 102 acres of land bordering the St. Johns River on the NAS. It returns almost \$200 million in payroll annually to the Jacksonville community. The depot currently performs airframe and component maintenance and repair to P-3, EA-6B, E-2/C-2, F-14 and F-18 series aircraft. In addition, the depot performs all but depot level phased maintenance interval work on SH-60 series helicopters at NAS Jacksonville, NAS Mayport and NAS Norfolk. It is the premier J52, F404 and TF34 engine repair facility in the world. It has a state-of-the-art engine test cell that conforms with all state and local air pollution regulations and is capable of testing engines through the entire range of their operating envelopes.

4.1.2 Demo Systems

The following section describes the test platforms and components identified for the demonstration/validation 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).

At the heart of the training system is the two-seat, single-engine T-45 Goshawk. The aircraft has a wingspan of 30.1 feet, a length of 39.3 feet, a tail height of 13.5 feet and a gross takeoff weight of 13,636 pounds. The U.S. Navy's T-45 Goshawk is powered by a Rolls-Royce Adour F405-RR-401 engine producing 5,845 pounds of thrust. Designed to excel in the rigorous environment of naval aviation training, it has been fatigue-tested successfully to demonstrate a service life that exceeds the required 14,400 flight-hours.

Described by instructor-pilots who fly it as “eminently forgiving,” the T-45 is the only jet trainer designed to land routinely at sink rates of greater than 700 feet per minute, which are required for aircraft carrier approach landings. The Boeing team has continually upgraded the aircraft with features such as leading-edge wing slats for better low-speed performance, high-gain nosewheel steering for safer ground handling, and a reinforced composite stabilator with increased span for better pitch control.

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 [26,27].

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 demonstration/validation 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 4-4 shows the cam surface of a pivot assembly returned from service. Significant wear can be noted on the cam surface. Both EHC and sulfamate Ni coatings will be stripped using mechanical and chemical stripping techniques.

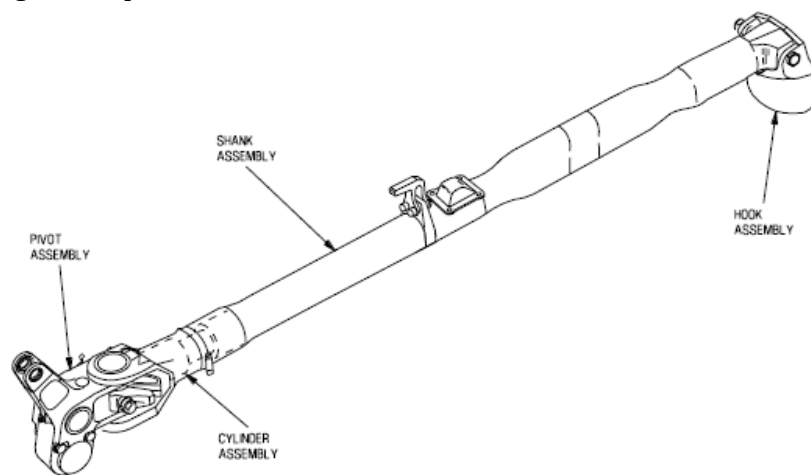


Figure 4-2. Schematic drawing of the arresting tail hook assembly.

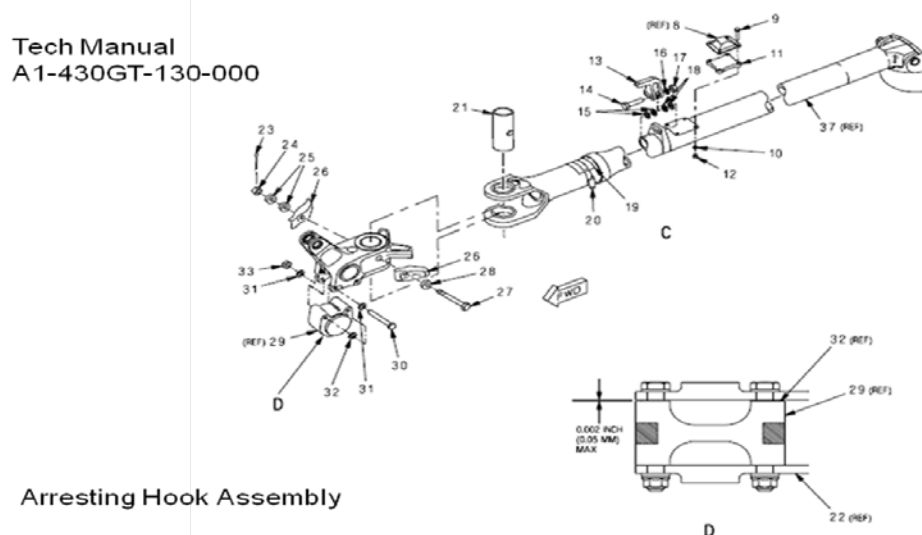


Figure 4-3. Technical manual excerpt with a detailed schematic of the arresting tail hook assembly.

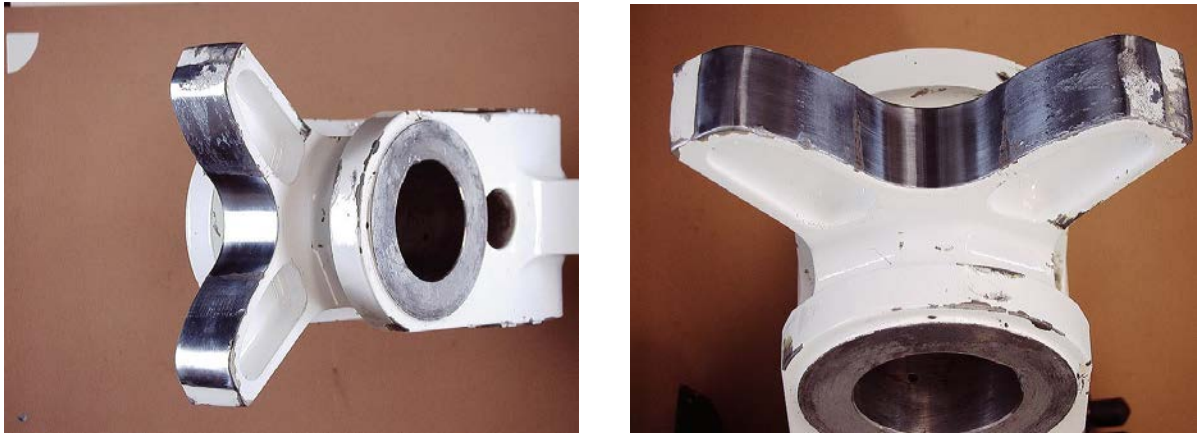


Figure 4-4. As-received cam surface of pivot assembly previously in-service.

4.1.2.2 Spotting Dolly

The A/S32A-32 Aircraft Towing Tractor (see Figure 4-5), also referred to as the “Spotting Dolly,” serves as the ground support equipment (GSE) nCoP demonstration component. Support equipment is the generic name applied to the class of equipment needed to support aircraft operations. 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. The spotting dolly positions aircraft on deck and must be compatible with all aircraft models.

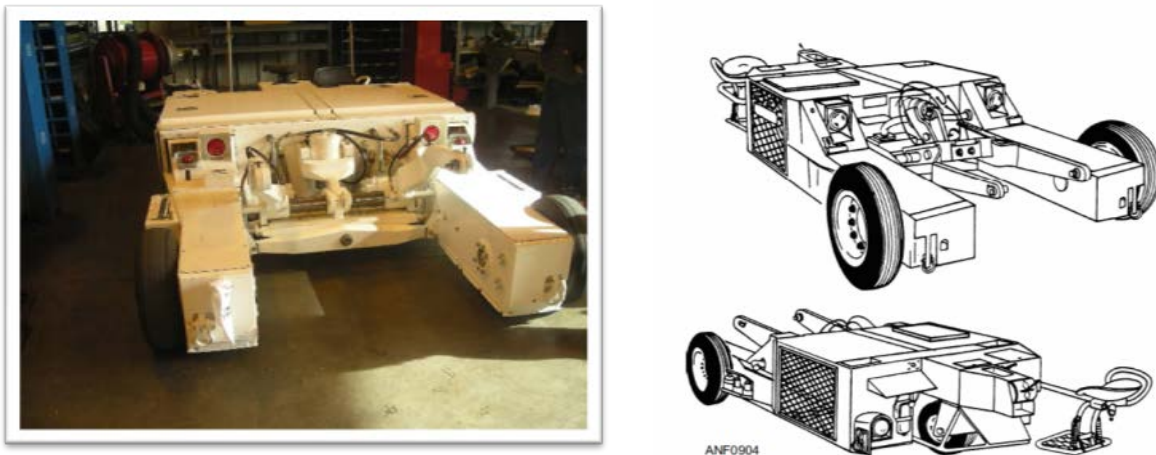


Figure 4-5. Image of the Spotting Dolly (left) and schematic drawing of the Spotting Dolly (right).

The spotting dolly is designed to tow, turn, and position various types of aircraft on the hangar deck of an aircraft carrier. Maximum maneuverability is achieved by moving the aircraft on its own main landing gear with the nose gear elevated. The tow tractor’s unique lifting linkage attaches to the aircraft’s nose wheel and physically lifts it off the ground. The tow tractor can maneuver aircraft within a zero radius.

Figure 4-6 shows a technical manual excerpt for the hydraulic system on the Spotting Dolly. The lifting linkage consists of a main telescoping hydraulic lift cylinder, two hydraulic spread

cylinders, two lifting arms, and support parts and hardware. The lift cylinder raises and lowers the lifting arms. The spread cylinders move the lifting arms in or out, keeping the arms pinned against the aircraft nose gear. Axle engagement pins, which fit a variety of aircraft nose wheels, can be attached to the lifting arms.

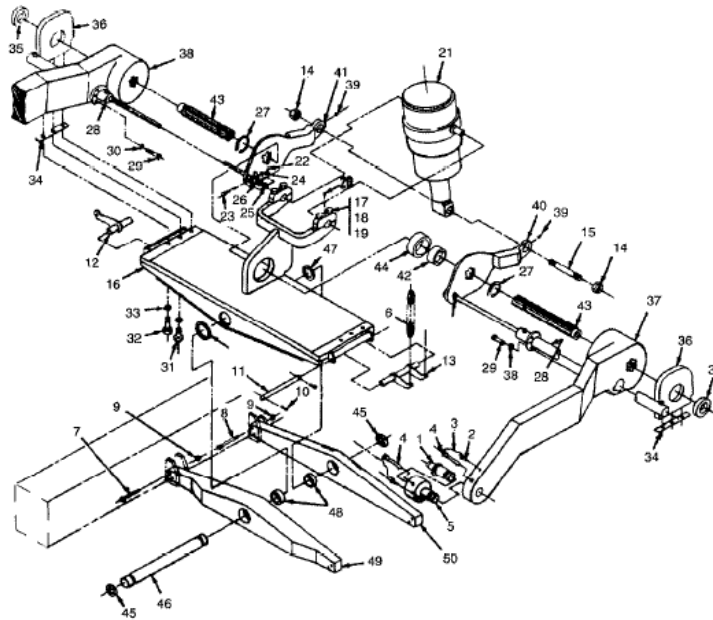


Figure 4-6. Technical manual excerpt for the hydraulic system on the spotting dolly.

The spotting dolly lifting pin (P/N: 1117AS243-1) has been selected as the component for demonstration/validation of nCoP electroplating. Figure 4-7 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 NAWC-AD (NAVAIR Lakehurst) during deployment by the vehicle's unique ID. 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 4-7. Image of lifting arm pin installed on Spotting Dolly (left) and close-up (right).

4.1.2.3 M9 ACE

The M9 Armored Combat Earthmover (ACE) is a fully tracked armored combat engineer vehicle (see Figure 4-8), that provides combat engineering support to front-line Marine 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.

This combat engineering vehicle has a welded and bolted aluminum hull structure with selected steel and aramid-laminated plates. The hull of the M9 ACE provides protection against small arms fire and artillery shell fragments. It is powered by a Cummins V903C turbocharged diesel engine. All operational M9 combat earthmovers are constantly upgraded to improve their performance, reliability, durability and readiness.



Figure 4-8. Image of M9 ACE vehicle with an inset image of the hydraulic cylinder coated with EHC plating.

In collaboration with NAVSEA (OSD & NESDI leveraged effort), a hydraulic cylinder on the M9 ACE was selected for the demonstration/validation 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". NSWC Panama City Division (PCD) 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 psi pressure testing. Once PPA validated that the cylinder assembly had no internal or external leakage, the Dem/Val prototype cylinder was shipped to NSWC PCD 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 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 4-9 shows disassembled hydraulic tube casings following service.



Figure 4-9. Representative hydraulic cylinder tube casings from MK48 LVS disassembled following service.

4.1.3 Present Operations

FRCSE has a large workforce including civilians, contractors, and military personnel that perform a complete range of depot-level rework operations on designed weapon systems, accessories and equipment. The core capabilities of the depot are:

- Aircraft Program: comprising 28% of the depot workload, depot level maintenance and repair
- Aircraft Modernization Program: utilizing 9% of the facilities workload, this effort provides modification and modernization to existing fleet aircraft
- Engine Program: using 4% of the workload, this program offers rework capability for all repairable components, assemblies and accessories
- Component Repair: comprising 25% of the depot workload, this program offers repair and overhaul of hundreds of components in support of all military services, as well as foreign military sales programs
- Support Services: 34% of the workload falls into this category, providing a full range of life-cycle support in calibration, support equipment repair, manufacturing, and engineering and logistics support

Building 794 at the depot 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 4-10). 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 4-11 and Figure 4-12 show the plating process line and process work flow respectively.



Figure 4-10. Image of a chrome plating tank currently in use at FRCSE.

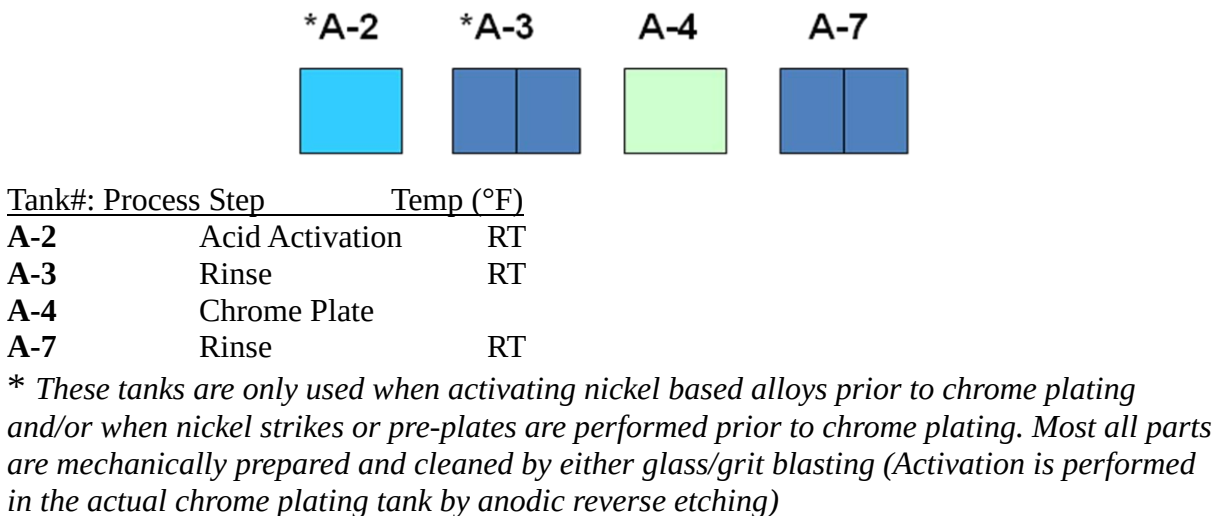


Figure 4-11. Schematic diagram of the process plating line for EHC at FRCSE.

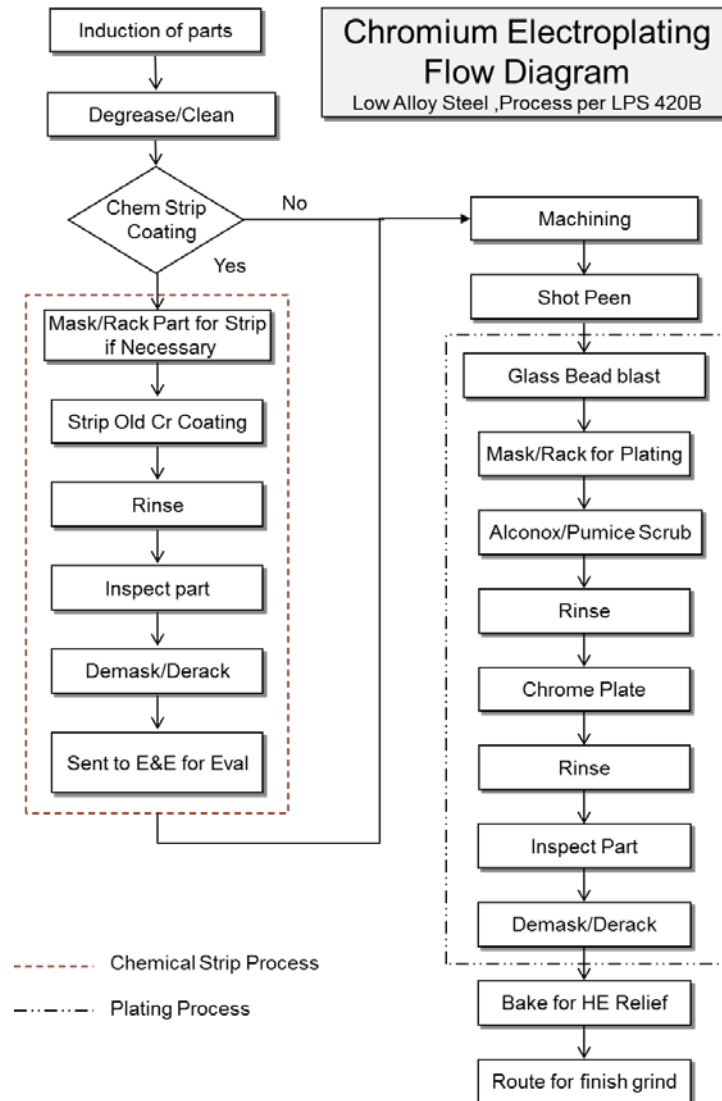


Figure 4-12. Process work flow diagram for EHC at FRCSE.

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. It can be found in the appendices.

4.2.2 Other Regulatory Issues

Cobalt is not covered under the Clean Air Act regulations. The PEL for Co metal is $0.1\text{mg}(\text{Co})/\text{m}^3$. From work conducted in the SERDP PP-1152 and ESTCP WP-0411 programs, the Co 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 (SVHC) under EU REACH 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/ORIGINAL EQUIPMENT MANUFACTURER (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 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.

Issues that will be relevant to repair depots and OEMs beyond showing that the nCoP coatings meet the acceptance criteria established in the JTP for performance characteristics are:

- Capability of grinding the coatings to achieve desired surface finishes with same amount of labor and materials required for grinding EHC
- Capability of removing the coatings at a rate equal to or faster than the current removal rates for EHC without damaging the base material
- Ability to develop conforming anodes or other plating strategies of no more complexity than required for EHC plating that results in reasonably uniform coating thicknesses on internal surfaces and complex geometries
- Deposition rates that exceed that of EHC plating
- Ability to selectively plate coatings using maskants that are compatible with this electrolyte

5.0 TEST DESIGN

5.1 JTP TESTING

This section details the work conducted under the 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. Note that extended tests detailed in Table 5-2 were not conducted at this time, but are presented for consideration as future work.

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, and AMS standards that are referenced are considered to be readily available and no further information needs to be provided.

Tests in this document may involve the use of hazardous materials, operations, and equipment. This document does not address all safety issues associated with its use. It is the responsibility of each user of this document to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to its use. Refer to the Health and Safety Plan developed for the Demonstration Plan corresponding to this document.

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 $\geq$ 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 $\geq$ EHC at a given thickness nCoP or Ni+nCoP $\geq$ 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 $\geq$ EHC per ASTM B537	ASTM B117 ASTM B537

Engineering Requirement	Test	JTR Test Description - Section	Acceptance Criteria	References
Corrosion	SO ₂ salt fog	4.3	Average appearance/protection ranking vs time curve: nCoP $\geq$ EHC per ASTM B537	ASTM G85 A4 ASTM B537
Corrosion	Beach exposure	4.4	Average appearance/protection ranking vs time curve: nCoP $\geq$ 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 – HE relief bake	Hydrogen embrittlement	4.7	1a1: four bars > 200h	ASTM F519
Hydrogen Embrittlement – no HE relief bake	Hydrogen embrittlement	4.7	load to failure: nCoP $\geq$ 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
Environmental Embrittlement	Sustained load in saltwater environment	4.9	Time to failure and load to failure: nCoP $\geq$ EHC	ASTM F519
Wear	Taber abrasive	4.10	Taber wear index: nCoP $\leq$ EHC	ASTM F1978 ASTM D4060
Wear	Pin on disk	4.11	Coating wear volume loss, coefficient of friction, static partner wear volume loss: nCoP $\leq$ EHC	ASTM G99
Wear	Endurance rig test	4.12	< one drop of hydraulic fluid in 25 cycles; wear not affecting leakage performance.	N/A

Engineering Requirement	Test	JTR Test Description - Section	Acceptance Criteria	References
Wear	Falex block on ring	4.13	Average weight loss and average wear volume: CoP $\leq$ 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 $\leq$ EHC	N/A
Corrosion	Cobalt oxide characterization	4.16	No acceptance criteria – characterization test	N/A

Table 5-2. Extended Performance and Testing Requirements.

Engineering Requirement	Test	JTR Test Description - Section	Acceptance Criteria	References	Participants Requiring Test
Adhesion ¹	Chisel-knife grind saw	N/A	Per NSWCCD Test Protocol (see note 1)	ASTM B571	NAVSEA
Corrosion ¹	Accelerated corrosion	N/A	Per NSWCCD Test Protocol (see note 1)	ASTM B117	NAVSEA
Adhesion/Wear ¹	Impact resistance	N/A	Per NSWCCD Test Protocol (see note 1)	ASTM B571	NAVSEA
Wear ¹	Taber abrasive	N/A	Per NSWCCD Test Protocol (see note 1)	ASTM D4060	NAVSEA
Wear ¹	Cyclic piston wear/corrosion	N/A	Per NSWCCD Test Protocol (see note 1)	ASTM B117 ASTM B610	NAVSEA
Corrosion	Accelerated corrosion	4.3	Average appearance ranking vs time curve: nCoP $\geq$ EHC per ASTM B537	GM9540P	NAVSEA

Fatigue – Various Substrates	Axial fatigue	4.2	S-N curves for given substrate material: nCoP $\geq$ EHC at a given thickness nCoP or Ni+nCoP $\geq$ Ni+EHC	ASTM E466	NAVAIR PAX
Fatigue – Hardening Study	Axial fatigue	4.2	S-N curves: nCoP $\geq$ EHC at a given thickness nCoP or Ni+nCoP $\geq$ Ni+EHC	ASTM E466	NAVAIR PAX
Fatigue – Alternate Bar ²	Axial fatigue	4.2	S-N curves: nCoP $\geq$ EHC at a given thickness	ASTM E466	Boeing
Hydrogen embrittlement – Type 1a2	Hydrogen embrittlement	4.7	1a2: four bars > 200h	ASTM F519	Boeing

¹Test 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.

²Test methods and acceptance criteria TBD pending stakeholder input.

	Proposed for future work
	Completed

5.2 DEM/VAL TESTING

5.2.1 Conceptual Experimental Design

The demonstration set-up commenced as a part of the previous project WP-0411. The initial part of implementation involved the establishment of a 350-gallon plating tank at Integran, which has been used for proof-of-principle plating depositions since 2003. As a part of WP-0411, supplemental work was conducted to optimize pulse plating parameters and production of large test specimens for material properties characterization. Additionally, successful demonstration of ID/OD plating on J52 components has been completed. The second phase of implementation involved the establishment of an nCoP plating capability at FRCSE. Integran previously acquired a 500 amp average/1,500 amp peak current pulse power supply and fully evaluated it prior to shipment to Jacksonville.

At FRCSE, Plating Tank A-11, which was formerly used for EHC plating, has been dedicated to the nCoP process. This tank has a capacity of 300 gallons, and was suitable for processing nCoP where EHC had previously been used. The pulse power supply was originally installed in the rectifier room; however it has since been relocated to the chrome plating area. The power supply is water-cooled with filtered city water. Chemicals for the plating bath are on hand. Sample plates have been produced. Numerous pits were observed on the deposits. The source of the pits was determined to be leaching from the tank liner.

Technology validation was underway following a site visit from Integran to FRCSE. The successful validation enabled the production of test coupons in advance of the demonstration/validation program. The test coupons produced were bright and smooth, with adherent deposits. Additionally, necessary analytical support has been transitioned in order to effectively run the process independently including conducting process control and determining corrective actions. Test samples and chemical analysis are to be completed on a weekly basis.

A technical process guide has been provided from Integran to FRCSE in order to successfully operate the nCoP plating tank. Within the document, explicit operating parameters, maintenance schedules and process specifications are contained. Over time, this document has been utilized to generate an internal military specification.

5.2.2 Pre-Demonstration Testing and Analysis

5.2.2.1 *Previous Testing and Development*

During the SERDP project, the microstructure of electrodeposited cobalt-based coatings was engineered in order to optimize the relevant properties such that the performance of the coating met or exceeded that of EHC. Electrodeposited nCoP demonstrated comparable and in some cases superior performance to EHC. Additionally, application to components with complex ID and OD geometries was successfully demonstrated.

During the ESTCP WP-0411 project, the nCoP process was fully scaled up to a production scale at Integran and subsequently FRCSE. Deposition parameters were optimized using a DOE

approach. Performance testing indicated that nCoP deposits showed excellent corrosion protection, good sliding wear and high hardness. The process was determined to be non-embrittling and fatigue testing indicated that the coating did not impart a fatigue debit to the underlying steel substrate.

Maskant materials have been identified. A commercial maskant, Enthone, Inc. EN-Plate Stop Off No.1, was used to selectively plate areas on components/test samples. No negative effects such as pitting or other signs of organic contamination have been noted to date. This maskant was used during processing of demonstration/validation components.

A stripping solution for removal of nCoP from ferrous substrates has been identified. A commercial stripping product, MacDermid, Inc. METEX SCB, was used to remove nCoP coatings. Testing showed the stripping rate to be approximately 0.001"/hr. The process chemistry and operating temperature can be changed to optimize the stripping rate. In most applications, grinding to a thickness of 0.002" will be completed followed by chemical stripping to remove the remaining coating.

5.2.3 Operating Parameters for the Technology

Since this is a pulse plating technology, the key parameters of investigation for optimization of coatings properties are:

- Current density
- Voltage
- Duty cycle
- Plating bath temperature
- pH of plating bath

All of these parameters have been studied previously. Furthermore, surface preparation for various materials and alloys has been completed.

Electroplating solution control and maintenance will be important and the requirements are expected to be similar to those of chromium plating solutions. Ongoing maintenance procedures should include filtration and monitoring various solution parameters such as: pH, solution density, impurities analysis (and subsequent corrective actions such as carbon filtration or dummy plating).

5.2.3.1 Design and Layout of Technology Components

Building 794 at FRCSE houses all cleaning, activation, and plating operations which will be performed during this demonstration/validation effort. The facility includes a closed-loop system that enables compliance with all EPA regulations. The chrome plating process tanks are located in a room separate from other plating operations with additional air exhaust capability to minimize worker exposure to plating chemical fumes. Utilizing existing infrastructure within this room a small line was modified for the purpose of the demonstration. 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.

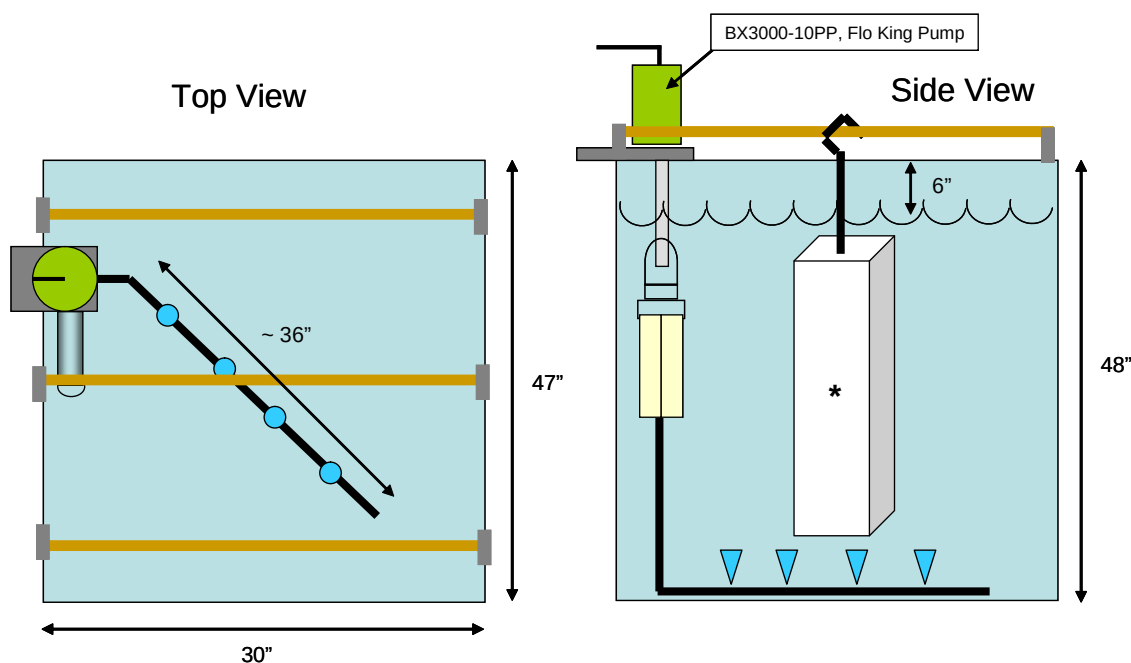
The nCoP electroplating deposition process was performed by following the NAVAIR Jacksonville Local Process Specification using the Process Plating Guidance provided by Integran. The document consists of various process steps which are described in more detail below.

5.2.3.2 Plating Process Tank

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 μ m nominal size polypro cartridges. A sparger equipped with eductors, nickel 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. Figure 5-3 shows a schematic of the plating tank design/setup.



Figure 5-2. Photograph of 300 gal nCoP plating tank at FRCSE.



** A 6" free board will be maintained on all process tanks. Above drawing is not per scale. **Largest size part to be within the following dimension: 20" x 20" x 36"***

Figure 5-3. Schematic showing top and side views of nCoP plating tank (A-11).

The constituents of the plating solution are shown below in Table 5-3. The process line was maintained according to Integran's Process Guide. The process guide is a technical document containing the windows of operation necessary to successfully obtain nCoP coatings with the desired material properties. Furthermore, it contains maintenance schedules which indicate the recommended frequency of analysis. When operating the process line, the key variables for control are pH, temperature, Cobalt ion concentration, nanoPLATE – A59 concentration and

agitation/flow conditions. Monitoring of iron and copper metal ion concentrations is required. In order to maintain the desired deposit composition, test coupons are produced on a regular basis. The test coupons are evaluated for appearance, microhardness and adhesion. The plating solution may be adjusted accordingly.

Table 5-3. Components of the nCoP process plating bath and their functions.

Chemical	Function
nanoPLATE® – Cobalt CL	Metal Content
nanoPLATE® – D60	Buffer
nanoPLATE® – A59	Brightner
nanoPLATE® – Cobalt CA	pH adjust (increase)
nanoPLATE® – E61	pH adjust (decrease)

5.2.3.3 Power Supply

The power supply or rectifier used in this project is a MODEL PMC201/1-500-1500 consisting of a 20 Volt / 500 Amp Average/1,500 Amp Peak pulse power supply equipped with a remote controller (MicroTouch) manufactured by Dynatronix. The approximate dimensions of the rectifier are 6.5 feet in height, 36 inches in depth, and 24 inches in width. The unit weighs approximately 900lbs. The rectifier used in this demonstration is water-cooled and must deliver a minimum water flow of 4 gallons per minute at a minimum pressure of 45 psi (60 psi max) at a temperature not to exceed 86°F at the input. The required input power connection is 480VAC / 3phase 50-60Hz (3-wire)/ 35Amp.

In order to maintain good pulse waveforms, the power supply was located as close to the tank as possible to avoid the inductance/capacitance problems associated with long power cables. Four twisted pairs of 1/0 cable in parallel were installed and terminated at the anode/cathode connections or busbars at the tank. A communications cable between the rectifier and the remote controller (MicroTouch) was also installed on the wall adjacent to the plating tank to control the current to the part.

A photo of the pulse rectifier and MicroTouch remote controller is shown in Figure 5-4.



Figure 5-4. Pulse Rectifier with filtered water system (left) and MicroTouch Remote (right).

6.0 PERFORMANCE ASSESSMENT

6.1 JTR RESULTS

6.1.1 Deposition Process

The following section briefly describes the deposition process conducted for preparation of test samples in accordance with Integran's Process Guide. Figure 6-1 and Figure 6-2 present a process flow chart and schematic tank diagram, respectively for the current configuration for the Dem/Val nCoP process line at NAVAIR FRCSE.

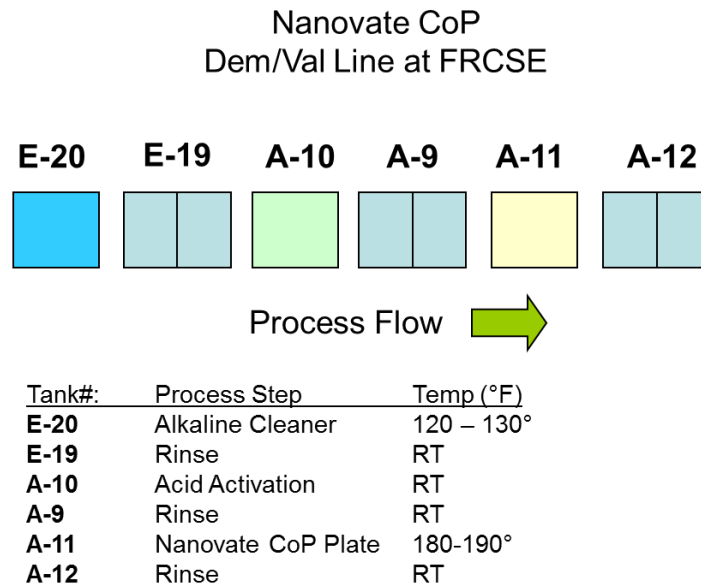


Figure 6-1. Process flow chart.

Tank A-11 Shown w/ Anodes, Buss Bars,
Filter Pumps, Sparger, & Steam Coil
Tank Dim: 30"x 47"x 48" w/ 6" freeboard
Tank Capacity: 300 gals

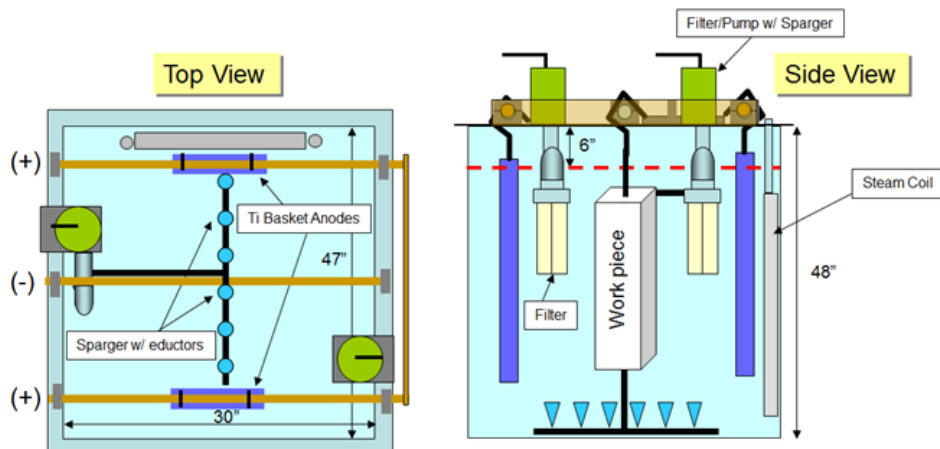


Figure 6-2. Schematic diagram of Tank A-11 Nanovate CoP plate (nCoP) plating tank.

Table 6-1 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.

Table 6-1. Process sequence for selected metals and alloys.

General Plating Process Steps nCoP electrodeposition on 4130 Steel				
Title		Solution		
nCoP Plate Electrocleaner Acid Activator		Proprietary Alloy Plating Bath (nCoP) P-C-535 (Alkaline Soak) or equivalent H ₂ SO ₄ /NH ₄ HF 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 min	4 to 6 volts anodic or (45 – 55 ASF)	125-135
10	Rinse			Room
11	Reverse Anodic Etch (activation)	40 sec 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 HE Relief/Deposit hardening	23 hr		375 ± 25

6.1.2 Coating Quality

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.

6.1.2.1 Appearance

Figure 6-3 shows representative images of nCoP and EHC deposits applied to 4130 steel. The nCoP samples were found to be smooth, bright and shiny, uniform in appearance, and free from blisters, pits, nodules, excessive edge build-up and other defects. The EHC coating was found to have a dull, matte finish and was free from blisters, pits, nodules, excessive edge build-up and other defects.

This result meets the acceptance criteria.

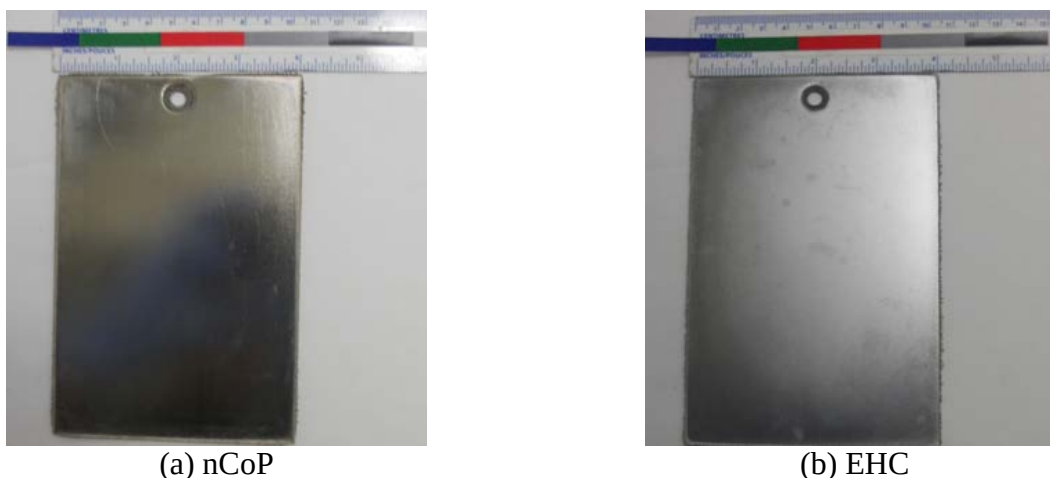


Figure 6-3. Images of representative (a) nCoP and (b) EHC deposits.

Table 6-2 indicates the average alloy composition of the nCoP samples. The deposit phosphorus content determined by SEM-EDS was found to be between 1.0-2.0 wt%, the acceptable range defined in Integran's Process Guide.

Table 6-2. Deposit phosphorus composition of nCoP deposits.

Coating	Sample 1 (%)	Sample 2 (%)	Sample 3 (%)	Average (%)
nCoP	1.7	1.8	1.8	1.8 ± 0.1

6.1.2.2 Thickness

Table 6-3 indicates the average nCoP and EHC deposit thicknesses as determined in cross-section by optical microscopy. The thickness of the nCoP was seen to be greater than 0.003". The thickness of EHC (Samples 1-3) was seen to be greater than 0.003" in cross-section by optical microscopy.

This result meets the acceptance criteria.

Table 6-3. Thickness of nCoP and EHC deposits.

Coating	Sample 1 (")	Sample 2 (")	Sample 3 (")	Average (")
nCoP	0.0030	0.0033	0.0030	0.0031 ± 0.0002
EHC	0.0033	0.0030	0.0032	0.0032 ± 0.0002

6.1.2.3 Porosity

Table 6-4 indicated the nCoP and EHC deposit porosity. nCoP had no spots/pits greater than 1/32" in diameter and less than 1 spot/pit in 150 square inches and no more than 1 in any 30 square inches of plating. EHC had no spots/pits greater than 1/32" in diameter and less than 4 spots/pits in 150 square inches and no more than 4 in any 30 square inches of plating.

This result meets the acceptance criteria.

Table 6-4. Porosity of nCoP and EHC deposits.

Coating	Sample 1 (spots)	Sample 2 (spots)	Sample 3 (spots)	Sample 4 (spots)	Sample 5 (spots)	Average (spots/150 sq")
nCoP	1	0	0	0	0	0.2
EHC	3	2	4	3	4	4.0

6.1.2.4 Hardness

Table 6-5 shows the hardness of nCoP and EHC at selected heat treatment conditions (i.e., hydrogen embrittlement relief bake-out, T-45 Aermet100 tempering limit, maximum hardening annealing treatment for precipitation hardening of nCoP).

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 (375°F for 23 hrs), T-45 bake out condition (475°F for 23 hrs), and max hardening condition (550°F for 4 hrs). The hardness of nCoP samples was equal 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 6-4.

Table 6-5. Hardness of nCoP and EHC coatings at various heat treatments.

Coating	Heat Treatment	Average Hardness (VHN)
nCoP	As Deposited	548 ± 15
EHC	As Deposited	807 ± 49
nCoP	HE, 375 °F, 23 hrs	640 ± 24
EHC	HE, 375 °F, 23 hrs	757 ± 33
nCoP	T-45, 475 °F, 23 hrs	699 ± 11
EHC	T-45, 475 °F, 23 hrs	740 ± 37
nCoP	nCoP Max, 550 °F, 4 hrs	692 ± 17
EHC	nCoP Max, 550 °F, 4 hrs	691 ± 61

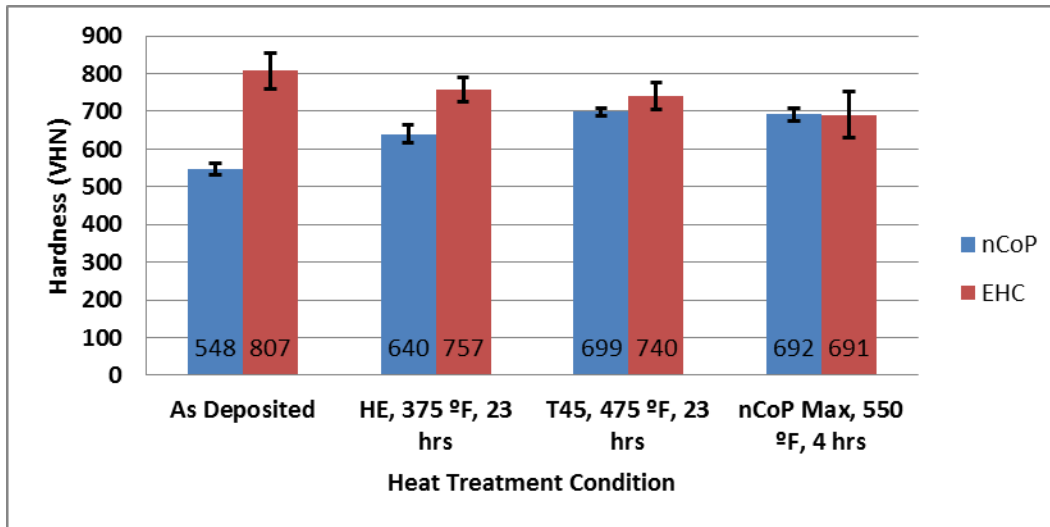


Figure 6-4. Microhardness of nCoP and EHC at various heat treatment conditions.

Figure 6-5 thru Figure 6-8 show results from a study that was previously conducted to evaluate heat treatments at various combinations of time and temperature for nCoP samples to determine an operating window for maximum hardening condition. In parallel, EHC samples were subjected to the same heat treatment conditions and their hardness was also determined. Both nCoP and EHC samples were prepared at NAVAIR FRCSE. The nCoP samples had a Co-1.7wt%P composition. Microhardness was measured on as-deposited and after heat treatment conditions. Heat treatment conditions included four temperatures (375, 475, 570 and 660°F) and four different bake times (1, 3, 5, 24 hours). At low temperatures, modest increases in hardness were observed in the nCoP samples. A maximum hardness of 755VHN₁₀₀ was obtained at 570°F and 5 hours, and a hardness in the range 714-732VHN₁₀₀ at 660°F and 1-3 hours. EHC samples softened at all temperature and time combinations when compared to their as-deposited hardness. It is interesting that the microhardness of EHC became 741VHN₁₀₀ when exposed to 375°F for 24 hours which corresponds to the standard hydrogen embrittlement relief bakeout. This hardness value is only 50 VHN₁₀₀ greater than that of nCoP also subjected to the same heat treatment conditions, and is comparable to nCoP at its maximum hardening condition. Despite the increases in microhardness which may be observed for nCoP following heat treatment, thus far wear testing has not revealed a significant performance benefit associated with the increase in microhardness.

This result meets the acceptance criteria for selected heat treatment conditions. The target hardness (>850 VHN) is defined by the EHC specification, but was not met. The threshold hardness (>530 VHN) which is defined by the alloy composition and microstructure of nCoP was met. Although the microhardness of the nCoP is generally lower than EHC, this does not adequately represent performance obtained in wear testing and field testing completed to date. The specification for EHC is likely defined for the specific performance of EHC plating processes or in other words, is used as a process control tool to ensure adequate wear and corrosion performance is obtained. The application of a heat treatment results in an increase in microhardness for nCoP samples as opposed to EHC samples which generally soften. No marked performance gains are observed by increasing microhardness of nCoP samples and therefore it is not specified as a requirement.

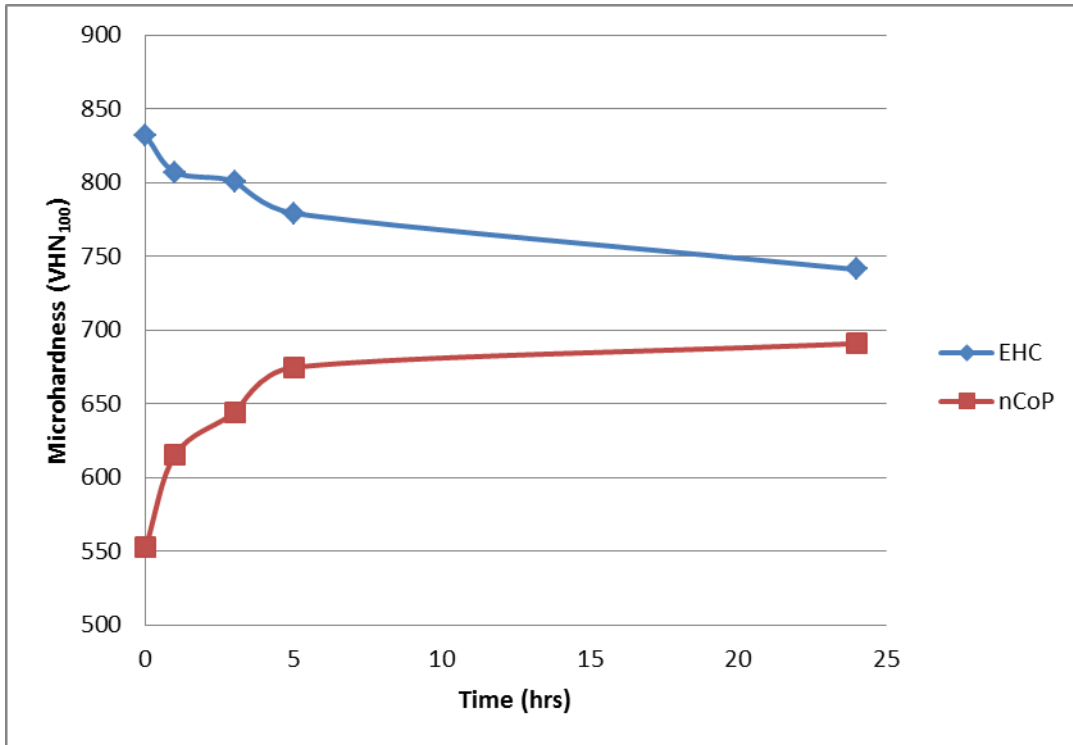


Figure 6-5. Microhardness of nCoP and EHC following heat treatment at 375°F at varying times.

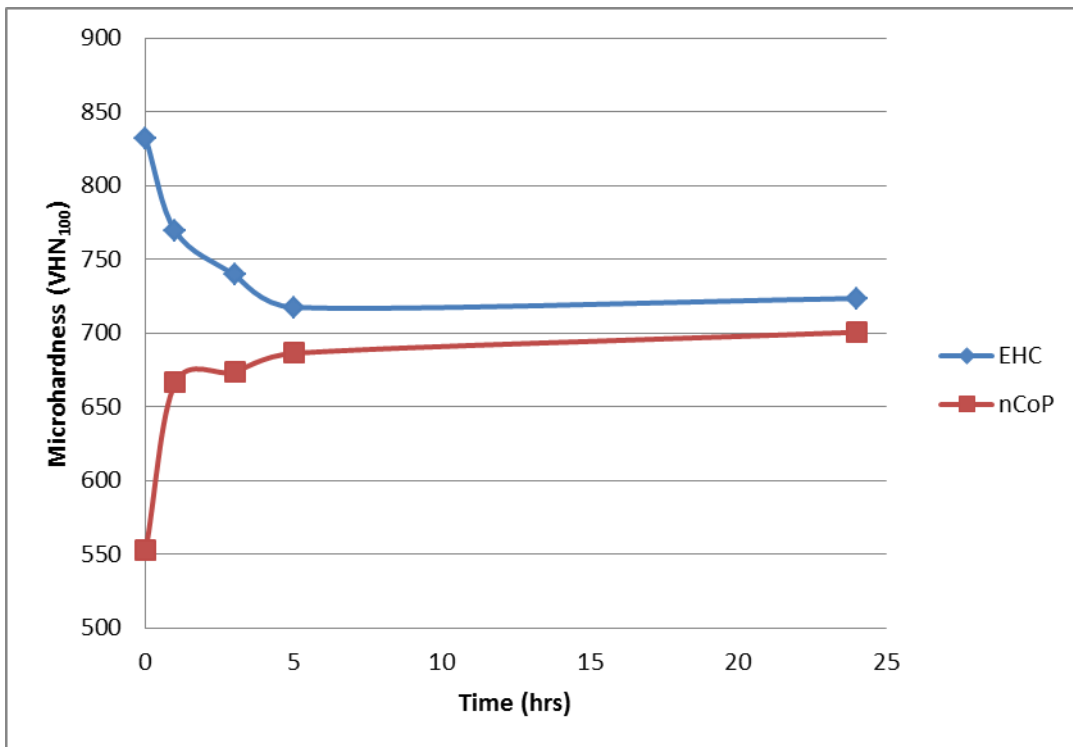


Figure 6-6. Microhardness of nCoP and EHC following heat treatment at 475°F at varying times.

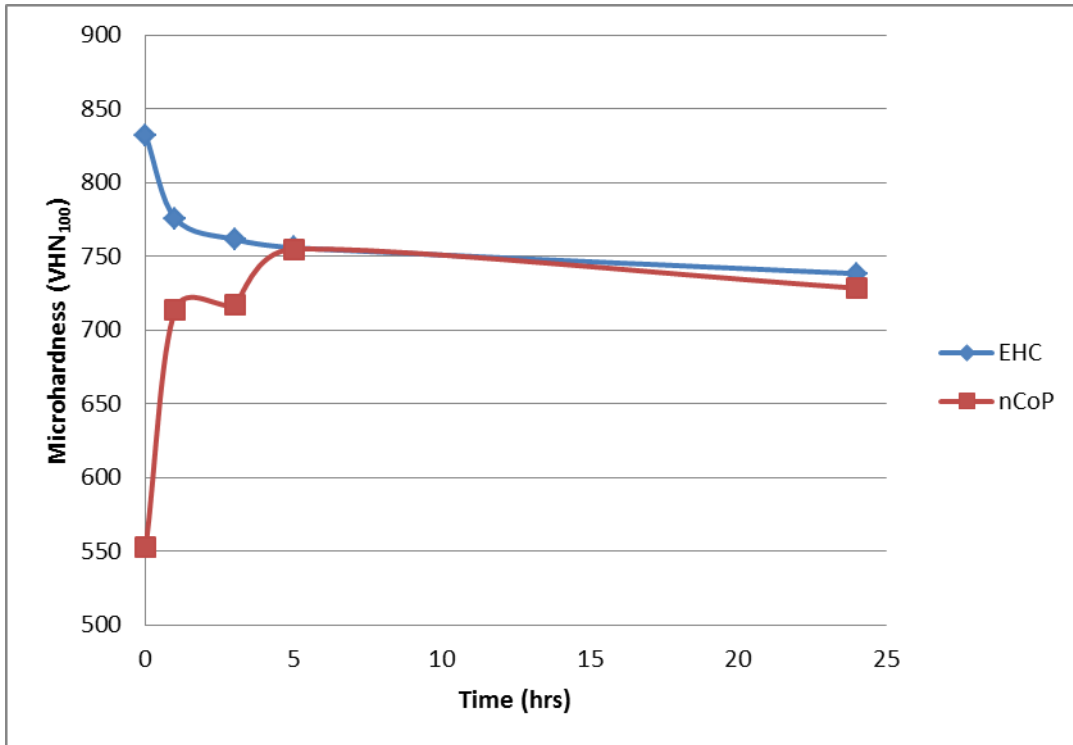


Figure 6-7. Microhardness of nCoP and EHC following heat treatment at 570°F at varying times.

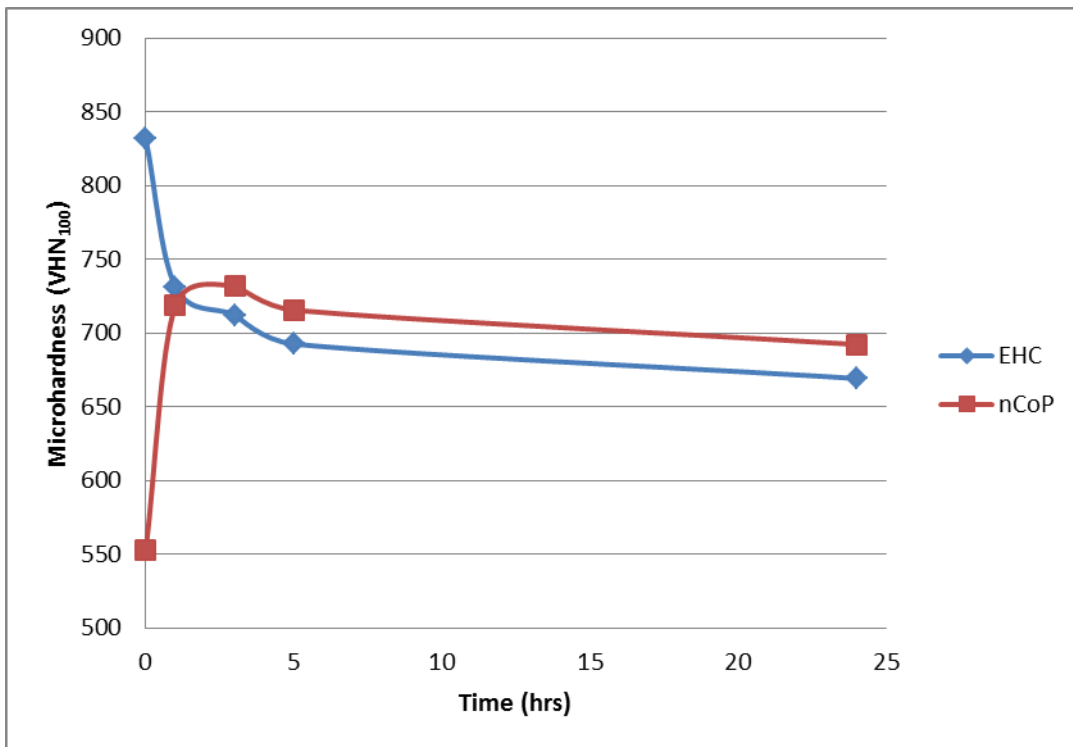


Figure 6-8. Microhardness of nCoP and EHC following heat treatment at 660°F at varying times.

6.1.2.5 Grain Size and Crystal Structure

Figure 6-9 shows the XRD spectra for the nCoP coatings evaluated in comparison to a theoretical cobalt HCP structure. Locations of diffraction peaks for nCoP indicate a HCP crystal structure. Samples 2 and 3 show the development of a peak at approximately $2\theta = 42$; this peak is a background peak from the sample holder. The calculated grain size is summarized in Table 6-6. The average grain size of nCoP was 6 nm.

This result meets the acceptance criteria.

Table 6-6. Grain Size of nCoP.

Coating	Sample 1 (nm)	Sample 2 (nm)	Sample 3 (nm)	Average (nm)
nCoP	6	5	8	6 ± 2

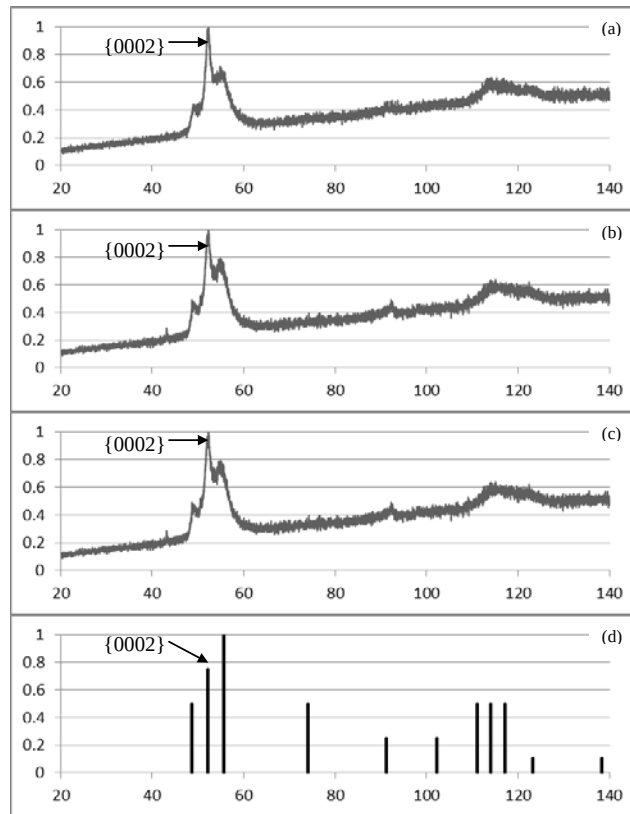


Figure 6-9. XRD Spectra for nCoP Samples 1-3 (a-c) compared to a Co-HCP standard (d).

6.1.2.6 Ductility

Table 6-7 lists the ductility for each specimen tested. The average ductility of nCoP was found to be 2.9%. The average ductility of EHC was seen to be less than 1%.

This result meets the acceptance criteria.

Table 6-7. Ductility of nCoP and EHC.

Coating	Sample 1 (%)	Sample 2 (%)	Sample 3 (%)	Average (%)
nCoP	2.8	2.9	2.9	2.9 ± 0.1
EHC	0.9	1.0	0.9	0.9 ± 0.1

6.1.2.7 Internal Stress

6.1.2.7.1 Spiral Contractometer

Table 6-8 below shows the internal stress of the nCoP coating obtained by spiral contractometer method. The stress was found to be 22-26 ksi tensile.

This result does not meet the acceptance criteria as outlined. However despite the relatively high internal stress, the ability to build thickness has not been compromised.

Table 6-8. Internal Stress of nCoP as determined by spiral contractometer.

Coating	Sample 1 (psi)	Sample 2 (psi)	Sample 3 (psi)	Average (psi)
nCoP	21645	24554	26042	24080 ± 1826

6.1.2.7.2 Cu Stress Strips

Table 6-9 below shows the internal stress of the nCoP coating obtained by copper stress strips method. The stress was found to be 15-20 ksi tensile. This result is not consistent with previous data obtained for nCoP which found the internal stress to be 10-15 ksi tensile. This inconsistency is due to the composition of the coating. Historically, nCoP deposits contained 2-4 wt% phosphorus; however more recently, the deposit phosphorus has been reduced to 1-2 wt% in order to optimize coating ductility and is defined as such in process specifications and documentation.

This result does not meet the acceptance criteria as outlined. However, despite the higher internal stress, the ability to build thickness has not been compromised. The result is not viewed to be detrimental to the coating and the accepted range for internal stress of nCoP with 1-2 wt% P is therefore redefined to be 15-20 ksi.

Table 6-9. Internal Stress of nCoP as determined by Cu stress strip.

Coating	Sample 1 (psi)	Sample 2 (psi)	Sample 3 (psi)	Average (psi)
nCoP	17850	16778	16091	16906 ± 887

6.1.3 Fatigue

6.1.3.1 Summary of Results

Fatigue is a very critical property in the aerospace industry, because of the repeated cyclic loading for landing gear, actuators, airframe parts, and gas turbine engine components. There is an extensive amount of fatigue data on metals and alloys that are used in such applications. When coatings are applied to the metals and alloys, the evaluation of fatigue essentially is the analysis of how the application of the coating affects the fatigue strength of the alloy. (i.e., a comparison is made between the cycles-to-failure at selected stress/strain values for coated and uncoated specimens). It is generally recognized that when EHC is applied to most alloys used in aerospace applications, the fatigue strength will be reduced because of micro-cracking and high residual tensile stresses present in the coatings.

The following work aimed to determine the impact of applying nCoP to 4340 steel (as per Table 6-10). The following general conclusions were drawn from the fatigue testing results:

- nCoP plated at 0.003” had fatigue performance equivalent to EHC at the same thickness (Figure 6-17).
- nCoP plated at 0.010” had fatigue performance equivalent to EHC at the same thickness (Figure 6-19).
- Sulfamate Ni + nCoP had worse fatigue performance than sulfamate Ni + EHC at one load condition (i.e., S=149ksi) (Figure 6-22).
- Sample populations at each load condition were evaluated using a one-way ANOVA and two sample t-test (Table 6-11/Figure 6-18, Table 6-12/Figure 6-20/Figure 6-21 and Table 6-13/Figure 6-23/Figure 6-24). nCoP performed best relative to bare and EHC at the highest load conditions (i.e., S=187ksi).
- None of the nCoP or EHC plated samples delaminated from the fatigue test specimens during the fatigue test or when the fatigue test specimens failed.
- Further fatigue testing is recommended in both axial and rotating bending configurations on various relevant aerospace substrates.

Table 6-10. Fatigue Test Matrix.

Coating	Coating Thickness	Quantity
		4340
None	-	16
EHC	0.003”	16
EHC	0.010”	16
Ni+EHC	0.002” Ni + 0.003” EHC	16
nCoP	0.003”	16
nCoP	0.010”	16
nCoP	0.020”	16
Ni+nCoP	0.002” Ni + 0.003” nCoP	16
Total		128

Integrity testing was verified by examining cross-sections of the fracture surfaces for all 16 nCoP samples coated to 0.020” thickness and on all 16 sulfamate Ni + nCoP samples coated to 0.002” and 0.003” thickness, respectively. Figure 6-10 is a representative image showing no indication of plating delamination on the cross-section of a bar. Attempts to delaminate the plating from the steel with a sharp knife was not possible on all samples tested demonstrating good adhesion of the plating to the steel.

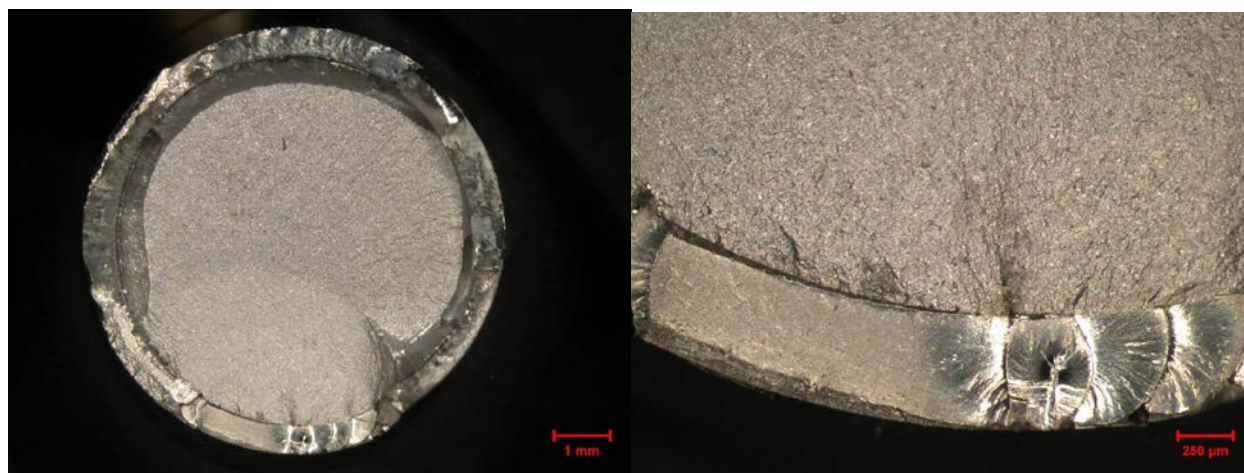


Figure 6-10. Representative micrograph at 10X showing the entire fracture surface (left); micrograph at 40X showing the condition of the interface between the coating and the base metal (right) for a nCoP sample coated to 0.020” thickness. The coating adhesion was verified and showed no delamination following testing.

Results of the fractographic analysis was completed on select fatigue test specimens to determine crack origin and failure surface characteristics. Most of the fractures appeared to be normal with crack origins occurring at the surface, or interface between the plating and steel, or in the coating itself (see Figure 6-11-Figure 6-15). However, one sample was quite different from the others. Specimen 88 (nCoP, 0.010”) showed the crack origin at the interface between the plating and the steel substrate, but the fracture surface also showed brittle features structure indicating the possibility of hydrogen embrittlement or some other type of embrittling mechanism (see Figure 6-16). Specimen 88 also failed prematurely. The applied stress was 168 ksi and it failed after only 846 cycles, while the other three in this set failed at between 12,000 to 20,000 cycles.

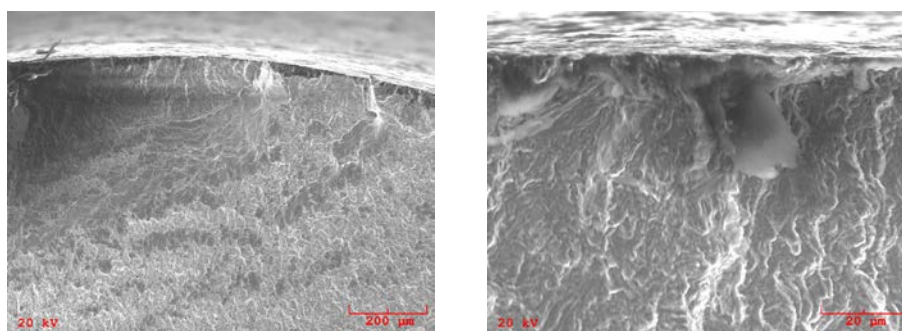


Figure 6-11. SEM image of fracture surface showing surface initiation – Specimen 3 (bare, S=187ksi, Nf=2,832 cycles).

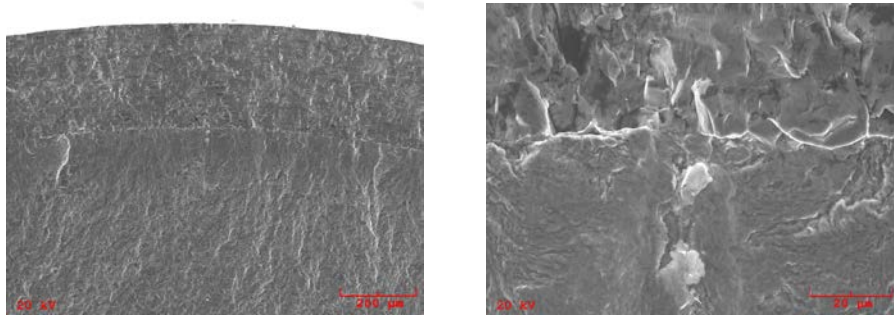


Figure 6-12. SEM image of fracture surface showing interface initiation – Specimen 46 (EHC 0.010”, S=130ksi, Nf=62,006 cycles).

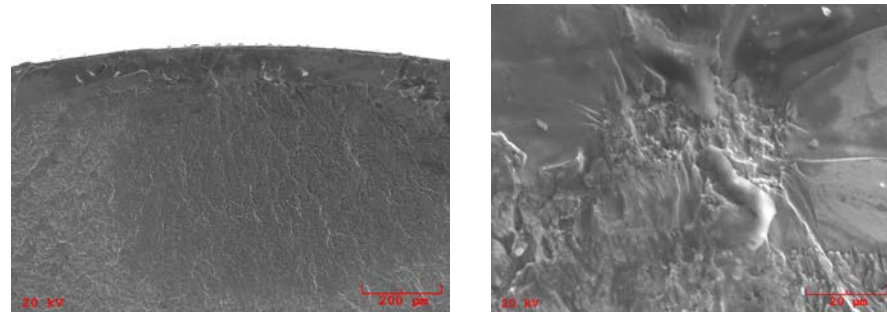


Figure 6-13. SEM image of fracture surface showing initiation near interface – Specimen 76 (nCoP 0.003”, S=149ksi, Nf=17,496 cycles).

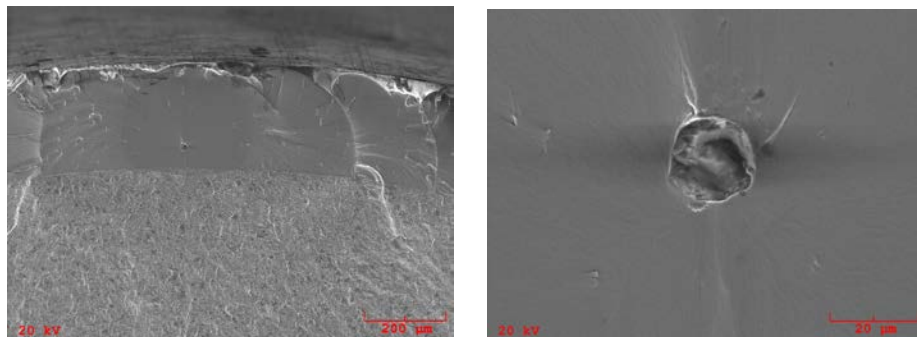


Figure 6-14. SEM image of fracture surface showing initiation at inclusion in coating – Specimen 94 (nCoP 0.010”, S=130ksi, Nf=130,348 cycles).

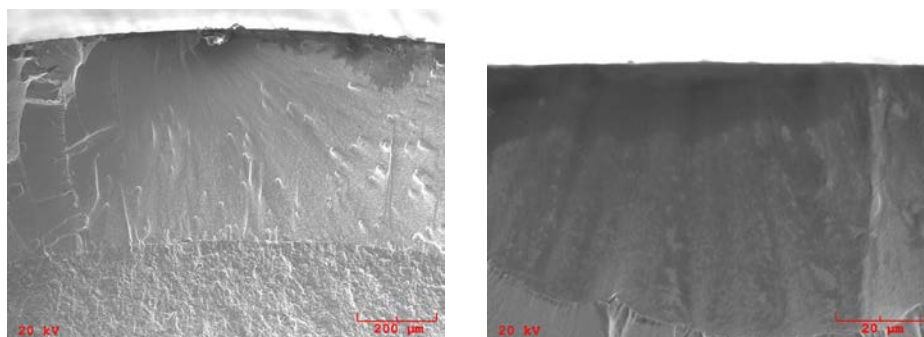


Figure 6-15. SEM image of fracture surface showing surface initiation – Specimen 101 (nCoP 0.020”, S=168ksi, Nf=13,122 cycles).

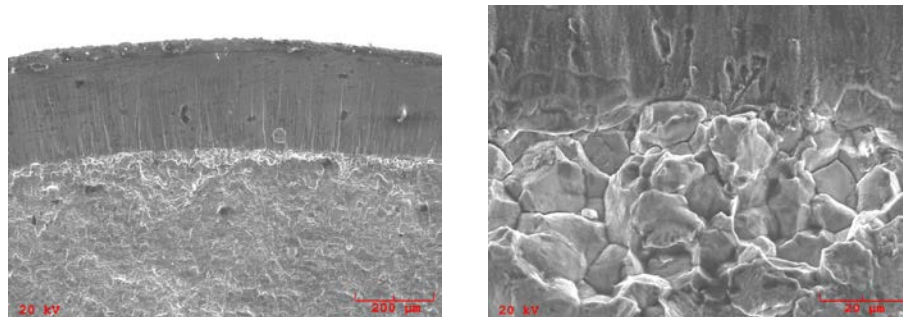


Figure 6-16. SEM image of Specimen 88 (nCoP, 0.010”) showing evidence of crack origin at the interface (left) and brittle features structure (right) indicating the possibility of hydrogen embrittlement.

6.1.3.2 Discussion

Fatigue data shows fatigue debit for EHC coating compared to bare material. In comparison, the nCoP coating shows a fatigue life similar to the bare material at higher loads. At lower applied loads there appears to be a debit compared to bare. Stress life data curves are represented in Figure 6-17, Figure 6-19 and Figure 6-22. Visual depictions of the statistical analysis are represented by boxplots in Figure 6-18, Figure 6-20, Figure 6-21, Figure 6-23 and Figure 6-24.

It is hypothesized that the fully dense microstructure of the nCoP relative to the micro-cracking found in hard chrome would necessitate hydrogen embrittlement relief baking prior to coating with nCoP. Given the ability to produce thick builds of nCoP (i.e., >0.010”) without resulting in significant fatigue debits, the necessity of a sulfamate nickel underlayer is minimized in contrast to hard chrome, which experiences a severe fatigue debit at thick builds. Statistical analysis of fatigue data revealed that at lower load conditions, nCoP possessed greater variability in cycles to failure. The lower load, high-cycle fatigue performance is typically influenced by material ductility. The deposit tensile ductility has been previously shown to be influenced by the presence of defects incorporated during deposition. In a tensile loading configuration, the presence of microscopic defects within the electrodeposit has a profound impact on overall mechanical performance. Previous fatigue testing in a bending load configuration showed less variability at lower loads. For future implementation of nCoP beyond traditional wear and corrosion resistance applications (i.e., with the objective of enhancing component life relative to hard chrome), use of optimized in-process filtration can significantly reduce the defect population, thus potentially improving fatigue performance.

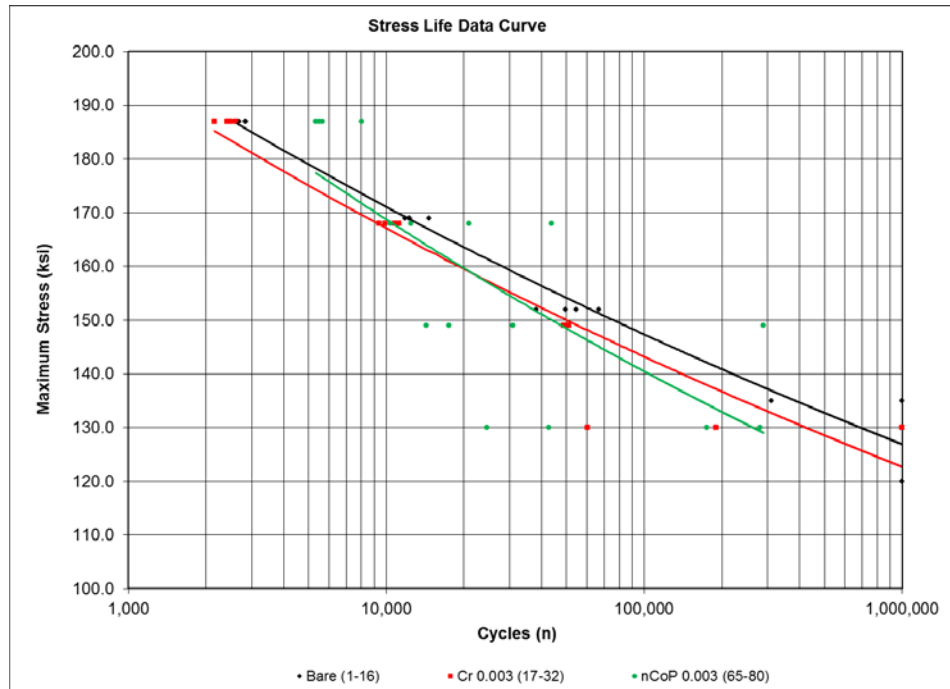


Figure 6-17. S-N curve comparing bare with EHC and nCoP coated samples at a thickness of 0.003". Test conditions: Ambient air, load control, R=-1, Frequency=20Hz.

Table 6-11. Statistical analysis by ANOVA and two sample t-test.

ANOVA test (For bare, nCoP-0.003", EHC-0.003")	
187 ksi	p=0.00
168 ksi	p=0.21
149 ksi	p=0.75
130 ksi	p=0.06

Two sample t-test (nCoP-0.003", EHC-0.003")	
187 ksi	p=0.010
168 ksi	p=0.228
149 ksi	p=0.609
130 ksi	p=0.197

Bold indicates that the $P < 0.05$, therefore, we reject the null hypothesis (i.e., the sample populations are statistically different).

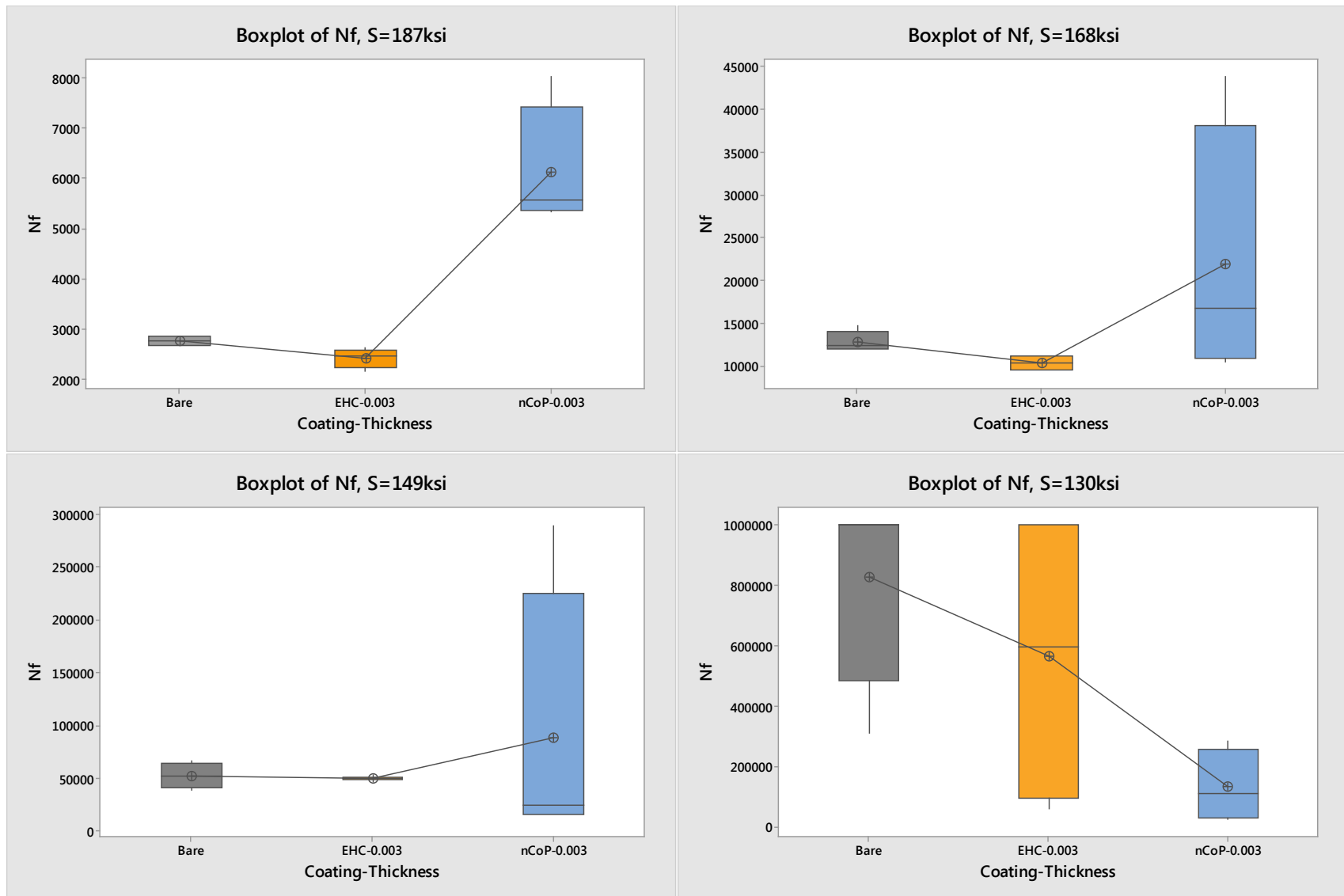


Figure 6-18. Boxplot comparing bare with EHC and nCoP coated samples at a thickness of 0.003” at each load level. Test conditions: Ambient air, R=-1, Frequency=20Hz.

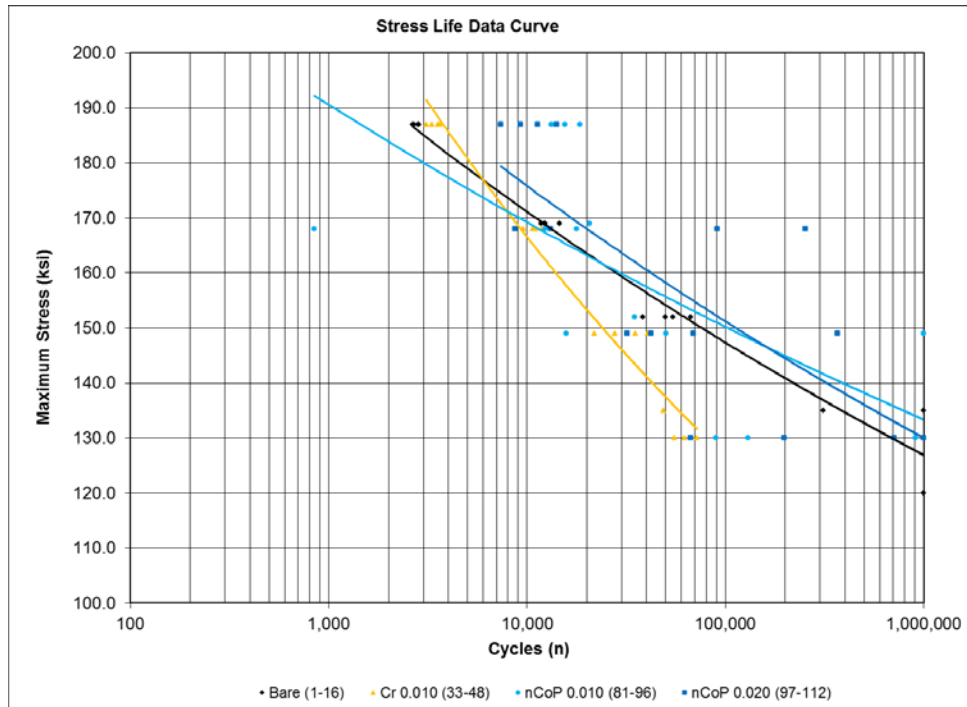


Figure 6-19. S-N curve comparing bare with EHC and nCoP coated samples at a thickness of 0.010” and 0.020”. Test conditions: Ambient air, load control, R=-1, Frequency=20Hz.

Table 6-12. Statistical analysis by ANOVA and two sample t-test.

ANOVA test (For bare, nCoP-0.010”, EHC-0.010”)	
187 ksi	p=0.00
168 ksi	p=0.81
149 ksi	p=0.43
130 ksi	p=0.03

Two sample t-test (nCoP-0.010”, EHC-0.010”)	
187 ksi	p=0.002
168 ksi	p=0.665
149 ksi	p=0.387
130 ksi	p=0.149

Bold indicates that the $P < 0.05$, therefore, we reject the null hypothesis (i.e., the sample populations are statistically different).

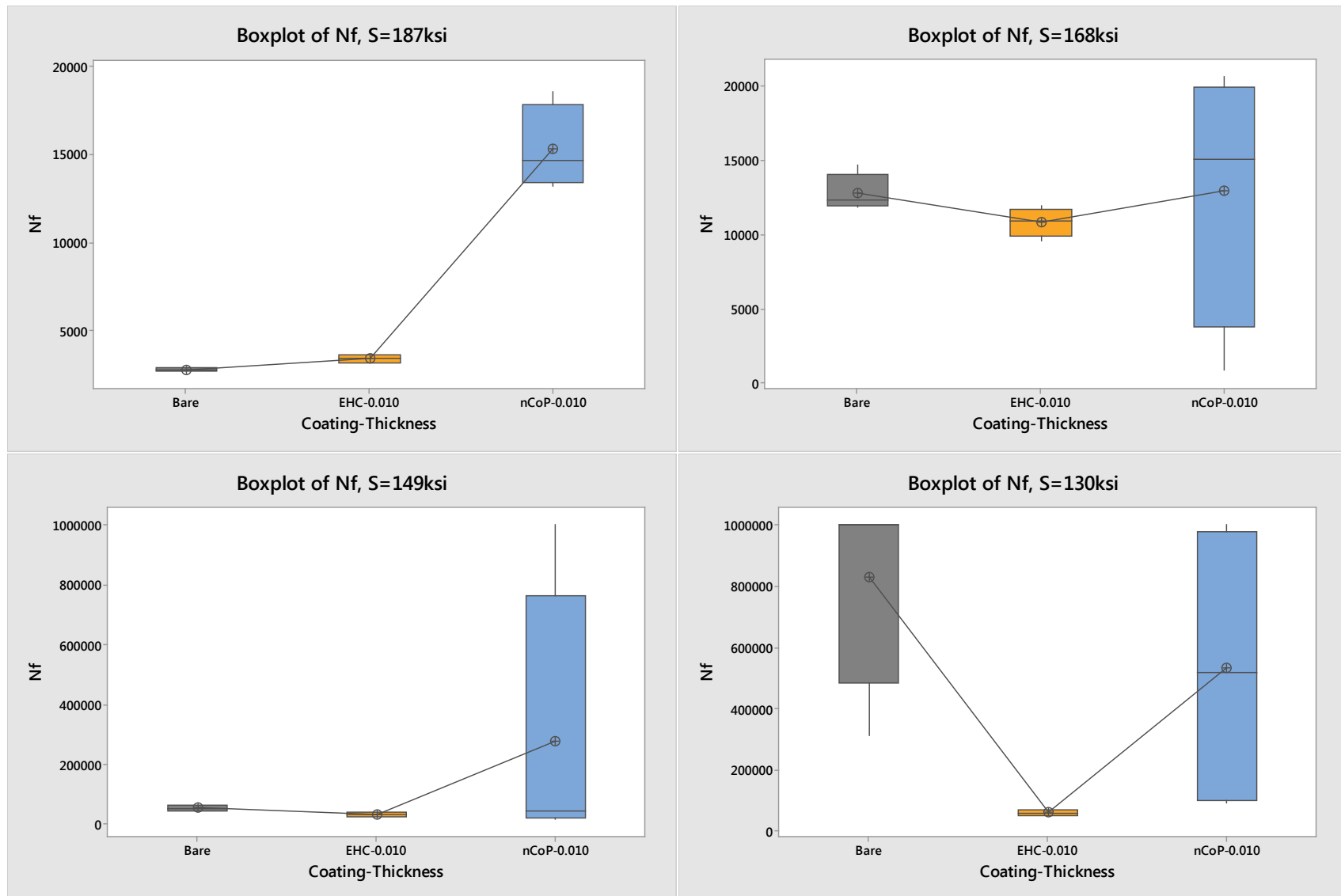


Figure 6-20. Boxplot comparing bare with EHC and nCoP coated samples at a thickness of 0.010” at each load level. Test conditions: Ambient air, R=-1, Frequency=20Hz.

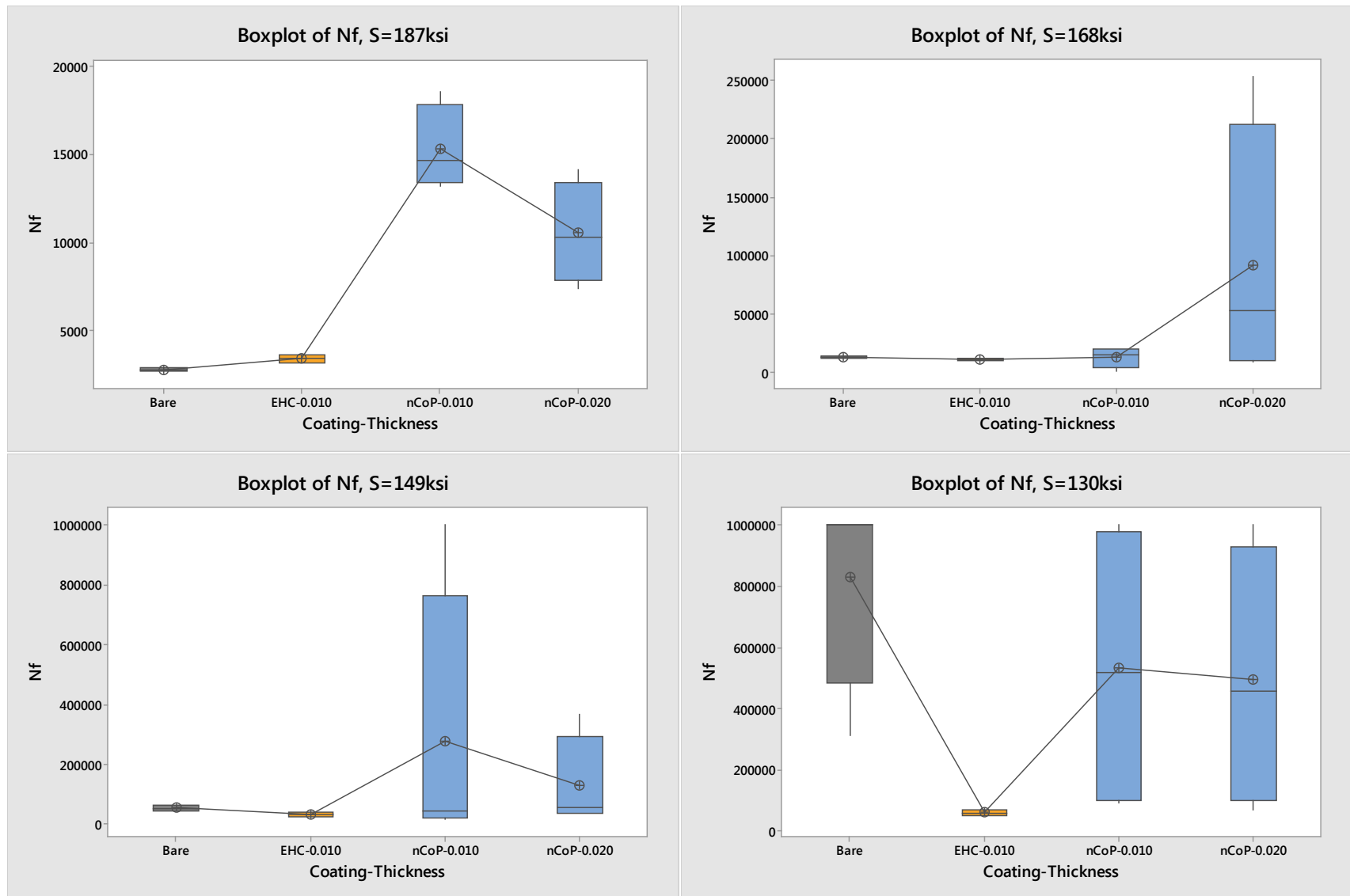


Figure 6-21. Boxplot comparing bare with EHC and nCoP coated samples at a thickness of 0.010" and 0.020" at each load level. Test conditions: Ambient air, R=-1, Frequency=20Hz.

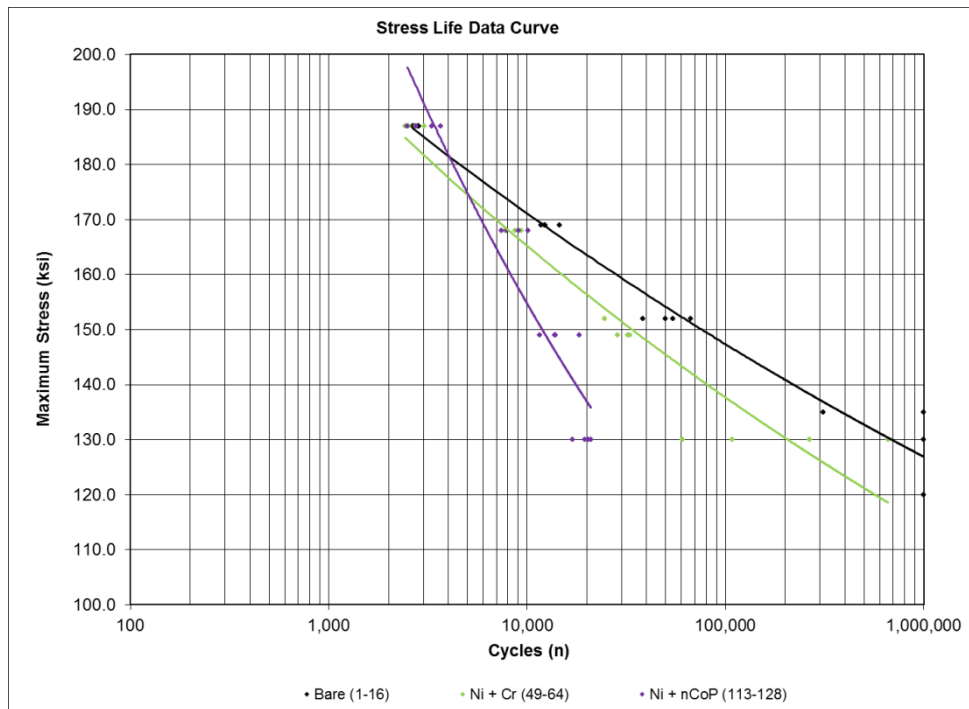


Figure 6-22. S-N curve comparing bare with Ni + EHC and Ni + nCoP coated samples at a thickness of 0.002" (Ni) + 0.003" (EHC or nCoP). Test conditions: Ambient air, load control, R=-1, Frequency=20Hz.

Table 6-13. Statistical analysis by ANOVA and two sample t-test.

ANOVA test (For bare, Ni-0.002" + nCoP-0.003", Ni-0.002" + EHC-0.003")	
187 ksi	p=0.31
168 ksi	p=0.00
149 ksi	p=0.00
130 ksi	p=0.00

Two sample t-test (Ni-0.002" + nCoP-0.003", Ni-0.002" + EHC-0.003")	
187 ksi	p=0.269
168 ksi	p=0.776
149 ksi	p=0.001
130 ksi	p=0.159

Bold indicates that the $P < 0.05$, therefore, we reject the null hypothesis (i.e., the sample populations are statistically different).

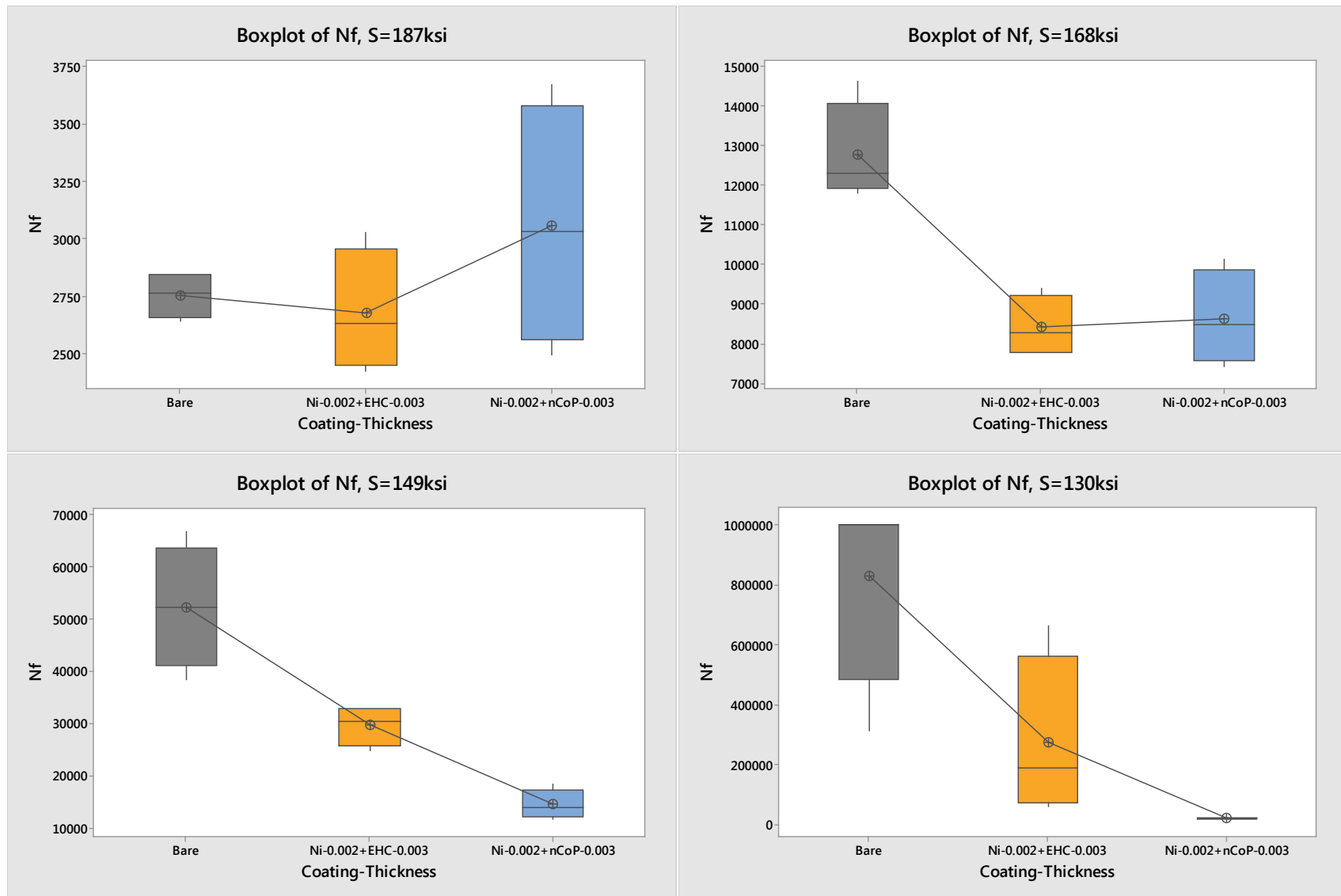


Figure 6-23. Boxplot comparing bare with Ni + EHC and Ni + nCoP coated samples at a thickness of 0.002” (Ni) + 0.003” (EHC or nCoP). Test conditions: Ambient air, load=187ksi, R=1, Frequency=20Hz.

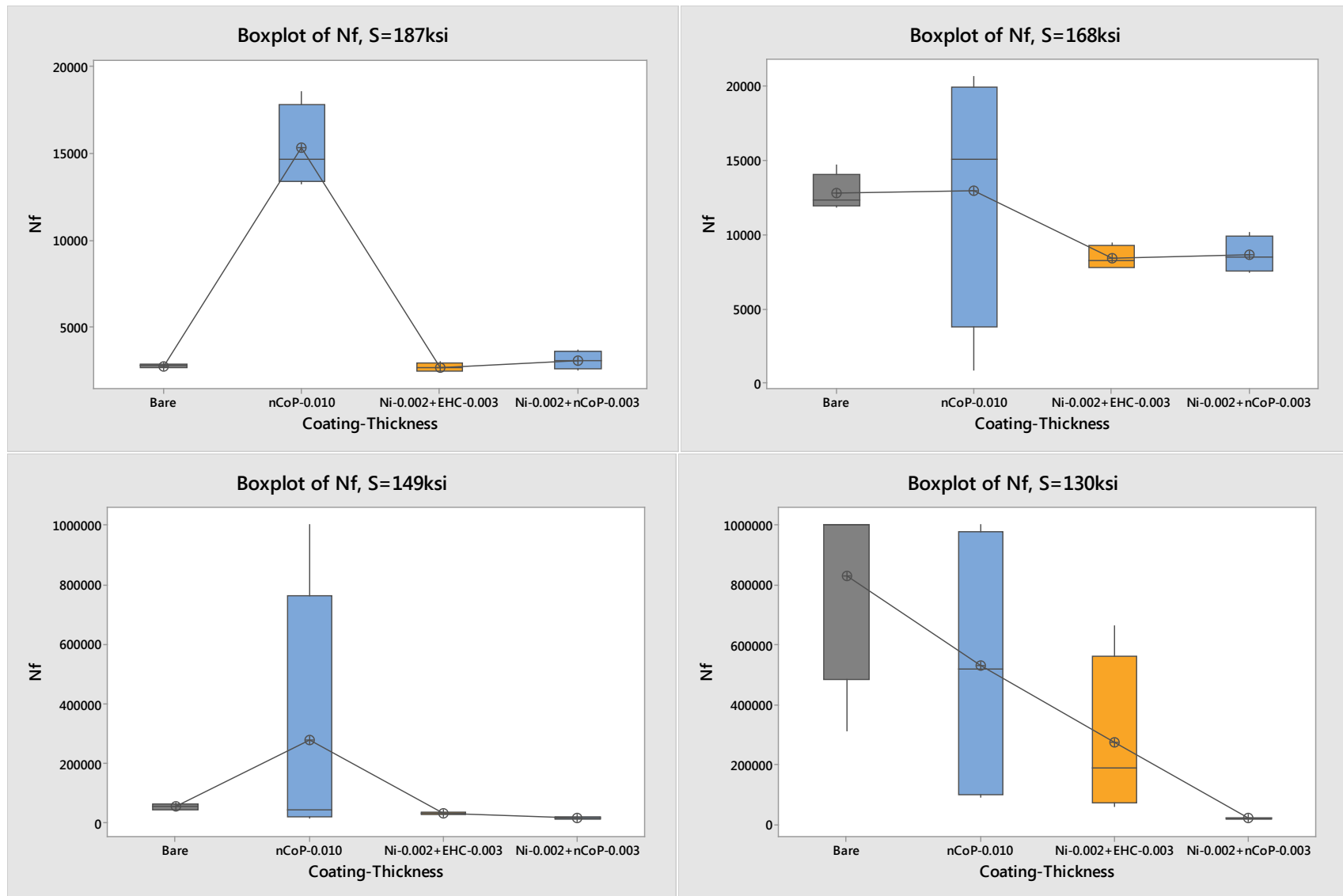


Figure 6-24. Boxplot comparing bare with Ni + EHC and Ni + nCoP coated samples at a thickness of 0.002” (Ni) + 0.003” (EHC or nCoP). Test conditions: Ambient air, load=187ksi, R=-1, Frequency=20Hz.

6.1.4 Corrosion – Neutral Salt Fog

Corrosion protection of steel in neutral salt fog is used widespread in aerospace applications in order to compare coating systems. This relatively simple test can be performed in cabinets with a controlled environment as defined in ASTM B117. The testing provides valuable insight into the coatings ability to prevent substrate corrosion following exposure for a given period of time. It is known that nCoP will form a brown-red oxide on the surface of samples when exposed to oxidizing conditions. The tarnish is a protective film and does not compromise the coatings ability to protect the underlying steel. However, in plan view images the oxide may be easily misinterpreted as red rust, while in actual view it is distinguishable from red rust by its denser, more compact appearance. It is recommended that operators perform a compositional test using XRF or other suitable methods in order to detect presence of Fe on the surface of nCoP salt spray panels to provide verification of red rust. Destructive testing of nCoP salt spray panels by metallurgical cross-sectioning and evaluation of porosity (no pores acceptable) and positive presence of corrosion pathways may also be employed for verification.

After 192 hours exposure to neutral salt fog, samples with EHC coated on steel showed varying degrees of corrosion, ranging from no corrosion to significant corrosion as evidenced by the appearance of red rust. For EHC with Ni underplate, no appearance of red rust was noted after 2000 hrs. The nCoP coatings with and without Ni underplate developed a superficial, brown colored oxidation layer, but protected the substrate from corrosion.

Following 3 weeks (500 hrs) of testing, there was little change in nCoP coatings except for a progressive darkening which is associated with the increasing surface oxide layer thickness. The EHC samples showed progressively greater rusting while the EHC with Ni underplate continued to protect the substrate against corrosion, except for slight rusting at the tape interface at the bottom of the panel.

Upon completion of the test (2000 hours), nCoP continued to protect against corrosion of the base metal while the EHC samples were highly corroded. The presence of Ni underplate with EHC successfully prevented corrosion of the substrate, but provided no benefit to the nCoP plated samples.

Figure 6-25 and Figure 6-26 show the appearance and protection ratings of EHC and nCoP coatings, respectively. Note that the protection ranking of Ni+EHC, Ni+nCoP and nCoP are identical and curves lie on top of each other. Figure 6-27 presents representative images of test panels taken at 0 hrs and 2000 hrs.

The results obtained meet the acceptance criteria.

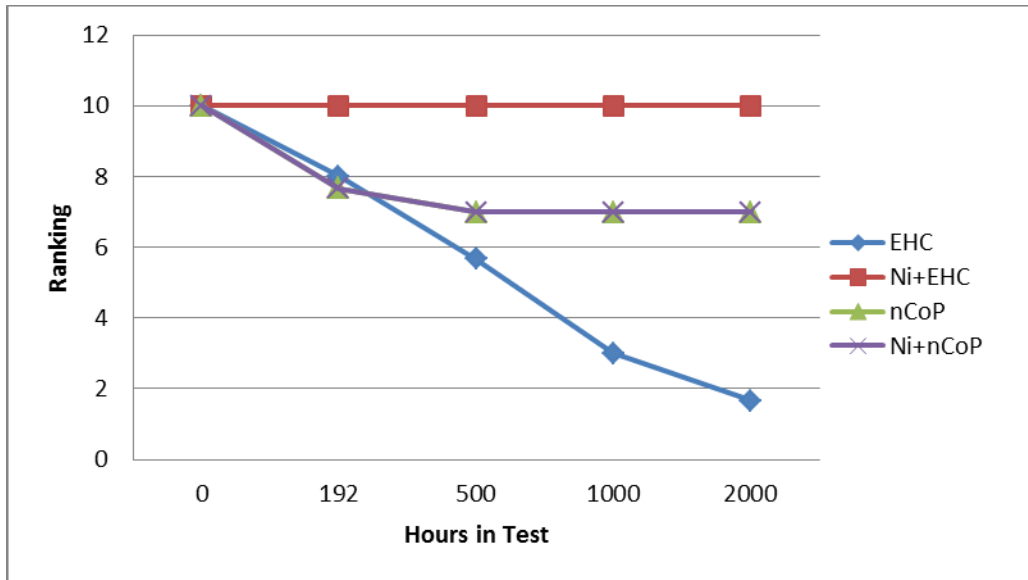


Figure 6-25. Appearance ranking for EHC and nCoP coatings in neutral salt fog corrosion testing.

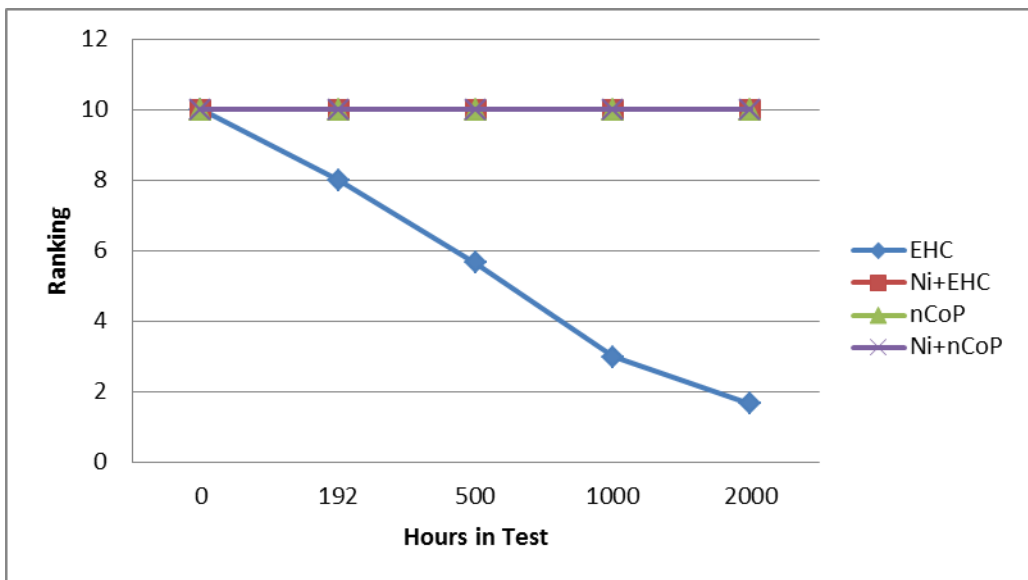


Figure 6-26. Protection ranking for EHC and nCoP coatings in neutral salt fog corrosion testing.

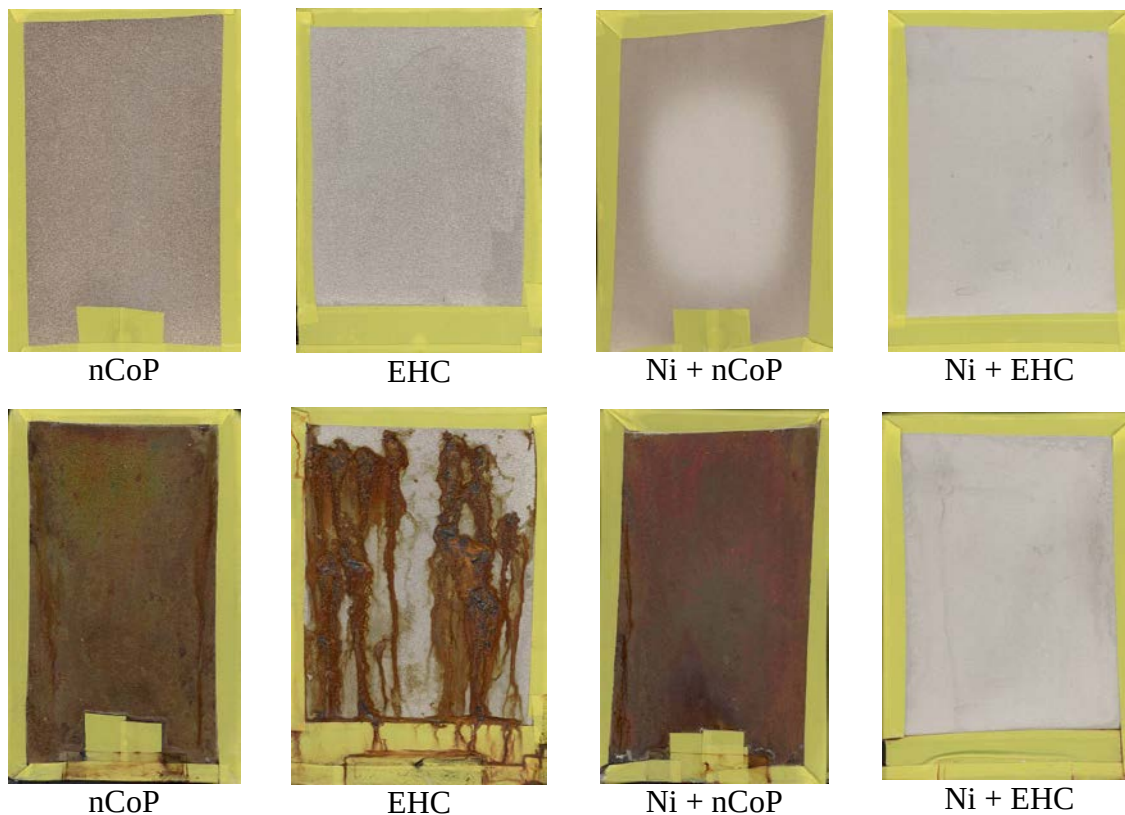


Figure 6-27. Representative images of coupons following exposure to neutral salt fog for 0 hrs (top) and 2000 hrs (bottom).

6.1.5 Corrosion – SO₂ Salt Fog

Corrosion protection of steel in acidic (SO₂) salt fog is used widespread and to rapidly evaluate coatings in aggressive conditions. Similar to neutral salt fog, this test is performed in cabinets with a controlled environment as defined in ASTM G85A.4. The testing provides valuable insight into the coatings ability to prevent formation of red rust following exposure to an acidified SO₂ fog environment for a given period of time.

After 24 hrs, EHC plated on steel samples showed signs of red rust. The EHC with Ni underlayer remained bright with no appearance of red rust. The nCoP with and without underplate developed a superficial brown colored oxidation layer, but protected the substrate from corrosion.

Following 336 hrs, significant red rust formed on the EHC samples while the EHC with Ni underlayer showed no appearance of red rust. For nCoP samples, the appearance was progressively darker which is associated with the increasing surface oxide layer thickness. Verification by cross-sectioning nCoP samples confirmed the absence of pits and substrate corrosion.

Figure 6-28 and Figure 6-29 show the appearance and protection ratings of EHC and nCoP coatings, respectively. Figure 6-30 presents representative images of test panels taken at 0 hrs and 336 hrs.

The results obtained meet the acceptance criteria.

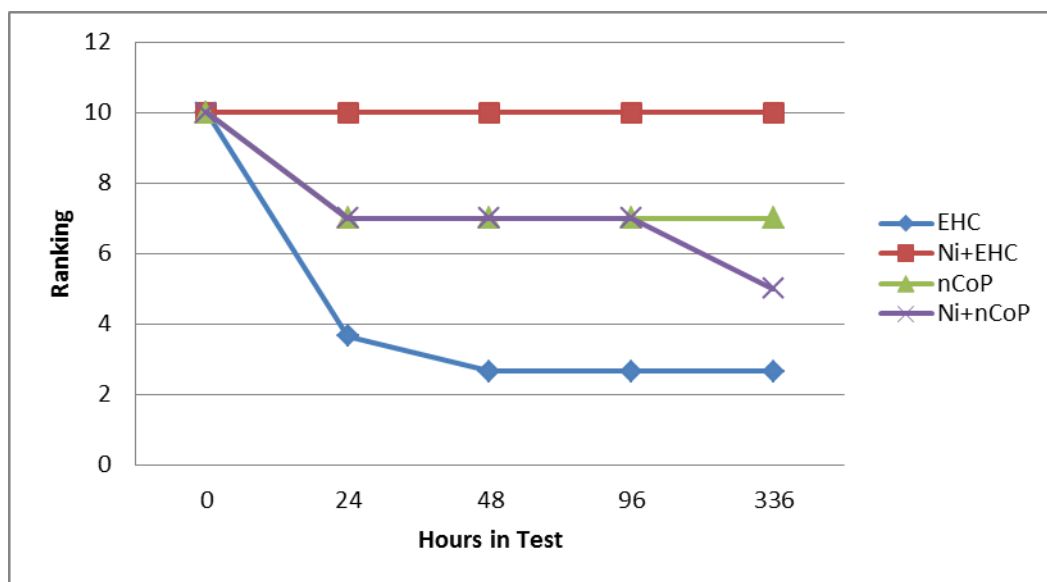


Figure 6-28. Appearance ranking for EHC and nCoP coatings in acidic salt fog corrosion testing.

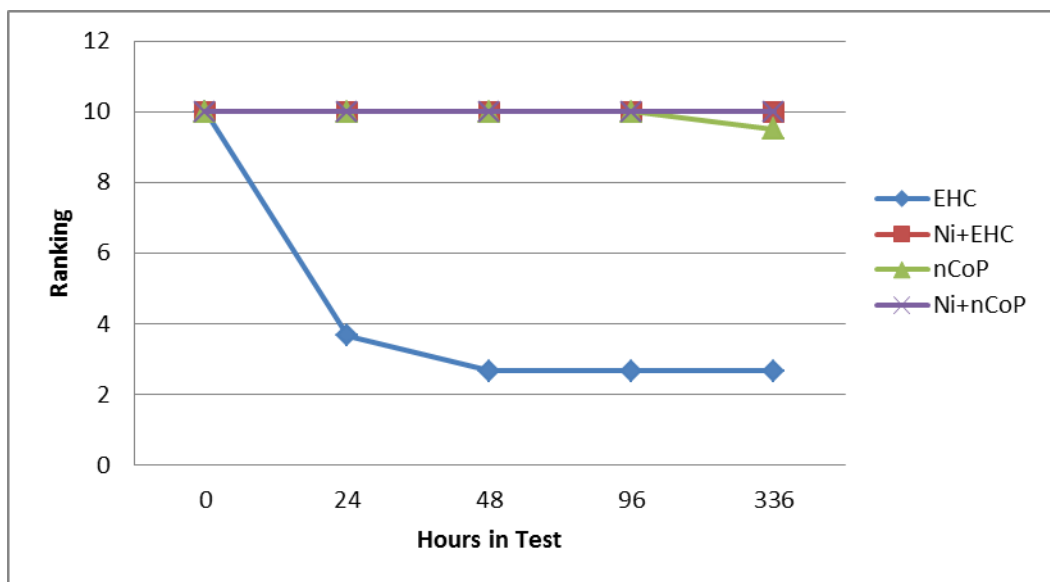


Figure 6-29. Protection ranking for EHC and nCoP coatings in acidic salt fog corrosion testing.

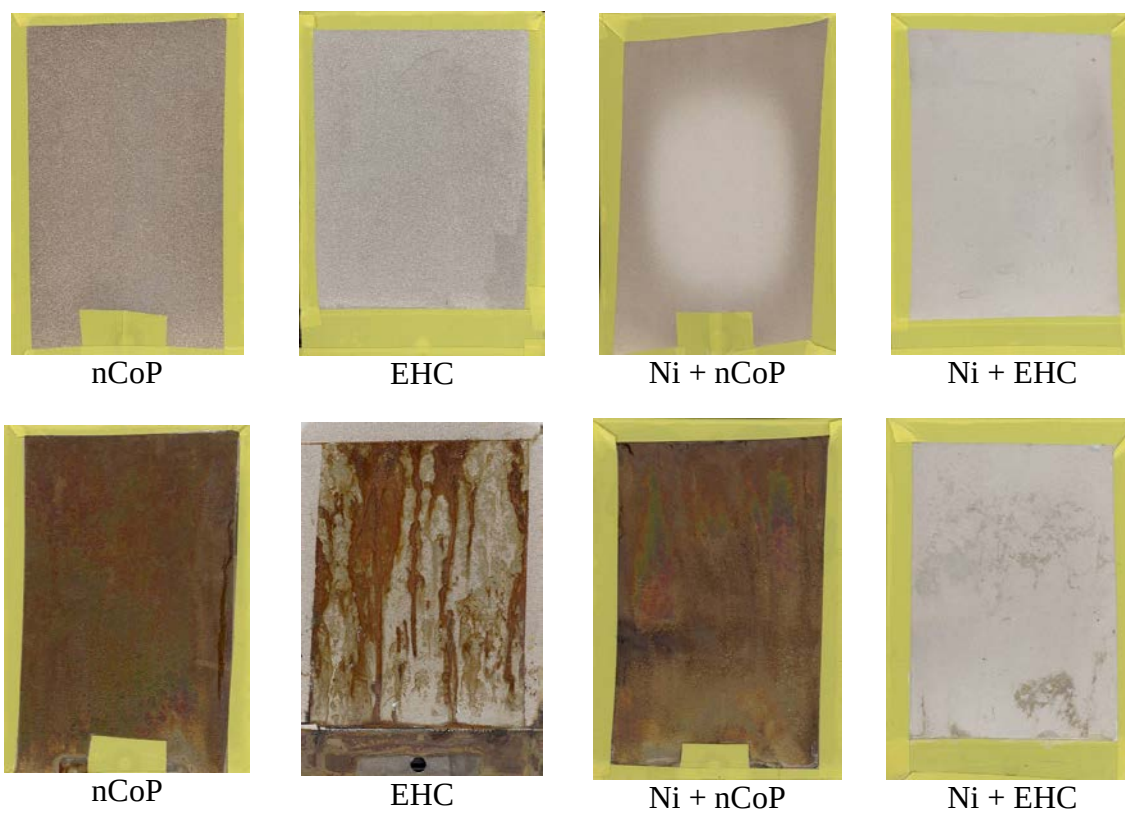


Figure 6-30. Representative images of coupons following exposure to acidic salt fog for 0 hrs (top) and 336 hrs (bottom).

6.1.6 Corrosion – Beach Exposure

A beach corrosion exposure evaluation was conducted at Kennedy Space Center (KSC). Coupons were mounted on test stands located 30 meters (100 feet) from the mean high-tide line and facing the water. The coupons were evaluated for substrate corrosion following 1, 2, 4, 8, 13 and 14 months from time of installation. At each examination time point, the appearance and protection rating was evaluated in accordance with ASTM B537.

After 1 month, EHC plated on steel samples showed signs of red rust. The EHC with Ni underlayer remained bright with no appearance of red rust. The nCoP with and without underplate developed a superficial light brown colored oxidation layer but protected the substrate from corrosion.

In subsequent inspections (i.e., 2, 4, 8, 13 and 14 months) significant red rust formed on the EHC samples while the EHC with Ni underlayer showed no appearance of red rust but became duller in appearance. For nCoP samples, the appearance was progressively darker which is associated with the increasing thickness of the surface oxide layer consistent with neutral salt spray testing.

Figure 6-31 and Figure 6-32 show the appearance and protection ratings, respectively, of EHC and nCoP coatings. Table 6-14 presents images of beach exposure samples on test racks at inspection periods of 2 and 14 months.

The results obtained meet the acceptance criteria.

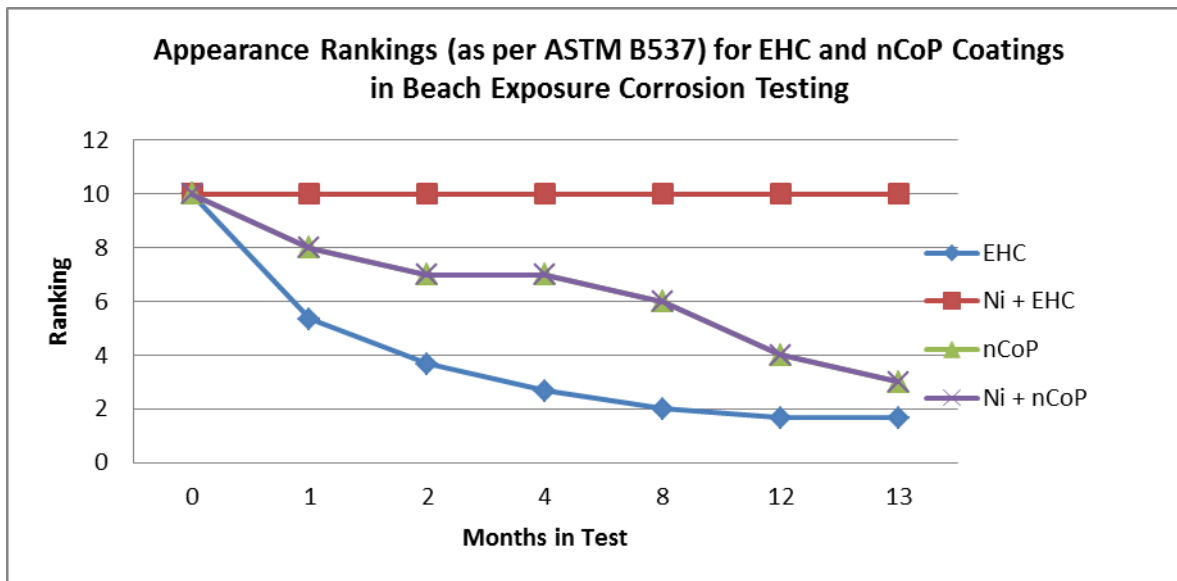


Figure 6-31. Appearance ranking for EHC and nCoP coatings in Beach Exposure Corrosion testing.

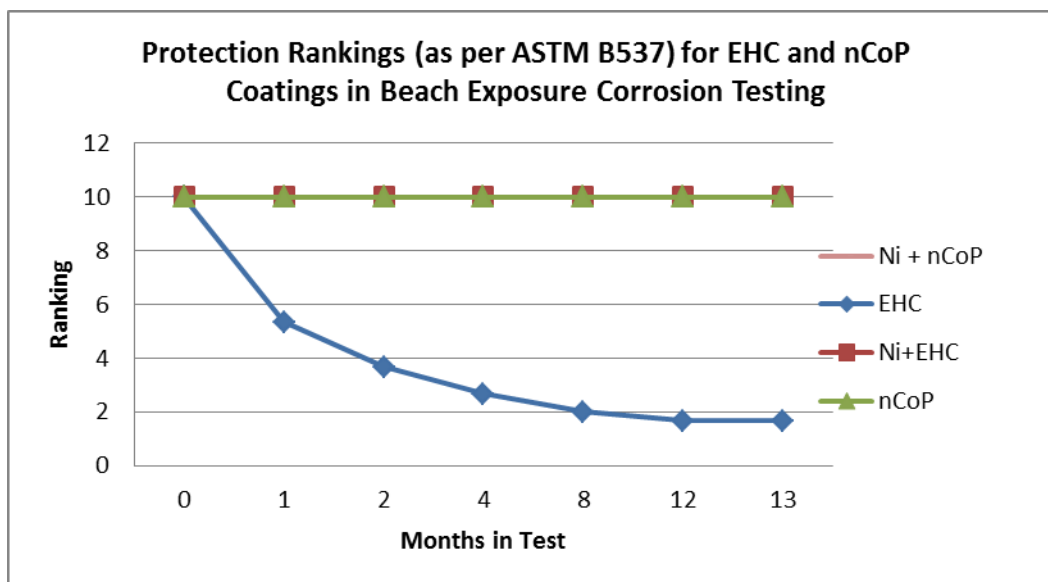


Figure 6-32. Protection ranking for EHC and nCoP coatings in Beach Exposure Corrosion testing.

Table 6-14. Images of beach exposure samples on test racks at inspection periods.

Coating type	Initial	Month 2	Month 14
nCoP			
EHC			
Ni + nCoP			
Ni + EHC			

6.1.7 Corrosion – Open Circuit Potential

The open circuit potential was determined for nCoP and EHC samples in neutral media. In all instances, mechanical polishing of the coatings using 1200 grit SiC was employed. In all instances, the working electrode was ultrasonically cleaned in isopropanol and rinsed with de-ionized water. The electrolytic test cell consisted of a commercially available glass cell, platinum wire counter electrode, an external saturated calomel reference electrode (SCE), a working electrode and a gas purge tube. Figure 6-33 shows a typical electrolytic test cell. The working electrode was inserted prior to testing. The SCE was interfaced to the test solution via salt bridge filled with the test solution. The bridge was connected to a Luggin probe with a porous glass tip that terminated 2mm from the working electrode surface. All corrosion tests were conducted in 0.5M NaCl in ambient air using an electrochemical interface controlled by a PC. The test solution was aerated. The open-circuit potential was monitored for 7 days to allow the working electrode to reach a steady state. The open-circuit potential was monitored for 1 day on an additional reference test on 4130 steel.

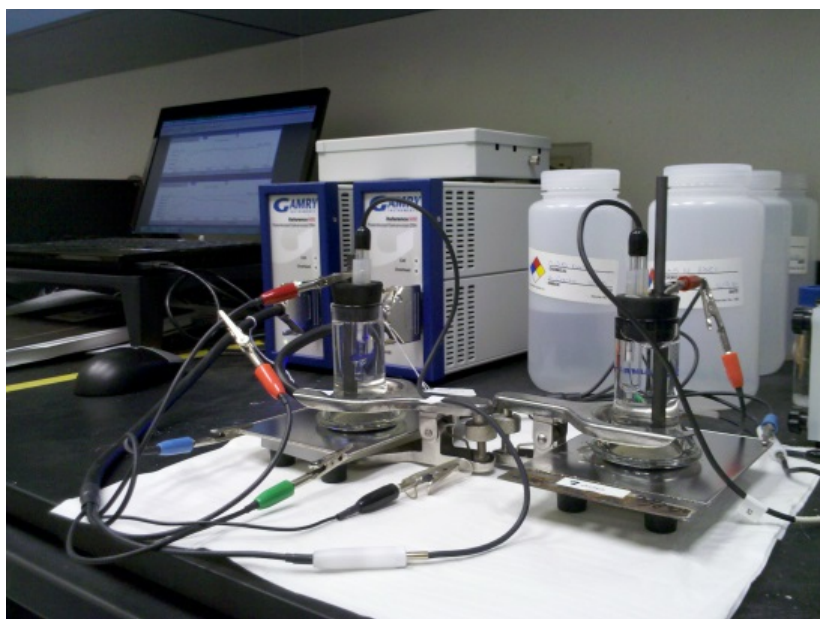


Figure 6-33. A typical electrolytic test cell with reference electrode, working electrode and counter electrode.

The measured open circuit potential was determined to be on average $-0.47V_{SCE}$ and $-0.60V_{SCE}$ for nCoP and EHC samples, respectively. Figure 6-34 and Figure 6-35 present the OCP monitoring curves for nCoP and EHC samples, respectively. For reference, Figure 6-36 shows the measured potential for 4130 steel substrates was $-0.77V_{SCE}$ after 1 day of monitoring. Table 6-15 presents the average and final OCP values for all samples tested. For nCoP samples, there is an initial ennoblement towards more passive potentials before reaching a stable value. For EHC samples, the initial potential reaches more active potentials before reaching a stable value, although some variability was seen across samples. This variation may be related to the micro-cracking present in the EHC coating.

Table 6-15. Open circuit potential monitoring for nCoP and EHC samples over 7 days.

Variable	Mean	StDev	Final OCP
OCP nCoP-1	-0.457	0.006	-0.451
OCP nCoP-2	-0.470	0.014	-0.469
OCP nCoP-3	-0.479	0.005	-0.484
OCP EHC-1	-0.595	0.011	-0.581
OCP EHC-2	-0.609	0.015	-0.619
OCP EHC-3	-0.596	0.015	-0.596

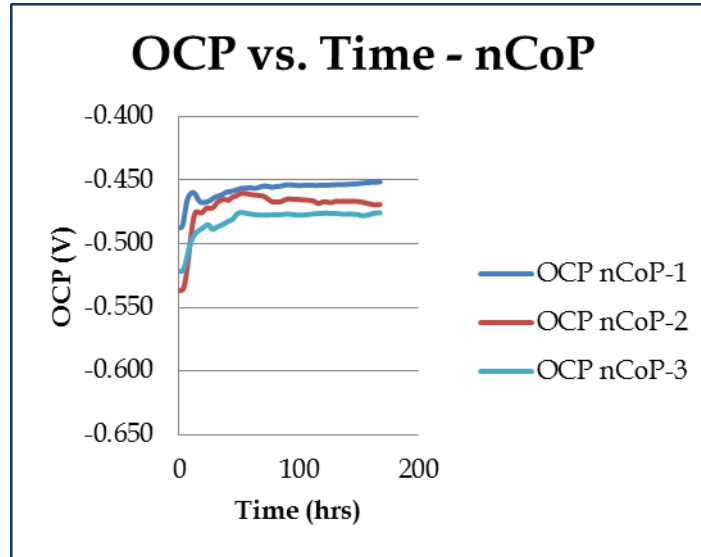


Figure 6-34. Open circuit potential (V_{SCE}) monitoring for 4130 steel/nCoP samples over 7 days.

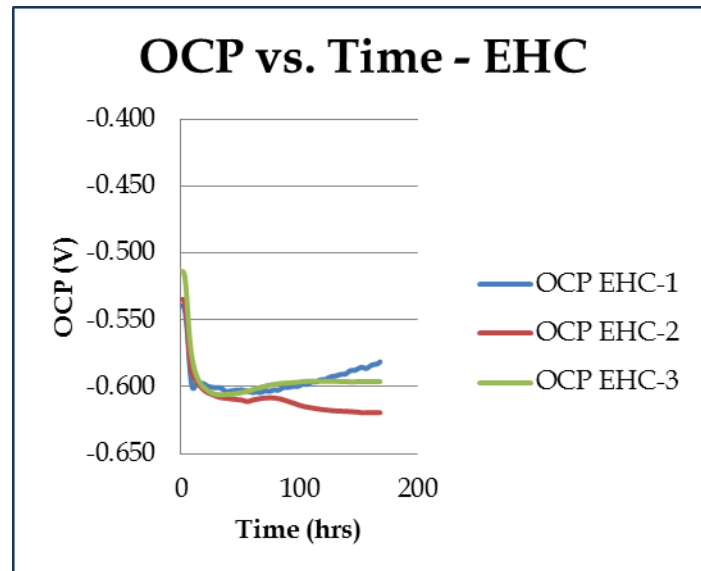


Figure 6-35. Open circuit potential (V_{SCE}) monitoring for 4130 steel/EHC samples over 7 days.

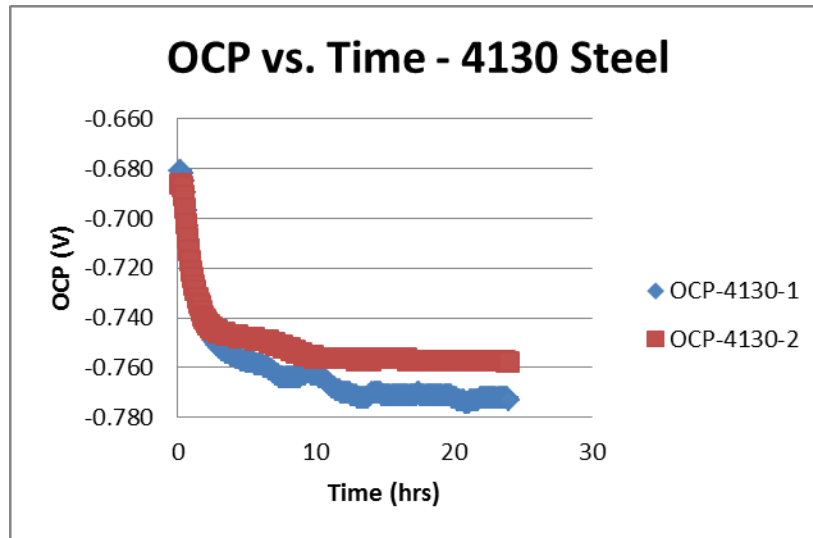


Figure 6-36. Open circuit potential (V_{SCE}) monitoring for 4130 steel over 1 day.

6.1.8 Adhesion

Adhesion testing was completed on substrates in order to validate that nCoP could be applied to various alloys applicable to aerospace applications. Samples were subjected to bend tests IAW ASTM B571. Figure 6-37 and Figure 6-38 show representative results of the adhesion testing of nCoP and EHC on 4130 substrates. As demonstrated in the images, both nCoP and EHC were found to be fully adherent to the underlying 4130 substrate with no evidence of flaking, delamination, or peeling of the deposited material.

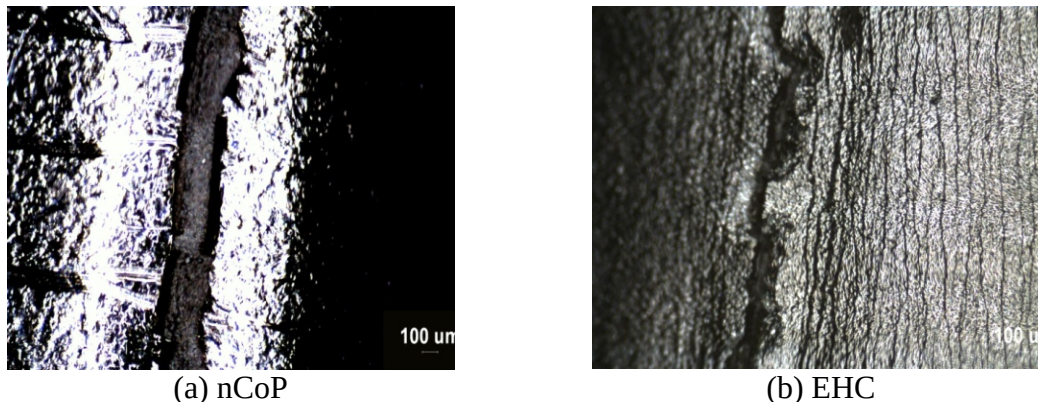


Figure 6-37. Representative images of (a) nCoP and (b) EHC deposits at the bend region following adhesion testing (4.6X).



Figure 6-38. Representative image of nCoP fracture surface following bending.

Table 6-16 summarizes results of the adhesion testing on applicable alloys. The nCoP deposit on all samples of 4130 and 1018 passed bend adhesion testing. The nCoP deposit on selected Hy-tuff samples (Hy-tuff-1, -3) passed bend adhesion testing. There was minor delamination of the nCoP coating on sample Hy-tuff-2 which indicated unsatisfactory adhesion of the coating as depicted in Figure 6-39. Smutting of the surface during surface activation/preparation prior to plating was observed, which is believed to have contributed to the adhesion failure. The nCoP/Ni sulfamate duplex plating on all samples of 4130 exhibited satisfactory adhesion following bend adhesion testing. The nCoP coating on all samples of 15-5PH, Inconel 718, Al 7075 and Aermet100 passed bend adhesion testing. Pitting was observed on some samples as noted. The pitting was found to be originating during surface preparation and not a result of the deposition process.



Figure 6-39. Image of Hy-tuff-2 which showed minor delamination.

Table 6-16. Adhesion testing results.

Test Sample Description	Coating	Pass/Fail	Coating Remarks
4130-1 (Integran)	EHC	Pass	N/A
4130-2 (Integran)	EHC	Pass	N/A
4130-3 (Integran)	EHC	Pass	N/A
4130-1 (Integran)	nCoP	Pass	N/A
4130-2 (Integran)	nCoP	Pass	N/A
4130-3 (Integran)	nCoP	Pass	N/A
4130-1	nCoP	Pass	N/A
4130-2	nCoP	Pass	N/A
4130-3	nCoP	Pass	N/A
1018-1	nCoP	Pass	Pitting observed
1018-2	nCoP	Pass	Pitting observed
1018-3	nCoP	Pass	Pitting observed
Hy-tuff-1	nCoP	Pass	Pitting observed
Hy-tuff-2	nCoP	Fail, minor delamination	Pitting observed
Hy-tuff-3	nCoP	Pass	Pitting observed
Ni Sulfamate 4130-1	nCoP	Pass	N/A
Ni Sulfamate 4130-2	nCoP	Pass	N/A
Ni Sulfamate 4130-3	nCoP	Pass	N/A
15-5PH-1	nCoP	Pass	N/A
15-5PH-2	nCoP	Pass	N/A
15-5PH-3	nCoP	Pass	N/A
Inconel 718-1	nCoP	Pass	N/A
Inconel 718-2	nCoP	Pass	N/A
Inconel 718-3	nCoP	Pass	N/A
Al 7075	nCoP	Pass	N/A
Al 7075	nCoP	Pass	N/A
Al 7075	nCoP	Pass	N/A
Aermet100*	nCoP	Pass	N/A
Aermet100*	nCoP	Pass	N/A
Aermet100*	nCoP	Pass	N/A

*leveraged efforts supported by Northrup Grumman Corporation. The successful process sequence utilized for adhesion to Aermet100 was mineral acid reverse etch (HF/H₂SO₄) followed by a Woods Ni strike.

6.1.9 Hydrogen Embrittlement

Hydrogen embrittlement testing was conducted using the sustained load test on coated notch round bar specimens. Overall, there were some relative performance differences noted for the nCoP coating compared to the EHC controls.

- For samples with no bake following coating, nCoP passed (4 of 4) while EHC failed (0 of 4) at a coating thickness of 0.003” as shown in Table 6-17. In contrast, for samples with standard hydrogen relief bake-out, nCoP failed (2 of 4) while EHC passes (4 of 4) at a coating thickness of 0.003” as shown in Table 6-18.
- The samples prepared with a sulfamate Ni underlayer (0.002”) and nCoP (0.003”) with standard hydrogen relief bake-out failed (0 of 4) while the samples prepared with a sulfamate Ni underlayer (0.002”) and EHC (0.003”) with standard hydrogen relief bake-out passed (4 of 4) as shown in Table 6-19. Figure 6-41 shows coating delamination for one sample of Ni + nCoP.
- For thicker builds (0.010”), both EHC and nCoP failed (0 of 4), while nCoP (0.020”) failed (2 of 4) as shown in Table 6-20. Application of thick builds (i.e. >0.003”) 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.
- Additional samples were prepared in order to determine if nCoP passed following a standard hydrogen embrittlement relief bake-out. For additional samples with standard hydrogen relief bake-out, nCoP passed (8 of 8) at a coating thickness of 0.003” as shown in Table 6-21.

Table 6-21 summarizes the results on 8 ea. additional notch round bar specimens coated to 0.003”. Bars were tested in batches of four by random selection. All 8 bars tested passed (4 of 4). The cause for the earlier failures is unknown, although in one instance plating defects were present (Figure 6-40 & Figure 6-41). It is recommended to perform additional tests in future in order to build confidence in application of hydrogen embrittlement relief bake-outs.

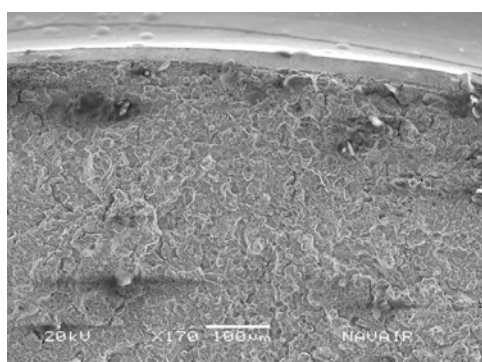
Table 6-17. Hydrogen embrittlement testing results for nCoP and EHC 1a.1 notched round bars at coating thickness of 0.003” with no bake.

Sample ID	Coating Type	Thickness	Heat Treatment	Fracture Load (%NFS)	Fracture time (hr)	Pass/Fail
HE-NCOP-NB-3-1(RSL)	nCoP	0.003”	No bake	90	202	Pass
HE-NCOP-NB-3-2(RSL)	nCoP	0.003”	No bake	90	202	Pass
HE-NCOP-NB-3-3(RSL)	nCoP	0.003”	No bake	91	203	Pass
HE-NCOP-NB-3-4(RSL)	nCoP	0.003”	No bake	90	202	Pass
HE-EHC-NB-3-1(RSL)	EHC	0.003”	No bake	20	3	Fail
HE-EHC-NB-3-2(RSL)	EHC	0.003”	No bake	20	3	Fail
HE-EHC-NB-3-3(RSL)	EHC	0.003”	No bake	30	5	Fail

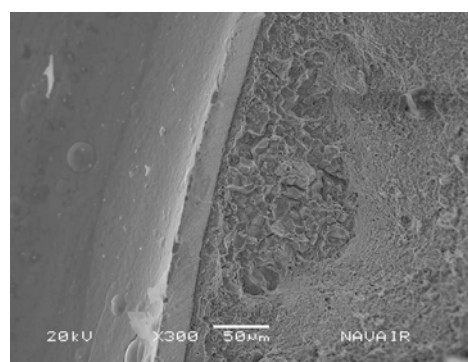
HE-EHC-NB-3-4(RSL)	EHC	0.003"	No bake	25	4	Fail
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Table 6-18. Hydrogen embrittlement testing results for nCoP and EHC 1a.1 notched round bars at coating thickness of 0.003" with bake.

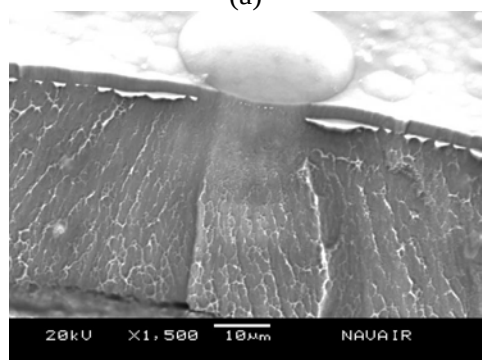
Sample ID	Coating Type	Thickness	Heat Treatment	Fracture Load (%NFS)	Fracture time (hr)	Pass/Fail
HE-NCOP-3-1	nCoP	0.003"	375°F for 24 hrs	80	201	Pass
HE-NCOP-3-2	nCoP	0.003"	375°F for 24 hrs	75	70	Fail
HE-NCOP-3-3	nCoP	0.003"	375°F for 24 hrs	90	202	Pass
HE-NCOP-3-4	nCoP	0.003"	375°F for 24 hrs	75	165	Fail
HE-EHC-3-1	EHC	0.003"	375°F for 24 hrs	92	204	Pass
HE-EHC-3-2	EHC	0.003"	375°F for 24 hrs	90	203	Pass
HE-EHC-3-3	EHC	0.003"	375°F for 24 hrs	94	204	Pass
HE-EHC-3-4	EHC	0.003"	375°F for 24 hrs	94	204	Pass



(a)



(b)

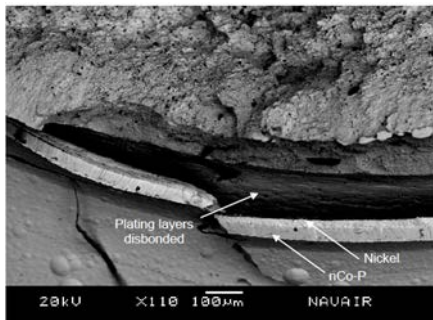


(c)

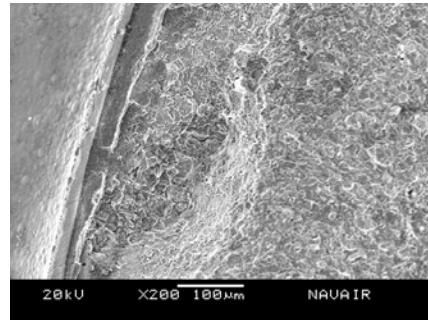
Figure 6-40. SEM cross-section of nCoP coating on HE-NCOP-3-2. A mixed mode intergranular/dimpled fracture surface is observed in (a) and (b). A plating defect (nodule) is present which may have led to premature failure observed in (c).

Table 6-19. Hydrogen embrittlement testing results for Ni + nCoP and Ni + EHC 1a.1 notched round bars at coating thickness of 0.005” total with bake.

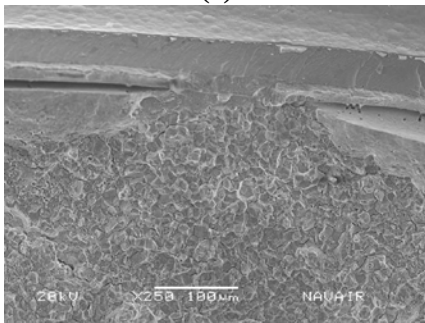
Sample ID	Coating Type	Thickness	Heat Treatment	Fracture Load (%NFS)	Fracture time (hr)	Pass/Fail
HE-NI/NCOP-3-1	Ni + nCoP	0.002” + 0.003”	375°F for 24 hrs	79	201	Pass
HE-NI/NCOP-3-2	Ni + nCoP	0.002” + 0.003”	375°F for 24 hrs	90	204	Pass
HE-NI/NCOP-3-3	Ni + nCoP	0.002” + 0.005”	375°F for 24 hrs	75	39	Fail
HE-NI/NCOP-3-4	Ni + nCoP	0.002” + 0.003”	375°F for 24 hrs	75	180	Fail
HE-NI/EHC-1	Ni + EHC	0.002” + 0.003”	375°F for 24 hrs	94	204	Pass
HE-NI/EHC-2	Ni + EHC	0.002” + 0.003”	375°F for 24 hrs	93	204	Pass
HE-NI/EHC-3	Ni + EHC	0.002” + 0.003”	375°F for 24 hrs	93	204	Pass
HE-NI/EHC-4	Ni + EHC	0.002” + 0.003”	375°F for 24 hrs	94	204	Pass



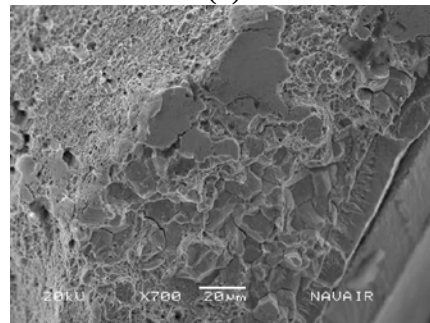
(a)



(b)



(c)



(d)

Figure 6-41. SEM cross-section of Ni + nCoP coating on HE-NI/NCOP-3-3. A coating disbond/cracking at the edge where the Ni layer separated from the steel specimen (a), however the nCoP/Ni has favorable cohesion to Ni. A mixed mode intergranular/dimpled fracture surface is observed (b thru d).

Table 6-20. Hydrogen embrittlement testing results for nCoP and EHC 1a.1 notched round bars at coating thickness of 0.010” and 0.020” with bake.

Sample ID	Coating Type	Thickness	Heat Treatment	Fracture Load (%NFS)	Fracture time (hr)	Pass/Fail
HE-NCOP-10-1	nCoP	0.010”	375°F for 24 hrs	75	2	Fail
HE-NCOP-10-2	nCoP	0.010”	375°F for 24 hrs	75	60	Fail
HE-NCOP-10-3	nCoP	0.010”	375°F for 24 hrs	75	<1	Fail
HE-NCOP-10-4	nCoP	0.010”	375°F for 24 hrs	74	0	Fail
HE-NCOP-20-1	nCoP	0.020”	375°F for 24 hrs	65	0	Fail
HE-NCOP-20-2	nCoP	0.020”	375°F for 24 hrs	75	200	Pass
HE-NCOP-20-3	nCoP	0.020”	375°F for 24 hrs	75	200	Pass
HE-NCOP-20-4	nCoP	0.020”	375°F for 24 hrs	65	0	Fail
HE-EHC-10-1	EHC	0.010”	375°F for 24 hrs	60	0	Fail
HE-EHC-10-2	EHC	0.010”	375°F for 24 hrs	71	0	Fail
HE-EHC-10-3	EHC	0.010”	375°F for 24 hrs	71	0	Fail
HE-EHC-10-4	EHC	0.010”	375°F for 24 hrs	58	0	Fail

Table 6-21. Repeat hydrogen embrittlement testing results for nCoP 1a.1 notched round bars at coating thickness of 0.003” with bake.

Sample ID	Coating Type	Thickness	Heat Treatment	Fracture Load (%NFS)	Fracture time (hr)	Pass/Fail
HE-NCOP-3-9	nCoP	0.003”	375°F for 24 hrs	84	200	Pass
HE-NCOP-3-10	nCoP	0.003”	375°F for 24 hrs	89	200	Pass
HE-NCOP-3-11	nCoP	0.003”	375°F for 24 hrs	92	203	Pass
HE-NCOP-3-12	nCoP	0.003”	375°F for 24 hrs	75	200	Pass
HE-NCOP-3-13	nCoP	0.003”	375°F for 24 hrs	75	200	Pass
HE-NCOP-3-14	nCoP	0.003”	375°F for 24 hrs	75	200	Pass
HE-NCOP-3-15	nCoP	0.003”	375°F for 24 hrs	75	200	Pass
HE-NCOP-3-16	nCoP	0.003”	375°F for 24 hrs	94	204	Pass

6.1.10 Fluid Compatibility

6.1.10.1 Maskant Evaluation

The following maskant evaluation aimed to understand if the maskants utilized in proceeding testing were resistant to the fluids for evaluation. Table 6-22 shows the mass change of the maskant compatibility coupons. With the exception of MIL-PRF-83282, no significant change in either the maskant, aside from staining, or fluids was seen. The maskant for MIL-PRF-83282 shows extremely heavy staining, and following immersion, the maskant was able to be picked from the sample. However, no significant mass change was seen and the fluid appeared in the same condition as prior to immersion. Removal of the maskant showed no damage to the underlying substrate indicating the maskant sufficiently protected the mild steel substrate. The Fasco Super Steelflex epoxy is considered sufficiently resistant to the fluids for use in nCoP fluid compatibility testing.

Table 6-22. Evaluation of maskant fluid compatibility coupons.

Fluid	Maskant Observation	Fluid Observation	Mass Change (g) (negative values correspond to material deposit)
MIL-PRF-83282	• Heavy orange/brown staining • Maskant appears brittle	• None	0.004
MIL-PRF-680	• None	• None	0.000
Fluorescent penetrant	• Slight yellow staining	• None	0.001
Nital	• Moderate yellow/brown staining	• None	-0.002
Ammonium persulfate	• None	• None	-0.004
Cimstar 40	• None	• None	
Bioact 280	• None	• None	-0.006
Chlorine Bleach	• Slight yellow staining	• None	-0.001

6.1.10.2 nCoP Fluid Immersion Testing

Table 6-23 summarizes the mass change and observations seen for the immersion of nCoP in common service and overall fluids. The nCoP shows resistance to most fluids. Nital etch, ammonium persulfate, and chlorine bleach caused attack to the nCoP coating.

Table 6-23. Evaluation of nCoP fluid compatibility coupons.

Fluid	Observations	Mass Change (g) (negative values correspond to material deposit)	Pass/Fail
MIL-PRF-83282	• Heavy orange/brown staining seen on all samples • Sample 2 showed delamination of the maskant along one edge • No macro- or microscopic changes seen to the coating • No changes seen to the fluid	-0.026 ± 0.038	Pass
MIL-PRF-680	• No macro- or microscopic changes seen to the coating • No changes seen to the fluid	0.000 ± 0.009	Pass
Fluorescent penetrant	• Slight yellow staining seen to the maskant on all samples • No macro- or microscopic changes seen to the coating • No changes seen to the fluid	0.001 ± 0.000	Pass
Nital	• Moderate purple/blue staining seen to the maskant • Roughening/etching of coating seen at macro scale • Clear evidence of chemical attack seen in microscopy • Solution turned from clear to purple hue	-0.095 ± 0.018	Fail
Ammonium persulfate	• Roughening/etching of coating seen at macro scale • Clear evidence of attack and change in surface texture seen in microscopy • Solution turned from clear to purple hue	-0.496 ± 0.006	Fail
Cimstar 40	• No macro- or microscopic changes seen to the coating • No changes seen to the fluid	-0.004 ± 0.001	Pass
Turco 4181L Alkaline Cleaner	• No macro- or microscopic changes seen to the coating • No changes seen to the fluid	-0.002 ± 0.002	Pass
MIL PRF 85570 type 2	• No macro- or microscopic changes seen to the coating • No changes seen to the fluid	-0.004 ± 0.001	Pass
Bioact 280	• No macro- or microscopic changes seen to the coating • No changes seen to the fluid	-0.001 ± 0.002	Pass
Chlorine Bleach	• Extreme powdered oxidation of surface seen at macro scale • Oxide non-adherent, red rust seen on some sections of coupon • Solution turned black and extensive bubbling seen with gas emission. Following, this solution turned clear • Container heated significantly to being warm to touch	-0.042 ± 1.181	Fail

6.1.10.3 EHC Fluid Immersion Testing

Nital etch, ammonium persulfate and chlorine bleach caused attack to the nCoP coating. To compare the above results with EHC coatings, compatibility of these fluids with EHC was tested using conditions previously outlined.

For these coupons the Fasco Super Steelflex epoxy did not provide adequate protection due to poor curing of the epoxy, an alternative maskant, Enthone EnPlate Stop-off, was used in this test. This maskant was sufficiently resistant to the subset of fluids being evaluated, as the condition of the maskant prior to and post testing was unchanged.

The EHC coating was resistant to attack in all three fluids. There was no significant mass change or degradation, visible by macro- or microscopic inspection, observed for any of the samples.

Table 6-24. Evaluation of EHC fluid compatibility coupons.

Fluid	Observations	Mass Change (g) (negative values correspond to material deposit)	Pass/Fail
Nital	• Sample 1 showed small amount of red rust at top of sample • No macro- or microscopic changes seen to the coating • No changes seen to the fluid	-0.002 ± 0.001	Pass
Ammonium persulfate	• No macro- or microscopic changes seen to the coating • No changes seen to the fluid	0.000 ± 0.000	Pass
Chlorine Bleach	• No macro- or microscopic changes seen to the coating • No changes seen to the fluid	-0.003 ± 0.001	Pass

6.1.11 Environmental Embrittlement

Environmental embrittlement testing was conducted using the sustained load test (45%NFS) on coated notch round bar specimens in DI water and synthetic seawater environments. Table 6-25 summarizes the results for nCoP and EHC samples at varying thicknesses and environments. Both nCoP and EHC samples subjected to DI water passed following 150hrs. The remainder of samples resulted in failures for all thicknesses and environments tested. The test is typically specified for sacrificial coatings. Therefore, the significance of this result is not well understood at this time.

This result passes the acceptance criteria.

Table 6-25. Environmental embrittlement testing results for nCoP and EHC 1a.1 notched round bars in D.I. Water or Salt Environments.

Sample ID	Coating/ Thickness	Environment	Fracture Load (%NFS)	Fracture time (hr)	Pass/Fail
HRE-NCOP-3-1	nCoP – 0.003” 375°F for 24hrs	D.I. Water	45	150 hrs +	Pass
HRE-NCOP-3-2	nCoP – 0.003” 375°F for 24hrs	D.I. Water	45	150 hrs +	Pass
HRE-NCOP-3-3	nCoP – 0.003” 375°F for 24hrs	D.I. Water	45	150 hrs +	Pass
HRE-NCOP-3-4	nCoP – 0.003” 375°F for 24hrs	D.I. Water	45	150 hrs +	Pass
HRE-EHC-3-1	EHC – 0.003” 375°F for 24hrs	D.I. Water	45	150 hrs +	Pass
HRE-EHC-3-2	EHC – 0.003” 375°F for 24hrs	D.I. Water	45	150 hrs +	Pass
HRE-EHC-3-3	EHC – 0.003” 375°F for 24hrs	D.I. Water	45	150 hrs +	Pass
HRE-EHC-3-4	EHC – 0.003” 375°F for 24hrs	D.I. Water	45	150 hrs +	Pass
HE-NCOP-3-5	nCoP – 0.003” 375°F for 24hrs	Salt Water	45	5 min.	Fail
HE-NCOP-3-6	nCoP – 0.003” 375°F for 24hrs	Salt Water	45	5 min.	Fail
HE-NCOP-3-7	nCoP – 0.003” 375°F for 24hrs	Salt Water	45	5 min.	Fail
HE-NCOP-3-8	nCoP – 0.003” 375°F for 24hrs	Salt Water	45	5 min.	Fail
HE-EHC-3-5	EHC – 0.003” 375°F for 24hrs	Salt Water	45	127 hrs	Fail
HE-EHC-3-6	EHC – 0.003” 375°F for 24hrs	Salt Water	45	5 min.	Fail
HE-EHC-3-7	EHC – 0.003” 375°F for 24hrs	Salt Water	45	10 min.	Fail
HE-EHC-3-8	EHC – 0.003” 375°F for 24hrs	Salt Water	45	10 min.	Fail
HRE-NCOP-10-1	nCoP – 0.010” 375°F for 24hrs	D.I. Water	45	1 min.	Fail

HRE-NCOP-10-2	nCoP – 0.010” 375°F for 24hrs	D.I. Water	45	1 min.	Fail
HRE-NCOP-10-3	nCoP – 0.010” 375°F for 24hrs	D.I. Water	45	1 min.	Fail
HRE-NCOP-10-4	nCoP – 0.010” 375°F for 24hrs	D.I. Water	45	1 min.	Fail
HRE-EHC-10-1	EHC – 0.010” 375°F for 24hrs	D.I. Water	45	10 min.	Fail
HRE-EHC-10-2	EHC – 0.010” 375°F for 24hrs	D.I. Water	45	20 min.	Fail
HRE-EHC-10-3	EHC – 0.010” 375°F for 24hrs	D.I. Water	45	150 hrs +	Pass
HRE-EHC-10-4	EHC – 0.010” 375°F for 24hrs	D.I. Water	45	150 hrs +	Pass
HRE-NCOP-10-5	nCoP – 0.010” 375°F for 24hrs	Salt Water	45	10 min.	Fail
HRE-NCOP-10-5	nCoP – 0.010” 375°F for 24hrs	Salt Water	45	50 min.	Fail
HRE-NCOP-10-7	nCoP – 0.010” 375°F for 24hrs	Salt Water	45	5 min.	Fail
HRE-NCOP-10-8	nCoP – 0.010” 375°F for 24hrs	Salt Water	45	10 min.	Fail
HRE-EHC-10-5	EHC – 0.010” 375°F for 24hrs	Salt Water	45	10 min.	Fail
HRE-EHC-10-6	EHC – 0.010” 375°F for 24hrs	Salt Water	45	15 min.	Fail
HRE-EHC-10-7	EHC – 0.010” 375°F for 24hrs	Salt Water	45	20 min.	Fail
HRE-EHC-10-8	EHC – 0.010” 375°F for 24hrs	Salt Water	45	20 min.	Fail

6.1.12 Wear - Taber

Taber wear testing was performed on EHC and nCoP samples at varying annealing conditions. An increase in microhardness is observed for nCoP samples following heat treatment at 375°F for 24 hrs and 550°F for 5 hrs. A summary of results is presented in Table 6-26 for both coatings tested using CS-17 (ASTM D4060) and H-22 (ASTM F1978) Taber Abraser Calibrade™ wheels (as shown in Figure 6-42). Figure 6-43 and Figure 6-44 summarizes results in TWI (mg/1000 cycles) for nCoP and EHC using CS-17 abrasive wheels. The EHC samples achieve an average TWI=3.3 as compared with nCoP samples which achieve TWI=17.9 (375°F for 24 hrs), TWI=18.0 (550°F for 5 hrs) and TWI=18.5 (as-deposited). Figure 6-45 summarizes results in TWI (mg/2000 cycles) for nCoP and EHC using a more abrasive H-22 wheel. Additional wear testing such as gravelometry was conducted in order to augment understanding of how nCoP performs when subjected to varying wear modes.

This result fails the acceptance criteria.

Table 6-26. Summary of results on Taber wear testing.

Test Configuration	Coating	Heat Treatment	Wear Loss	Taber Wear Index (TWI)
CS-17 Wheels (ASTM D4060)	nCoP	No Bake	18.4 mg / 1000 cycles	18.4
		HE Bake	17.9 mg / 1000 cycles	17.9
		Max Hardness	18.0 mg / 1000 cycles	18.0
	EHC	HE Bake	3.3 mg / 1000 cycles	3.3
H-22 Wheels (ASTM F1978)	nCoP	No Bake	145 mg / 2000 cycles	72.5 [†]
		HE Bake	123 mg / 2000 cycles	61.5 [†]
		Max Hardness	134 mg / 2000 cycles	67.0 [†]
	EHC	HE Bake	20 mg / 2000 cycles	10.0 [†]

[†] TWI for H-22 Wheels determined as (ave. wt loss in mg/2000 cycles)*1000



Figure 6-42: Image of Taber 5155 Abraser.

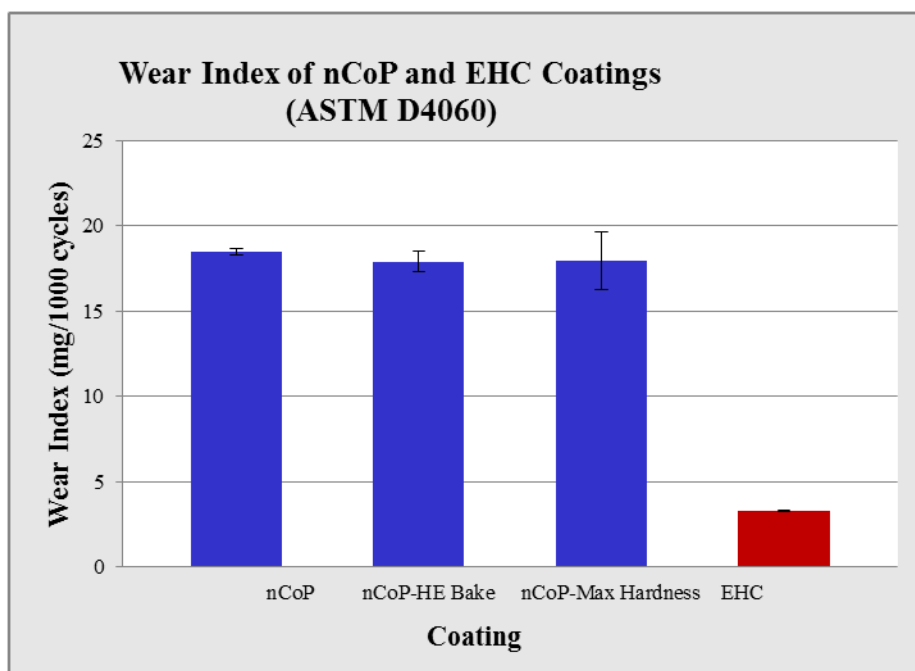


Figure 6-43. TWI for nCoP and EHC coatings using CS-17 wheels.

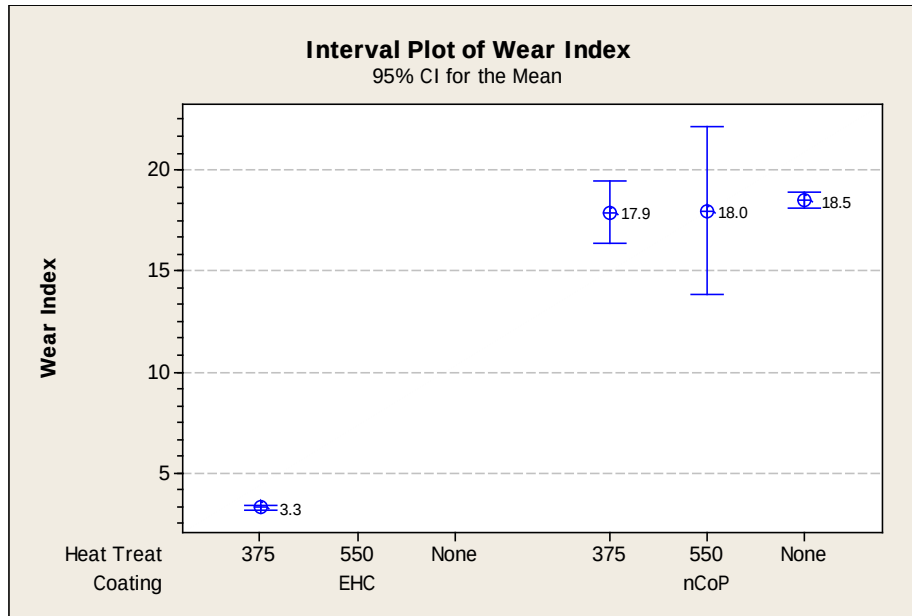


Figure 6-44. Interval plot compared TWI for nCoP and EHC coatings using CS-17 wheels.

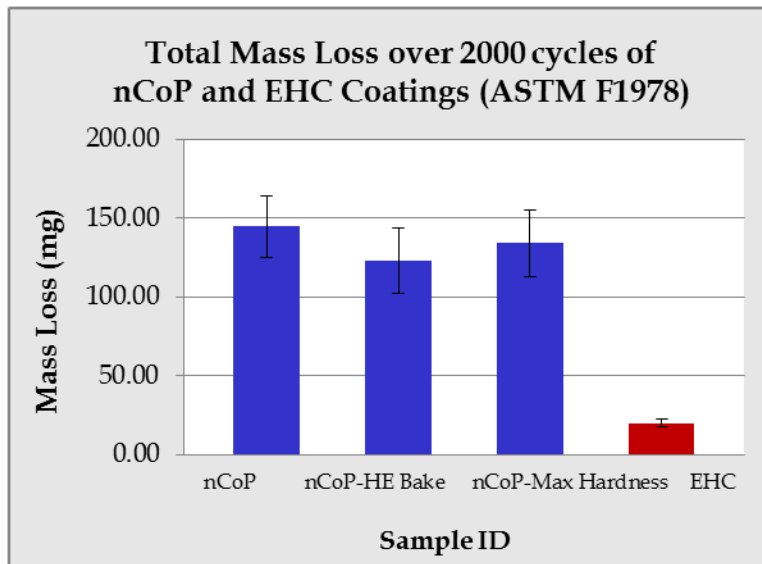


Figure 6-45. TWI for nCoP and EHC coatings using H-22 wheels.

6.1.13 Wear – Pin on Disk

Pin on disk wear testing was performed on EHC and nCoP samples at varying annealing conditions (disc) mated against 4130, 13-8 stainless steel, Al 7075, Cupronickel (70-30), nCoP and EHC (pin). The heat treatment of nCoP results in an increase in microhardness however, no clear trends were apparent among nCoP samples with no heat treatment, HE bake (375°F for 24 hrs) and max hardness (550°F for 5 hrs). The summary of results including sample volume loss (disc) and static partner volume loss (pin) are presented in Table 6-27 and Table 6-28. Note that the cumulative wear between the sample volume loss and static partner volume loss is the criteria for pass/fail. Refer to Figure 6-47 through Figure 6-52 for visual representation of data.

Table 6-27. Summary of results on Pin-on-Disc testing.

Static Partner Material	Coating	Heat Treatment	Coefficient of friction	Sample Volume Loss (mm ³)	Static Partner Volume Loss (mm ³)
nCoP coated on 4130	nCoP	-	0.362 ±0.059	0.027 ±0.013	0.007 ±0.005
		HE Bake	0.347 ±0.051	0.026 ±0.001	0.010 ±0.005
		Max Hardness	0.392 ±0.046	0.031 ±0.008	0.014 ±0.015
	EHC	HE Bake	0.421 ±0.001	Below Detection	0.101 ±0.013
EHC coated on 4130	nCoP	-	0.674 ±0.047	0.072 ±0.006	Below Detection
		HE Bake	0.636 ±0.046	0.067 ±0.008	Below Detection
		Max Hardness	0.650 ±0.089	0.077 ±0.000	Below Detection
	EHC	HE Bake	0.744 ±0.013	0.104 ±0.001	Below Detection
4130	nCoP	-	0.509 ±0.109	0.038 ±0.002	Below Detection
		HE Bake	0.483 ±0.047	0.032 ±0.006	Below Detection
		Max Hardness	0.479 ±0.041	0.039 ±0.032	Below Detection
	EHC	HE Bake	0.832 ±0.005	Below Detection	0.428 ±0.308
13-8 stainless	nCoP	-	0.440 ±0.017	0.036 ±0.003	Below Detection
		HE Bake	0.429 ±0.013	0.039 ±0.015	Below Detection
		Max Hardness	0.473 ±0.095	0.037 ±0.005	Below Detection
	EHC	HE Bake	0.900 ±0.013	Below Detection	0.481 ±0.136
7075 Al	nCoP	-	0.495 ±0.053	0.031 ±0.003	Below Detection
		HE Bake	0.424 ±0.049	0.032 ±0.004	Below Detection
		Max Hardness	0.461 ±0.063	0.035 ±0.009	Below Detection
	EHC	HE Bake	0.458 ±0.007	Below Detection	0.660 ±0.086
70-30 Cu-Ni	nCoP	-	0.453 ±0.029	0.023 ± 0.004	Below Detection
		HE Bake	0.554 ±0.148	0.027 ± 0.000	Below Detection
		Max Hardness	0.455 ±0.042	0.047 ± 0.001	Below Detection
	EHC	HE Bake	0.476 ±0.003	Below Detection	0.394 ±0.083

6.1.13.1 Coefficient of Friction

For all static partner materials the coefficient of friction when paired with all nCoP coatings is less than or equivalent to the coefficient of friction when the same material is paired with EHC coatings (Figure 6-46).

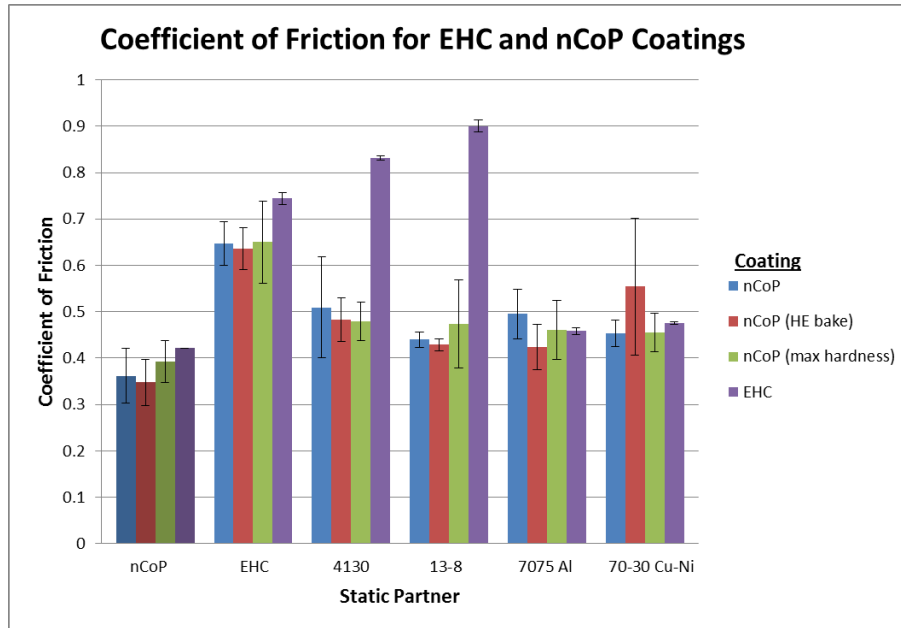


Figure 6-46. Coefficient of friction for nCoP and EHC mated against several static partners.

Table 6-28. Acceptance criteria for nCoP and EHC mated against several static partners.

Coating	Pin Material				
	EHC coated	4130	13-8 stainless	7075 Al	70-30 Cu-Ni
nCoP	✓PASS	✓PASS	✓PASS	✓PASS	✓PASS
nCoP (HE bake)	✓PASS	✓PASS	✓PASS	✓PASS	✓PASS
nCoP (Max hardness)	✓PASS	✓PASS	✓PASS	✓PASS	✓PASS

These results meet acceptance criteria.

6.1.13.2 Volume Wear Loss

Although EHC coatings did not show any material loss on the sample, except when mated against itself, the coating caused damage to the static partner in the form of volume loss. For all static partner materials, the volume loss caused to the nCoP coatings is significantly less than the volume loss inflicted on the static partner by EHC coatings. The losses on both the sample and the static partner are compared for nCoP and EHC coatings against all static partner materials in Figure 6-47 thru Figure 6-52.

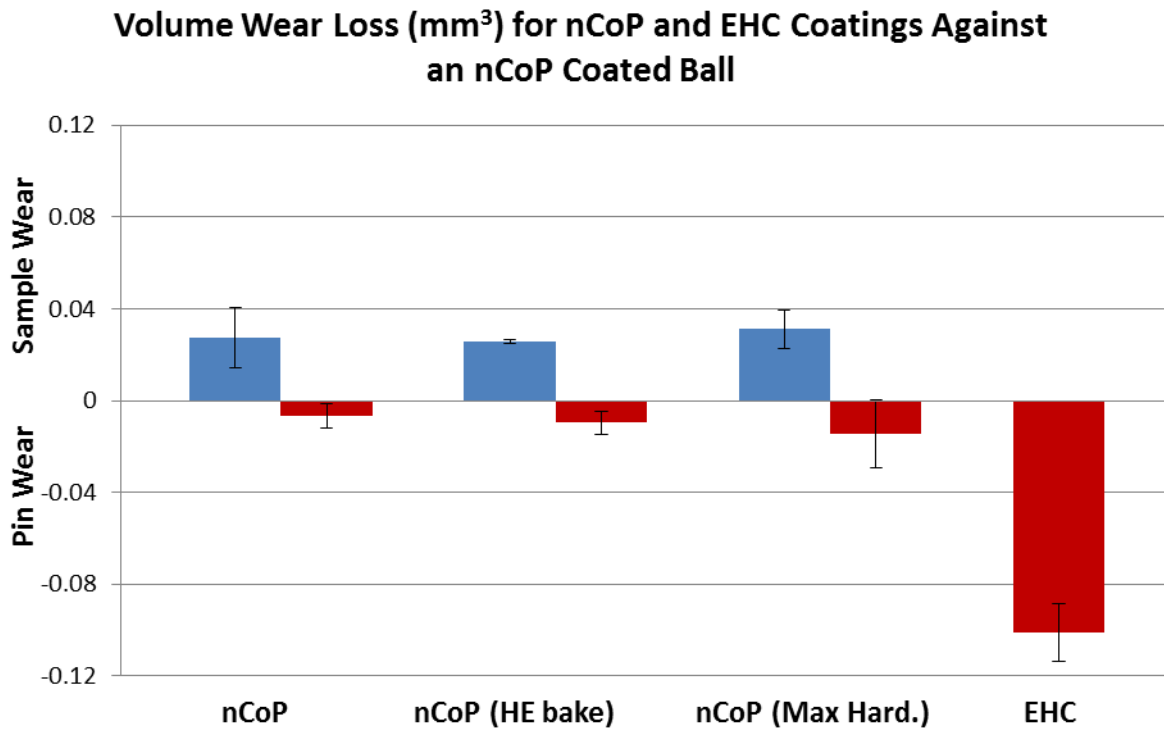


Figure 6-47. Volume wear loss of nCoP and EHC coatings against an nCoP coated ball.

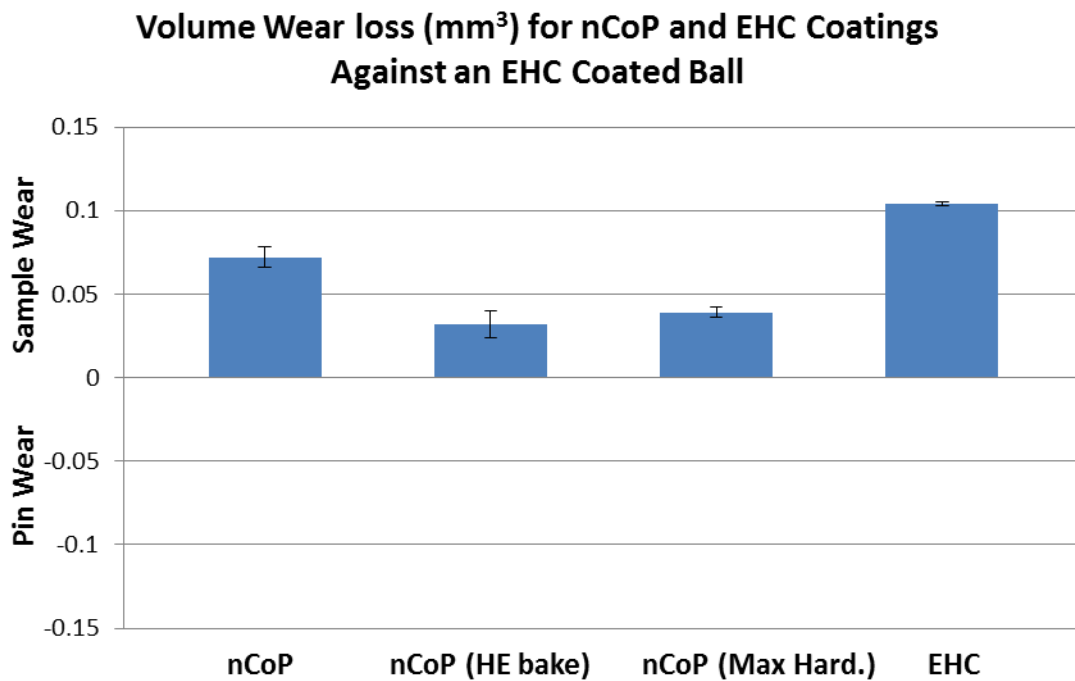


Figure 6-48. Volume wear loss of nCoP and EHC coatings against an EHC coated ball.

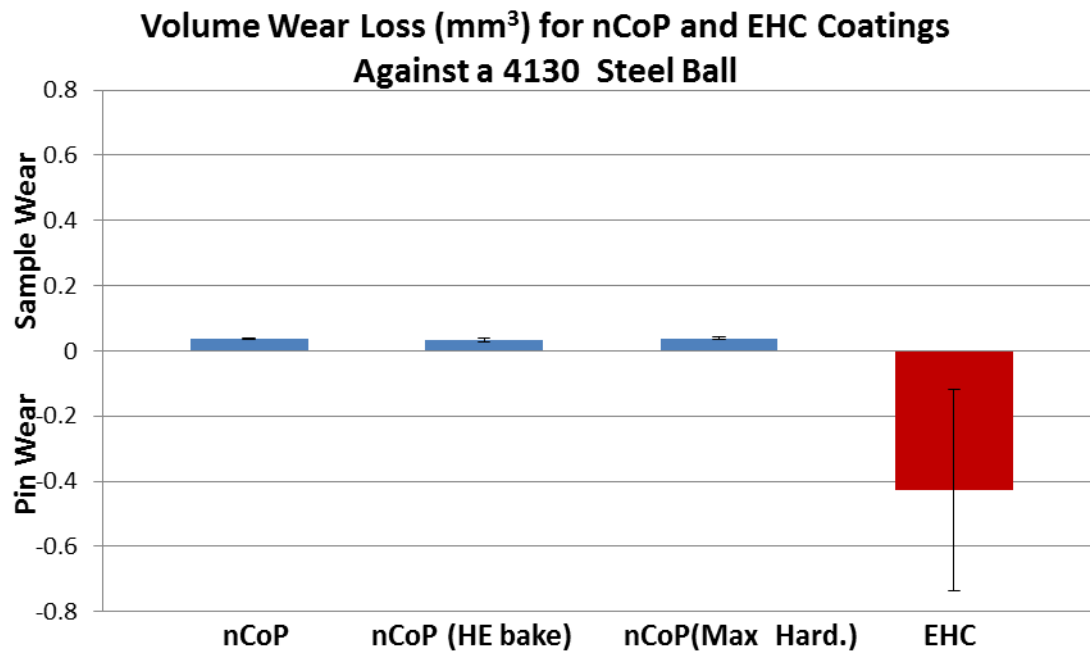


Figure 6-49. Volume wear loss for nCoP and EHC coatings against a 4130 steel ball.

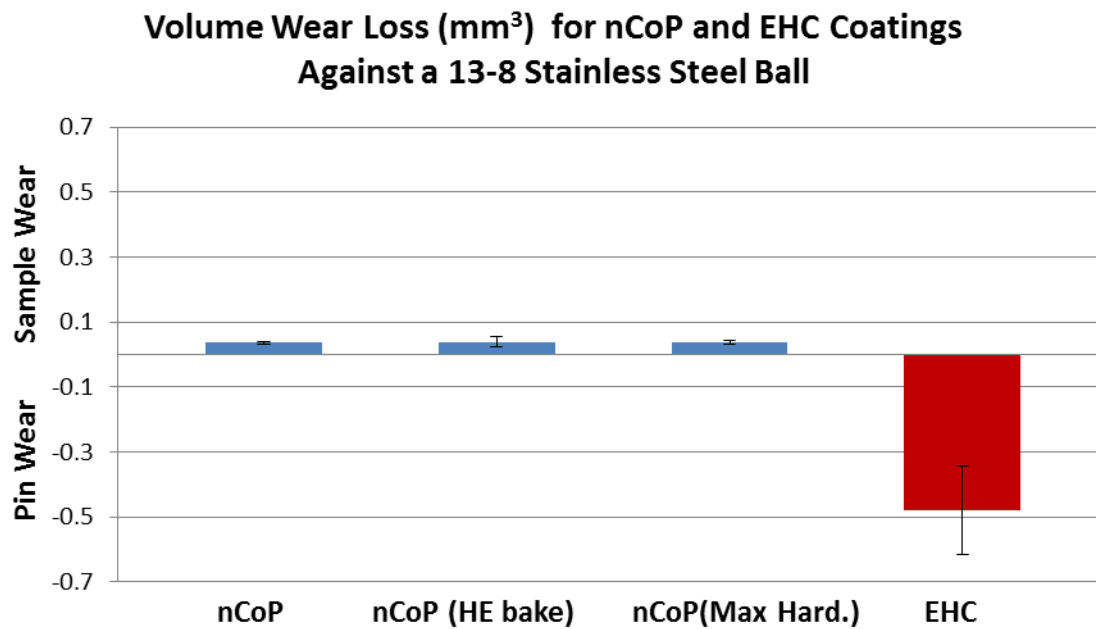


Figure 6-50. Volume wear loss for nCoP and EHC coatings.

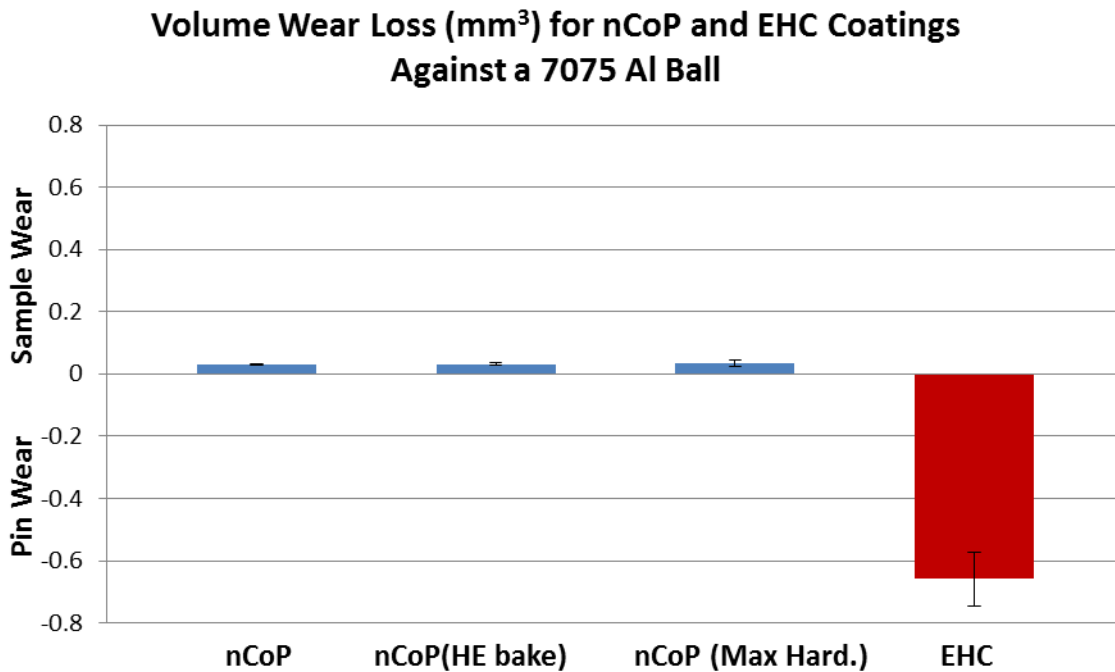


Figure 6-51. Volume wear loss for nCoP and EHC coatings against a 7075 Al ball.

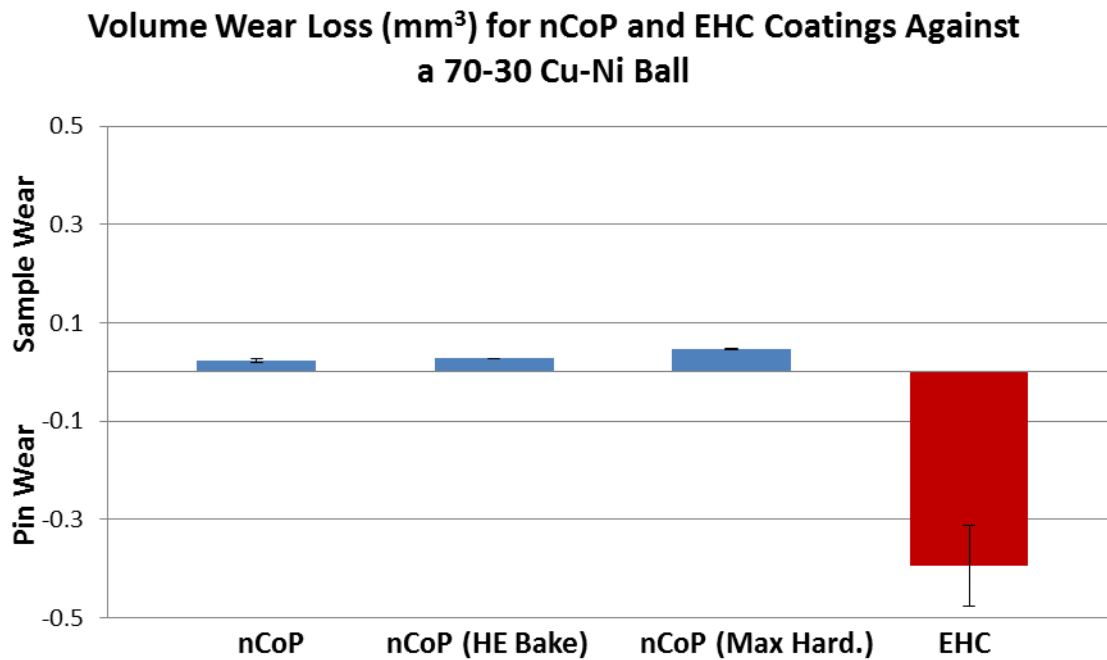


Figure 6-52. Volume wear loss for nCoP and EHC coatings against a 70-30 Cu-Ni ball.

6.1.14 Wear – Endurance Rig Test

The following testing was completed with Messier Bugatti Dowty. Actuator assemblies consisted of rods and cylinders with EHC and nCoP coatings with various surface finishes mated against different seal materials on an endurance rig test machine (see Figure 6-53). Due to malfunctioning of the testing rig, several conditions as stated in the JTP could not be tested.

6.1.14.1 Test article configuration

For each actuator assembly, the coating type and surface roughness of the rods and cylinders, and the seal configuration is present in Table 6-29.

During the testing of actuator #2 and #3, it was discovered that the piston seals had extruded outside of the seal groove. For testing involving actuator #5 and #9, backup rings (P/N 7218MR-P8) were used in conjunction with Buna-N Tee Seals to prevent seal extrusion.

Table 6-29. Test article configuration.

Actuator	Rod S/N	Cylinder S/N	Coating	Surface Finish (Microinches)	Piston Seal	Rod Seal
1	R01	C01	EHC	12-16	Buna-N Tee Seal Nitrile Butadiene Rubber P/N 7218MR-964-P3	Buna-N Tee Seal Nitrile Butadiene Rubber P/N 7114FT-964-P3
2	R02	C02	EHC	4-6		
3	R03	C03	nCoP	12-16		
4	R04	C04	nCoP	4-6		
5	R05	C05	nCoP with HE Bake	12-16		
9	R09	C09	EHC	12-16		
11	R11	C11	EHC	12-16	O-Ring/Cap PTFE Cap P/N TF936M-218N902P (O-Ring, P/N M83461/1-218, Included)	Spring Energized PTFE P/N C1988L114-902C902
12	R12	C12	nCoP	12-16		

6.1.14.2 Chronology of Testing

2012 Aug	–	Actuator #1 and #2 (incomplete, #1 was damaged)
2013 Feb to Mar	–	Actuator #3 and #4 (incomplete, #4 was damaged)
2013 Jul to Aug	–	Actuator #2 and #3 (incomplete, seal extrusion)
2013 Aug to Oct	–	Actuator #11 and #12 (complete)
2013 Dec	–	Actuator #5 and #9 (complete)

6.1.14.3 Summary

6.1.14.3.1 Actuator #1 and #2 (incomplete, actuator #1 was damaged)

During testing, the connection between the lever arm and the displacement actuator was sheared and broken. The displacement actuator then shifted, and its top end was caught and jammed by the rod end of the lower actuator (actuator #1). As the displacement actuator extended, it applied pressure on the side of the rod end. Consequently, the rod end of the lower actuator bent under pressure load.

6.1.14.3.2 Actuator #3 and #4 (incomplete, actuator #4 was damaged)

Following the increase in pressure inside the actuators and the hydraulic fluid temperature, the lower actuator (actuator #4) went into a hydraulic lock state during further testing. It was deduced that when the displacement actuator was retracting, and it overpowered the resistance of the hydraulically locked lower actuator to the point that the piston rod of the lower actuator buckled and broke in half

6.1.14.3.3 Actuator #2 and #3 (incomplete)

At around cycle 2400/20000, it was noticed that there was heat build-up in the lower actuator (actuator #3), and it was warmer than the upper actuator (actuator #2). Later, a “clicking” noise was heard from the test rig. With the excessive amount of heat from the lower actuator and the “clicking” noise from the upper one, it was decided to disassemble the two actuators. Upon disassembling, it was discovered that the seal/back-up rings on the piston head had severely extruded. The test was aborted.

6.1.14.3.4 Actuator #11 and #12 (complete)

On both actuators, the coatings exhibited some surface wear marks, but appeared to be in good condition. The surfaces of the rods felt smoother in the wear test region compared to the unworn portions, but had no measurable wear depth.

Actuator #11 had the EHC coating on the cylinder and the rod and exhibited more leakage than actuator #12 (58 cycles/drop vs. ~330 cycles/drop). The measure of surface roughness before the test was omitted unintentionally, but after testing the rod of actuator #11 had an indicated roughness in the order of 7 to 8 μin average R_a in the wear test region, compared to 11 μin average R_a in an unworn portion of the rod. The rod seal could not be removed for closer inspection without destroying it. The piston seal cap had formed a lip on both edges, extruding into the gap between the seal groove shoulder of the piston and the ID of the cylinder. The cylinder of actuator #11 had an indicated roughness in the order of 1.6 to 2.3 μin average R_a in the wear test region, compared to 4.1 μin average R_a in an unworn portion of the cylinder.

Actuator #12 had the nCoP coating on the cylinder and the rod. After testing, the rod of actuator #12 had an indicated roughness in the order of 3 to 5 μin average R_a in the wear test region, compared to 7 μin average R_a in an unworn portion of the rod. As with the other actuator, the rod seal could not be removed for closer inspection without destroying it, and the piston seal cap had formed a lip on both edges, extruding into the gap between the seal groove shoulder of the piston and the ID of the cylinder. The piston seal on actuator #12 exhibited more wear than that of actuator #11, with the annular rings on the sealing surface of the cap having been smoothed

out in places, with an increase in the amount of extrusion. The cylinder of actuator #12 had more visible wear marks. The indicated roughness was in the order of 5.6 to 7.7 μin average R_a in the wear test region, compared to 8.2 μin average R_a in an unworn portion of the cylinder.

6.1.14.3.5 Actuator #5 and #9 (completed)

On both actuators, the coatings exhibited some surface wear marks, but appeared to be in good condition. The surfaces of the rods felt smoother in the wear test region compared to the unworn portions, but had no measurable wear depth.

Actuator #5 had the nCoP coating on the cylinder and the rod, and exhibited more leakage than actuator #9 (510 cycles/drop vs. 209 cycles/drop). The rod of actuator #5 had an indicated roughness in the order of 11.0 to 12.5 μin average R_a before the test. After the test, the indicated roughness on the rod was in the order of 7.2 to 9.9 μin average R_a in the wear test region. The piston seal did not exhibit any sign of damage. There was a very small amount of feathering on the piston seal back-up rings. The rod seal also showed no sign of damage. There was slight feathering on the rod-side back-up ring. The cylinder of actuator #5 had an indicated roughness in the order of 8.1 to 11.7 μin average R_a before test. After the test, the indicated roughness was in the order of 7.6 to 9.0 μin average R_a in the wear test region.

Actuator #9 had the EHC coating on the cylinder and the rod. The rod of actuator #9 had an indicated roughness in the order of 11.0 to 12.1 μin average R_a before the test. After the test, the indicated roughness on the rod was in the order of 10.0 to 11.1 μin average R_a in the wear test region. Same as that of actuator #5, the piston seal showed no sign of damage. The piston seal back-up rings exhibited slightly more feathering than those of actuator #5. The rod seal also showed no sign of damage. There was an observable reduction in thickness and surface deterioration of the rod-side back-up ring. The cylinder of actuator #9 had an indicated roughness in the order of 3.5 to 3.9 μin average R_a before test. After the test, the indicated roughness was in the order of 2.7 to 3.7 μin average R_a in the wear test region.

Both nCoP coated and EHC coated articles met the acceptance criteria of less than one drop of hydraulic fluid in 25 cycles and acceptable wear as defined as wear not affecting leakage performance (Table 6-30 and Figure 6-54).

Table 6-30. Summary of results on endurance rig testing.

Actuator	Coating	Piston Seal	Rod Seal	Surface Finish On Rod ($\mu\text{in. } R_a$)		Surface Finish on Cylinder ($\mu\text{in. } R_a$)		Leakage Rate (cycles/drop)	Acceptance Criteria Pass/Fail (>25 cycles/drop)
				Before	After	Before	After		
5	nCoP with HE Bake	Buna-N Tee Seal Nitrile	Buna-N Tee Seal Nitrile	11.8	8.6	9.9	8.3	209	Pass
9	EHC	Butadiene Rubber P/N 7218MR-964-P3	Butadiene Rubber P/N 7114FT-964-P3	11.6	10.6	3.7	3.2	510	Pass

11	EHC	O-Ring/Cap PTFE Cap P/N	Spring Energized PTFE P/N	11	7.5	4.1	2.0	58	Pass
12	nCoP	TF936M- 218N902P (O-Ring, P/N M83461/1- 218, Included)	C1988L114- 902C902	7	4	8.2	6.7	330	Pass



Figure 6-53. Image of the rod-seal wear testing rig at Messier-Bugatti-Dowty.



Rod #11 (EHC)



Rod #12 (nCoP)



Piston Seal #11 (EHC)



Piston Seal #12 (nCoP)

Figure 6-54. Images of the rods and piston seals after testing.

6.1.15 Wear – Block on Ring

This test was conducted to provide information on manufacturing variables and wear conditions including coating material, surface finish, lubrication, velocities, type of wear, and other wear environment factors with a minimum number of tests. The performance of nCoP coatings in terms of weight loss and wear volume was compared to that of EHC.

Samples were prepared by depositing EHC or nCoP coatings to the outer diameter of rings and blocks to a minimum thickness of 0.006”. All samples were ground and polished to a coating thickness of 0.003”- 0.005” and to a surface finish of 4 to 8 microinches or finer IAW MIL-STD-866.

As part of the results presented, only tests which lasted longer than 5 minutes were used for analysis. The first 5 minutes could be considered a break in period. Positive wear rates show the material was wearing away. Negative wear rates show material deposition.

6.1.15.1 Phase I: Plating on Both Block and Ring

The first phase of testing showed little in terms of performance differences between the various material options (see Table 6-31). The testing showed good performance characteristics for all plating options against each other. When both the block and ring were plated with EHC, the friction and wear loss was greater, though still very close to the other combinations.

Table 6-31. Phase I testing results on 4130 steel/nCoP (block) and 4620 steel/EHC coated (ring) specimens.

Block Coating	nCoP		nCoP		nCoP		EHC	
Ring Coating	EHC		nCoP (HE bake)		nCoP (Max hardness)		EHC	
Avg. CoF	0.133		0.130		0.133		0.146	
Avg. Wear Rate of Block	1.27x10 ⁻⁸	g/cycle	0.168x10 ⁻⁸	g/cycle	-4.63x10 ⁻¹⁰	g/cycle	-1.34x10 ⁻⁸	g/cycle
	0.127	µg/1000 cycles	0.017	µg/1000 cycles	-0.046	µg/1000 cycles	-0.134	µg/1000 cycles
Avg. Wear Rate of Ring	-6.17x10 ⁻¹⁰	g/cycle	1.91x10 ⁻⁹	g/cycle	1.62x10 ⁻⁹	g/cycle	1.13x10 ⁻⁷	g/cycle
	0.062	µg/1000 cycles	0.191	µg/1000 cycles	0.162	µg/1000 cycles	1.13	µg/1000 cycles

6.1.15.2 Phase II: Plating on Ring, PH13-8Mo Steel Block

Against PH13-8Mo steel, the nCoP plating, both HE bake and max hardness, had lower friction and block wear (see Table 6-32). The EHC plating friction was significantly higher, 0.147 average compared to 0.133 average. The EHC plating ring specimen also had a negative wear rate, indicating that the steel was being deposited on the ring during testing.

Table 6-32. Phase II testing results on coated ring specimens 4620 steel/nCoP and 4620 steel/EHC (rings) mated against PH13-8Mo Steel (block).

Block Material	PH13-8Mo Steel		PH13-8Mo Steel		PH13-8Mo Steel	
Ring Coating	EHC		nCoP (HE bake)		nCoP (Max hardness)	
Avg. CoF	0.147		0.134		0.132	
Avg. Wear Rate of Block	3.72x10 ⁻⁸	g/cycle	5.96x10 ⁻¹⁰	g/cycle	5.17x10 ⁻¹⁰	g/cycle
	0.372	µg/1000 cycles	0.006	µg/1000 cycles	0.005	µg/1000 cycles
Avg. Wear Rate of Ring	-2.29x10 ⁻⁹	g/cycle	5.04x10 ⁻⁹	g/cycle	9.55x10 ⁻⁹	g/cycle
	-0.023	µg/1000 cycles	0.050	µg/1000 cycles	0.096	µg/1000 cycles

6.1.15.3 Phase III: Plating on Ring, Al-Ni-Bronze Block

The HE bake nCoP plating was the superior performer in this series of testing (see Table 6-33). Both the friction and block wear rates were significant improvements over the other options. The EHC plating showed much higher friction and block wear. The EHC plated ring showed material deposition. The max hardness nCoP plating performance was between the other two material combinations.

Table 6-33. Phase III testing results on coated ring specimens 4620 steel/nCoP and 4620 steel/EHC (rings) mated against Al-Ni-Bronze (block).

Block Material	Al-Ni-Bronze		Al-Ni-Bronze		Al-Ni-Bronze	
Ring Coating	EHC		nCoP (HE bake)		nCoP (Max hardness)	
Avg. CoF	0.159		0.137		0.148	
Avg. Wear Rate of Block	3.03x10 ⁻⁷	g/cycle	1.98x10 ⁻⁹	g/cycle	6.75x10 ⁻⁸	g/cycle
	3.03	µg/1000 cycles	0.020	µg/1000 cycles	0.678	µg/1000 cycles
Avg. Wear Rate of Ring	-2.01x10 ⁻⁹	g/cycle	2.31x10 ⁻⁷	g/cycle	3.12x10 ⁻⁷	g/cycle
	-0.020	µg/1000 cycles	2.31	µg/1000 cycles	3.12	µg/1000 cycles

The nCoP performed similar or better than EHC in most of the wear testing completed during this investigation. There was not any performance increase for the nCoP that had been heat treated to a max hardness condition; some of the tests showed the max hardness as performing worse than the HE bake specimens.

According to the test report prepared by The Boeing Corporation, the nCoP coating performed similar or better than EHC in most of the wear testing completed during this investigation. It was noted that nCoP and EHC samples exhibited flaking and cracking following testing. This is likely to be related to sample preparation or grinding.

The measured coefficient of friction was lower for nCoP compared to EHC for all combinations evaluated. The average wear rate varied greatly for various sample/material combinations.

This result passes the acceptance criteria.

6.1.16 Wear – Gravelometry

Gravelometry testing was performed on EHC and nCoP coated samples. Photographs were taken of the twelve test specimens before and after the gravelometry test. Areas with staining or other defects in the plating were marked on samples. Following gravelometry testing, marked areas were not considered.

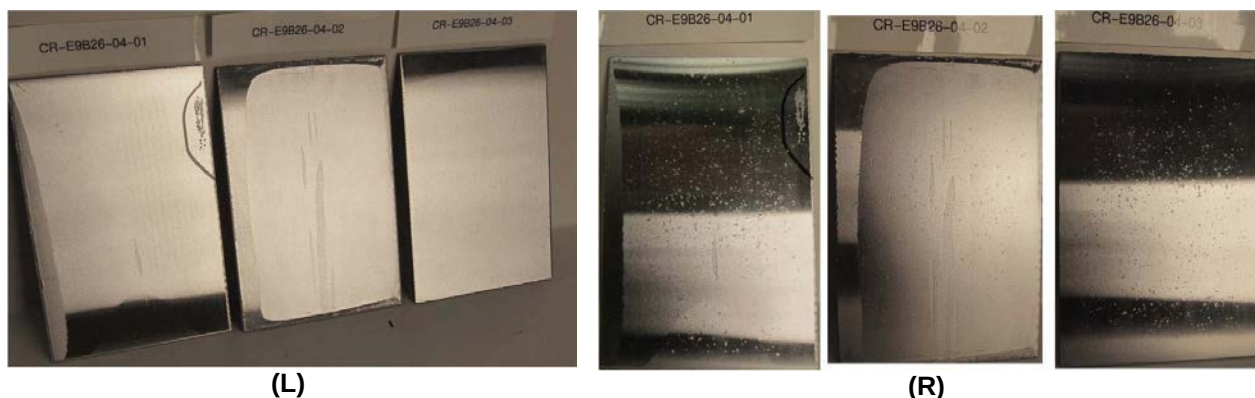


Figure 6-55. EHC Plated (with HE Bake) Test Specimens Before (L) and After (R) Gravelometry Testing.

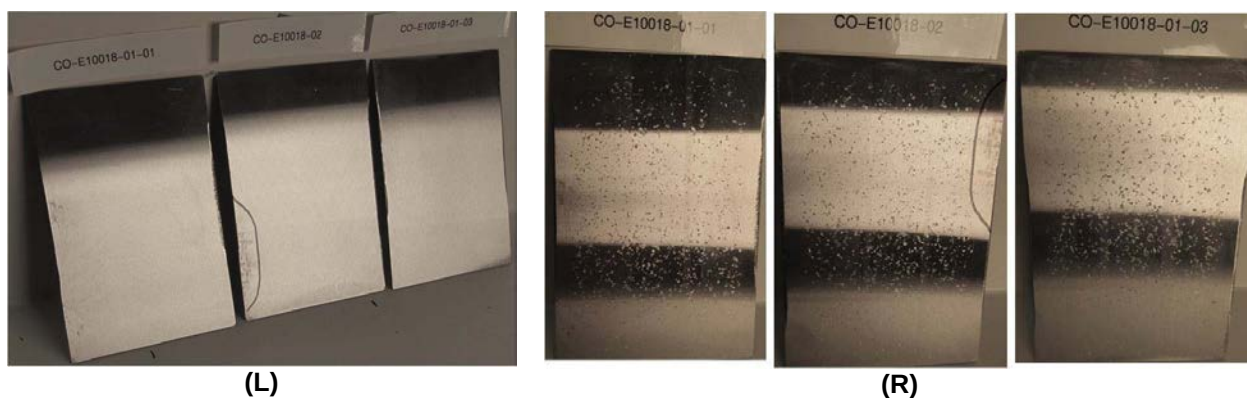


Figure 6-56. nCoP Plated (with no HE Bake) Test Specimens Before (L) and After (R) Gravelometry Testing.

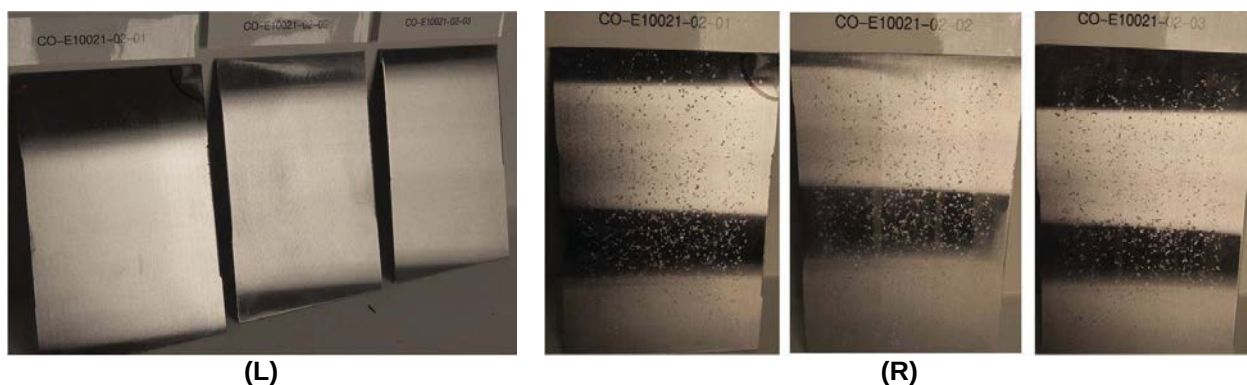


Figure 6-57. nCoP Plated (with HE Bake) Test Specimens Before (L) and After (R) Gravelometry Testing.

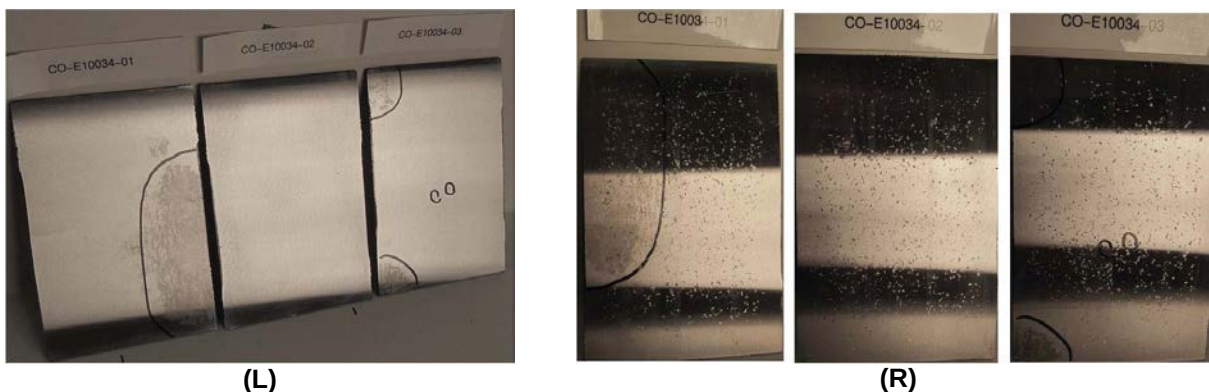


Figure 6-58. nCoP Plated (with Max Bake) Test Specimens Before (L) and After (R) Gravelometry Testing.

Figure 6-55 thru Figure 6-58 show that small dents were found on all 12 of the EHC and nCoP plated test specimens, but no chips or plating removal were found on any of the test specimens. In addition, no chrome or nCoP plating could be removed from the gravelometry test specimens using the 3M #250 tape. All 12 test panels passed the gravelometry test.

Three Almen N test strips were placed in the gravelometry test cabinet and subjected to the 10 second controlled gravel impact. There was no deflection resulting from the impact of the gravelometry testing – the three Almen N test strips were still flat. Figure 6-59 shows the three Almen N test strips after gravelometry testing. Test strips shown may appear to be bowed, but they are actually flat.



Figure 6-59. Almen N Test Strips After Gravelometry Testing.

Metallographic cross-sections were made of representative samples taken from the four different plating conditions:

- EHC (HE Baked) – Test Specimen CR-E9B26-04-01
- nCoP (No Bake) – Test Specimen CO-E10018-01-02
- nCoP (HE Baked) – Test Specimen CO-E10021-02-03
- nCoP (Max Hardness) – Test Specimen CO-E10034-03-03

Figure 6-60 show the photographs of the four metallographic cross-sections prepared. The thickness of the plating is shown on the photograph. A nital etch was used to improve the contrast and accurately determine the interface between the plating and the steel substrate. The nCoP plating was thin compared to the EHC coated specimens. This was probably due to inaccurate measurements made on the thickness of the test specimens before and after plating. Those inaccurate measurements were used by the grinding shop which resulted in the final specimens thicknesses.

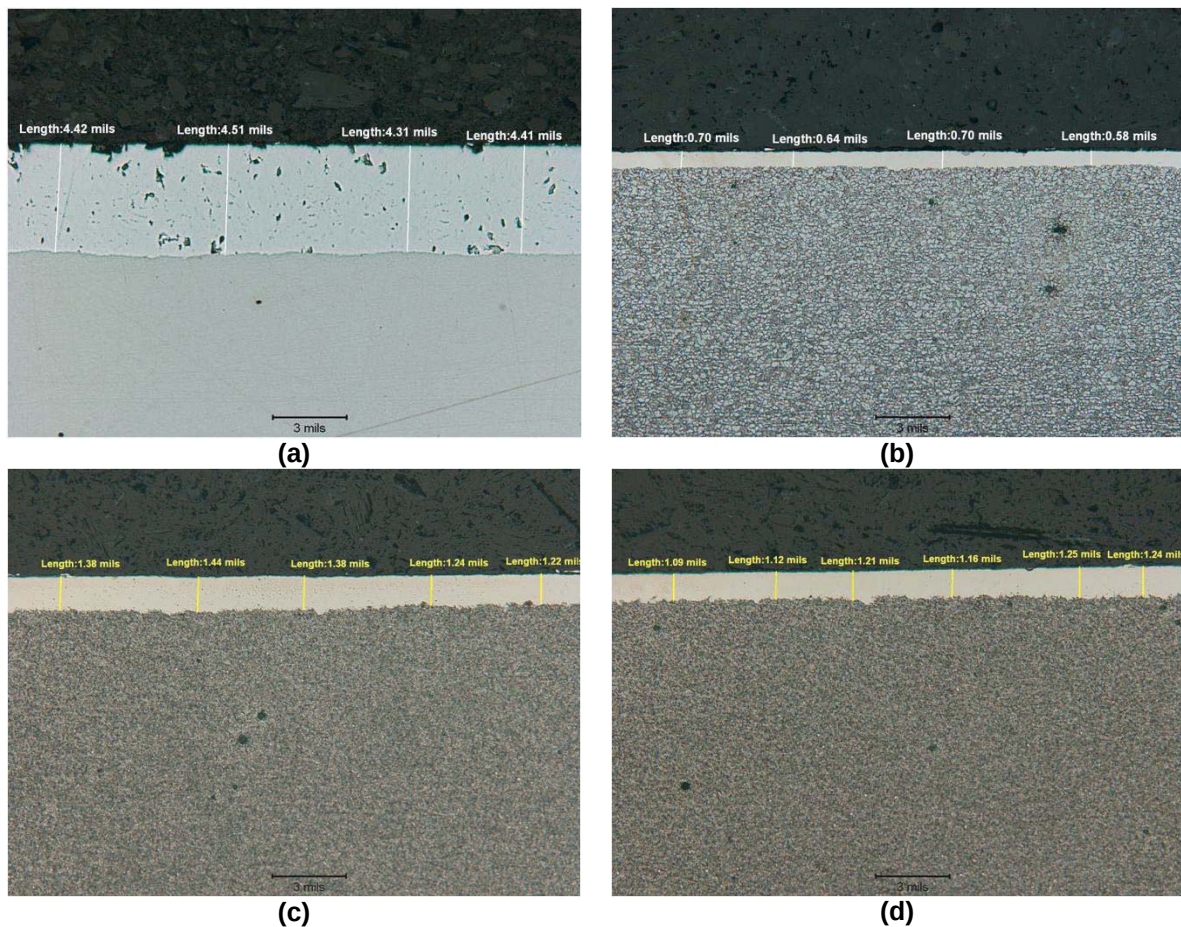


Figure 6-60. Metallographic Cross-Section of Gravelometry Test Specimen (a) EHC (HE Baked) – CR-E9B26-04-01, (b) nCoP (No Bake) – CO-E10018-01-02, (c) nCoP (HE Baked) – CO-E10021-02-03, (d) nCoP (Max Hardness) – CO-E10034-03-03.

Vickers microhardness readings were taken of the EHC and nCoP plating on the metallographic cross-sections from the gravelometry test specimens. Table 6-34 shows the microhardness results for the four different plating materials. The nCoP microhardness readings were lower than the

EHC, and the nCoP with max hardness heat treat had higher hardness than the nCoP with no bake and nCoP with HE bake.

Table 6-34. Vickers Hardness Values for nCoP and EHC Plating Materials on Gravelometry Test Specimens.

Plating Material	Load	Vickers Hardness
EHC (HE Baked) – CR-E9B26-04-01	500 g	898 +/- 11
nCoP (No Bake) – CO-E10018-01-02	10 g	646 +/- 28
nCoP (HE Baked) – CO-E10021-02-03	100 g	684 +/- 11
nCoP (Max Hardness) – CO-E10034-03-03	100 g	794 +/- 14

All of the nCoP test specimens performed as good as the EHC test specimens in the ASTM D 3170 gravelometry test (see Table 6-35). None of the plating chipped or was removed during the gravelometry test and the subsequent 3M #250 tape adhesion tests. The nCoP plating thickness was significantly lower than EHC due to an error during machining. Furthermore, the nCoP plating has a lower Vickers microhardness than the EHC plating but this did not adversely affect the gravelometry test results.

All nCoP samples met or exceeded the performance of EHC samples.

Table 6-35. Summary of results on gravelometry testing.

Coating	Thickness after grinding	Microhardness	Gravelometry Damage	Tape Adhesion
EHC (HE Baked)	0.0045"	898 +/- 11	No chipping or other damage	Pass
nCoP (No Bake)	0.0005"	646 +/- 28	No chipping or other damage	Pass
nCoP (HE Baked)	0.00125"	684 +/- 11	No chipping or other damage	Pass
nCoP (Max Hardness)	0.00125"	794 +/- 14	No chipping or other damage	Pass

6.1.17 Wear – SATEC Oscillating Load

This wear test is a pin-bushing test that simulates the use of the nCoP coating on a pin that is inserted through a bushing to simulate a single pin joint. Synchronous, sinusoidal load and motion test profiles and traditional, constant load were both run. The sinusoidal load and motion test profiles simulate cyclic, intermittent-load, in-service conditions, to establish representative wear rates for various stresses/cycles and to better represent how bushings and plain bearings are loaded and unloaded in service. Constant load testing will represent in-service use where the bushings are under constant load. Table 6-36 below represents the testing conditions utilized.

Samples were prepared by depositing EHC or nCoP coatings to the outer diameter of the pin to a minimum thickness of 0.006". All samples were ground and polished to a final coating thickness of 0.003"- 0.005" and to a surface finish of 12 microinches or finer IAW MIL-STD-866.

Table 6-36. Bushing Wear Test Variables.

Type of Loading	Stress Level	Grease Intervals (No. of cycles)	Rotation angle of pin (degrees)	Rate (cpm)	Number of Cycles	Number of tests	Number of counterface material
Sinusoidal	30 ksi peak	5000	30	20	100,000	2	2
Constant	20 ksi	5000	30	20	50,000	2	2

6.1.17.1 *Plating Condition*

The quality of the plating on the test pins was less than satisfactory for both nCoP and EHC samples. During testing, the majority of samples experienced some level of cracking and flaking. This may have been avoided by having a plating run out. This would alleviate the stress concentration along the edges and could prevent cracking the flaking. The cracks did not propagate into the test area. The integrity of the samples was a potential cause for erratic test results (i.e., completion of testing prior to reaching 100,000 and 50,000 cycles for sinusoidal and constant load testing, respectively).

6.1.17.2 *Sinusoidal Loading*

6.1.17.2.1 *PH13-8Mo Steel Bushing*

The SATEC tests using PH13-8Mo steel bushings were almost all stopped manually before hitting the 100,000 cycle mark. The tests were loud or exhibiting crunching noises and were considered to have "failed" at that point. As such, wear comparisons for the PH13-8Mo steel specimens were made using the wear rate instead of total wear. Overall, the nCoP plating showed lower friction and wear (Table 6-37). The HE bake and max hardness bake for nCoP were similar in performance. The EHC plating had marginally higher friction and wear.

6.1.17.2.2 CuBe Bushing

When mated against a CuBe bushing, the nCoP plating performed better in wear and friction than the EHC plating (Table 6-38). Results were similar between the HE bake and max hardness bake for the nCoP plating.

6.1.17.3 Constant Loading

6.1.17.3.1 PH13-8Mo Steel Bushing

The SATEC tests using PH13-8Mo steel bushings were almost all stopped manually before hitting the 50,000 cycle mark. The tests were loud or exhibiting crunching noises and were considered to have “failed” at that point. As such, wear comparisons for the PH13-8Mo steel specimens were made using the wear rate instead of total wear.

In the constant loading condition, not much difference was seen among the various plating options. Generally speaking, the EHC plating gave the most consistent results, though performance figures for wear and friction are all similar or too erratic for any conclusions to be made (Table 6-39).

6.1.17.3.2 CuBe Bushing

In the constant load condition against a CuBe bushing, the EHC and HE bake nCoP performed better than the max hardness bake nCoP in the bushing wear category (Table 6-40). For friction, both nCoP bakes performed at lower friction values than the EHC.

Table 6-37. Summary of SATEC Oscillating Load testing for Sinusoidal Loading on nCoP and EHC pins against 13-8 Steel Bushing.

	EHC		nCoP (HE bake)		nCoP (max hardness)	
Coefficient of Friction	0.348 ± 0.082	0.357 ± 0.049	0.206 ± 0.068	0.127 ± 0.025	0.125 ± 0.018	0.203 ± 0.094
Pin Wear Total	-0.00115”	-0.00090”	-0.00080”	-0.00005”	-0.000010”	0.01960”

Table 6-38. Summary of SATEC Oscillating Load testing for Sinusoidal Loading on nCoP and EHC pins against CuBe Bushing.

	EHC		nCoP (HE bake)		nCoP (max hardness)	
Coefficient of Friction	0.106 ± 0.040	0.112 ± 0.044	0.098 ± 0.019	0.114 ± 0.021	0.068 ± 0.019	0.077 ± 0.018
Pin Wear Total	0.00015”	0.00010”	0.00015”	0.00000”	0.00005”	0.00005”

Table 6-39. Summary of SATEC Oscillating Load testing for Constant Loading on nCoP and EHC pins against 13-8 Steel Bushing.

	EHC		nCoP (HE bake)		nCoP (max hardness)	
Coefficient of Friction	0.433 ± 0.030	0.356 ± 0.079	0.423 ± 0.072	0.351 ± 0.069	0.480 ± 0.086	0.399 ± 0.083
Pin Wear - Total	-0.00390"	0.00095"	0.03030"	-0.00100"	0.01725"	0.00340"

Table 6-40. Summary of SATEC Oscillating Load testing for Constant Loading on nCoP and EHC pins against CuBe Bushing.

	EHC		nCoP (HE bake)		nCoP (max hardness)	
Coefficient of Friction	0.257 ± 0.041	0.216 ± 0.044	0.135 ± 0.052	0.153 ± 0.051	0.133 ± 0.061	0.137 ± 0.052
Pin Wear - Total	0.00010"	0.00000"	-0.00010"	0.00000"	0.00005"	0.00475"

The nCoP samples met the acceptance criteria.

6.1.18 Corrosion – Cobalt Oxide Characterization

Following exposure to salt spray, nCoP samples have shown discoloration associated with formation of a cobalt oxide as confirmed by compositional analysis. The appearance of the oxide is similar to red rust and is often misinterpreted by technicians or engineers conducting the evaluation during testing. The composition of the oxide which forms on nCoP when exposed to salt spray corrosion environments and beach exposure environments was compared using XPS across multiple areas of the specimens according to the test matrix in Table 6-41. Representative images of samples can be found in earlier sections of the report.

Table 6-41. Cobalt Oxide Characterization Matrix.

Coating	Description	Quantity	
		Ni underlayer	No underlayer
nCoP	As-deposited	-	1
nCoP	After Neutral Salt Fog	-	1
nCoP	After Beach Exposure	3	3
Total		3	5

For the as-deposited nCoP sample, a surface layer of mostly CoO is seen above the metallic cobalt. Calculations from these results suggest an oxide film thickness of 1.7 nm (in Area 1) and 2.1 nm (in Area 2). Analysis of the O 1s spectra (Figure 6-61) suggests a relatively pure CoO (lattice oxide) for Area 1 and a small amount of hydroxide/defective oxide in the lattice oxide for Area 2.

For the neutral salt fog nCoP sample, both areas analyzed suggest that the major component present is Co_3O_4 . After factoring out the contributions of organic oxygen, the O 1s results suggest an approximately equal contribution of lattice oxide and hydroxide/defective oxide.

For the after beach exposure nCoP and Ni/nCoP samples, the major component is Co_3O_4 , with small to moderate amounts of CoO also detected. Sample Ni/nCoP-211 Beach shows the most CoO with one area analyzed having ~86 % CoO.

The O 1s spectra (Figure 6-61) show a mix of lattice oxide, hydroxide and defective oxide, organic oxygen and possibly water to be present on the surfaces. After factoring out the contributions of organic oxygen, the results suggest an approximately equal contribution of lattice oxide and hydroxide/defective oxide. While a portion of the hydroxide/defective oxide component will be from defective structure in the lattice oxides, this data would also suggest that

the Co_3O_4 and CoO oxides also contain some hydroxide type components incorporated into their structures. It may also suggest a certain amount of water incorporated within the oxides.

In all instances, samples from before and after corrosion environment exposure have natural cobalt oxide formation (Co_3O_4 , CoO and other cobalt-based defective oxides). No iron based corrosion products were present which confirms that no red rust formation occurs. Through corrosion testing, it has been established that the cobalt oxide is dense, compact and adherent unlike red rust.

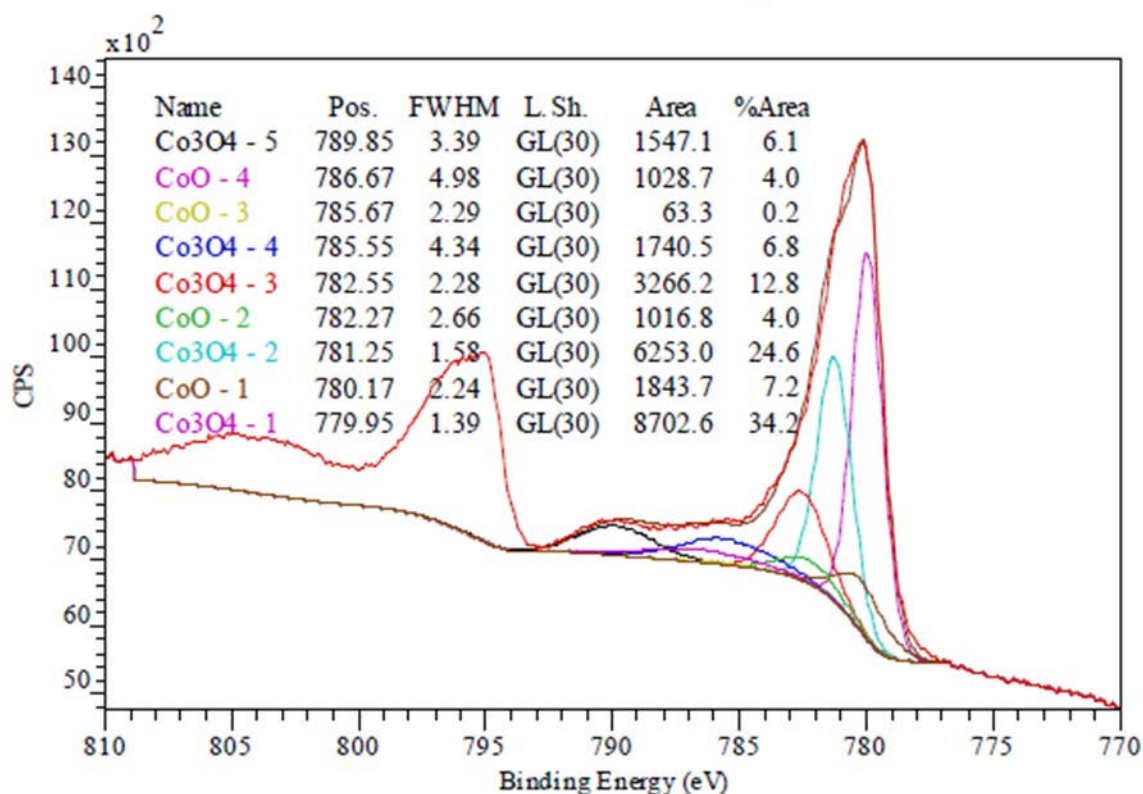


Figure 6-61. Representative high-resolution XPS spectra obtained for nCoP samples showing CPS (counts per second) for binding energy over the range identified for cobalt oxides.

6.1.19 NAVSEA – Adhesion, Impact, Corrosion, Wear Testing

NAVSEA conducted a project to evaluate several alternative surface treatments for use on USMC hydraulic cylinders. 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. The following 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).

6.1.19.1 *Adhesion: Chisel-knife grind saw*

Coating/plating adhesion was evaluated in accordance with ASTM B571 Method 5, Chisel-Knife Test, and Method 8, Grind-Saw Test. In the Chisel-Knife Test, a cold chisel was used to penetrate the coating/plating. If the coating was removed, then adhesion was considered insufficient. In the Grind-Saw Test, a hack saw was run across the sample, moving the saw in the direction that tends to separate the coating from the substrate. Lifting or peeling of the coating indicated insufficient adhesion. Tests were performed on the same 6" x 12" x 1/8" sample panels coated with nCoP and EHC. Results indicated that all samples tested passed adhesion testing (Table 6-42).

Table 6-42. Adhesion: Chisel-knife grind saw test results.

Sample ID	Coating Type	Chisel-Knife Test	Grind-Saw Test
HC-A-1	EHC	Pass	Pass
HC-A-2	EHC	Pass	Pass
HC-A-3	EHC	Pass	Pass
COP-A-1	nCoP	Pass	Pass
COP-A-2	nCoP	Pass	Pass
COP-A-3	nCoP	Pass	Pass

6.1.19.2 *Adhesion/Wear: Impact resistance*

Impact resistance tests were performed and evaluated in accordance with ASTM B571 Method 10, Impact Test. In this test an indenter with 0.5-inch hemispherical impact head was placed into contact with a test panel. From a known height a 2 lb. weight was dropped through a guide tube, impacting the strike end of the indenter. The intrusion side of the sample was evaluated for evidence of cracking with a magnifying glass. The height from which the weight was dropped was varied systematically until the impact results changed from mostly failure to mostly passing; this height was recorded as the failure end point. The test was repeated five times at this end point, five times just above this value and five times just below this value. The final failure end point was reported in inch-pounds.

Impact resistance testing results for each set of samples can be found in Table 6-43. It should be noted that impact testing samples were not carburized or polished because these processes cause warping and cracking of thin gage panels. The failure point is the height and weight combination at which the coating/plating system begins to crack on impact. No cracking was observed in

EHC samples while cracking was found on the nCoP samples at a failure point of 68 inch-pounds. This value is characteristic of a highly impact resistant coating/plating system.

Table 6-43. Adhesion/wear: Impact resistance test results.

Sample ID	Coating Type	Failure Point (in-lbs)
HC-I	EHC	>80
COP-I	nCoP	68

6.1.19.3 **Wear: Taber abrasive**

Abrasion resistance was evaluated using a Teledyne Taber Rotary Platform Abrasion Tester Model 5150 with 1000 gram weights and CS-17 abrasion wheels per ASTM G195. Samples were tested for 1000 cycles and the wheels were resurfaced every 500 cycles. Sample mass was measured before (m_i) and after (m_f) cycling and total mass loss was calculated.

Abrasion resistance testing results can be found in Figure 6-62. The nCoP experienced less mass loss due to abrasion than the hard chrome sample. A limited number of cycles were performed which may have led to the unexpected improvement observed.

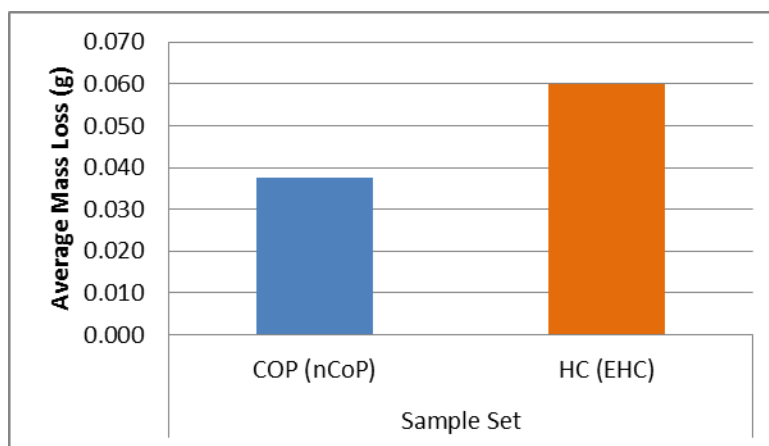


Figure 6-62. Wear: Taber abrasive test results.

6.1.19.4 **Corrosion: Accelerated corrosion**

Corrosion testing was performed using an ASTM B117 salt spray chamber. The nCoP sample was placed in the salt spray chamber for 720 hours and evaluated every 240 hours. The samples were evaluated using the following methods:

- ASTM D610 —Evaluating Degree of Rusting on Painted Steel Surfaces
- Visual inspection – changes in color or gloss were noted and photographed; samples were examined for cracking or other means of degradation

Corrosion resistance test results can be found in Figure 6-63. Samples with lower ASTM D610 ratings have higher percentages of rusting. As the data clearly shows, the nCoP samples significantly outperformed the EHC samples. The nCoP samples experienced minimal general

rusting on less than 0.03% of the surface area over the duration of the test as shown in Figure 6-64.

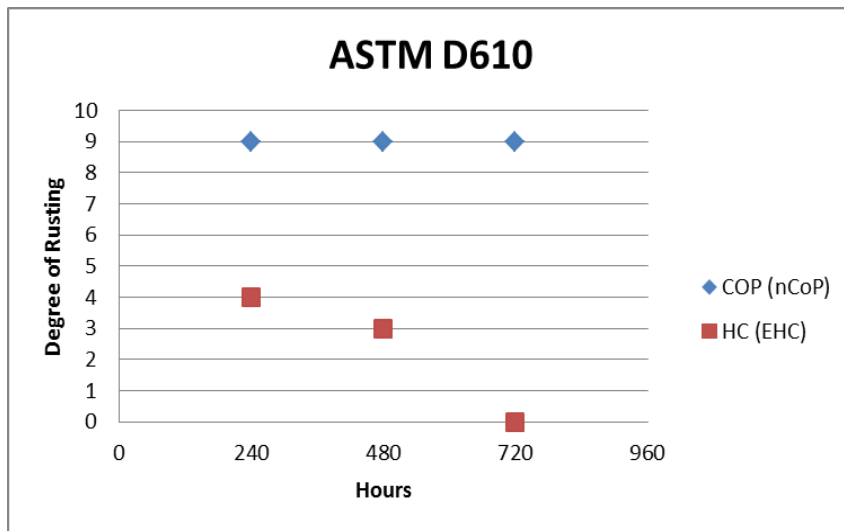


Figure 6-63. Corrosion: Accelerated corrosion test results.



Figure 6-64. Image of nCoP sample after 720hrs in ASTM B117 showing minimal red rust.

6.1.19.5 Wear: Cyclic piston wear/corrosion

This test was intended to simulate the operation of hydraulic cylinders in the field. The process included a hydraulic cycling step followed by an accelerated corrosion event. Modified yaw cylinder pistons, shown in Figure 6-65, and yaw cylinder shells were used for this test. The nCoP (COP) coating thickness applied was 0.001”.

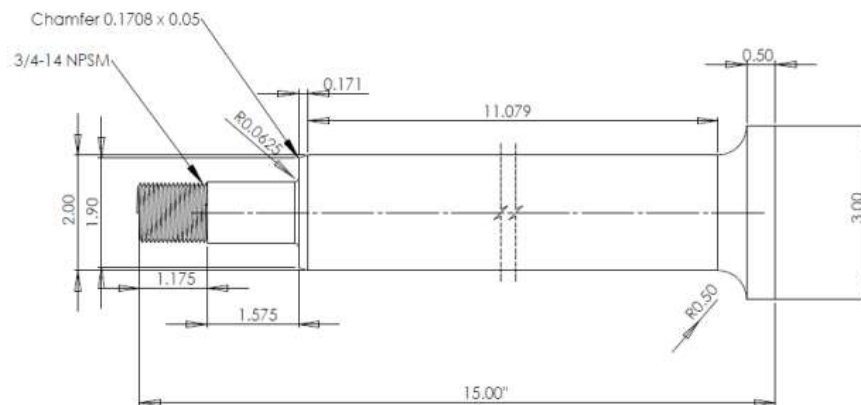


Figure 6-65. Drawing of the Modified Yaw Cylinder Piston.

The samples were first loaded into the piston manifold shown in Figure 6-66, run for 1000 cycles, and inspected for leaks. The samples were then placed with the pistons extended into an ASTM B117 salt fog chamber for 240 hours. After the salt fog exposure, pistons were inspected for corrosion damage using ASTM D610 and the TACOM rating system. These steps were repeated on the samples until a Class III seal leak was observed at which time the failed sample was removed from testing. Sample pistons were then disassembled from the seals and shells, photographed, and a final evaluation was performed.



Figure 6-66. Image of hydraulic cylinder manifold with pump.

The hydraulic cycling with corrosion events test results can be found in Figure 6-67. This graph shows the cumulative number of hours that the cylinders were exposed to ASTM B117 as indicated on top of each bar and the total number of hydraulic cycles until a seal failure was observed on the left axis. These results are consistent with the results from the corrosion resistance testing. The EHC (HC) sample experienced seal failure between 960 and 1200 hours of exposure. Similar to the corrosion resistance testing, the nCoP (COP) samples significantly outperformed EHC. Both nCoP samples lasted more than twice as long as EHC; one sample did not experience seal failure when testing ended after 13,000 cycles and 2880 hours of ASTM B117 salt fog exposure.

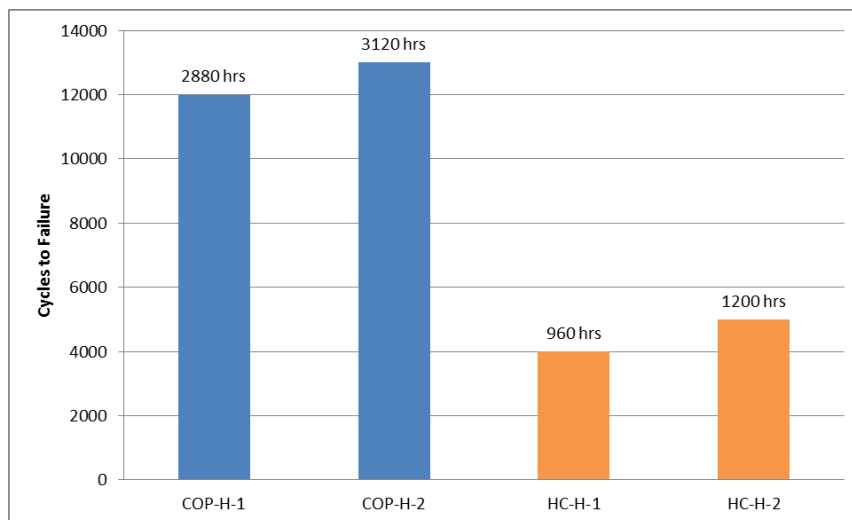


Figure 6-67. Hydraulic cycling results with cumulative exposure to salt spray results.

Figure 6-68 shows that, even after seal failure after 2,880 hours of B117 salt fog exposure, the nCoP sample exhibited two pits but very little rusting. These pits were not observed during testing and may have been a result of crevice corrosion because they remained under the seal at all times. This, coupled with the fact that the other nCoP sample did not fail, may suggest that the failure was due to the lifecycle of the seal itself and not wear from corrosion damage.



Figure 6-68. Image of nCoP Sample after hydraulic cycling with corrosion events testing.

6.1.19.6 *Summary*

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

6.2.1.1 Preparation for Field Demonstration of T-45 Pivot Assemblies

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”) nickel followed by a minimum of 3 mils (0.003”) chromium in the as plated condition. Figure 6-69 illustrates the area requiring plating and the amount of wear that is typically experienced in the field. In this study, two (2) 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.

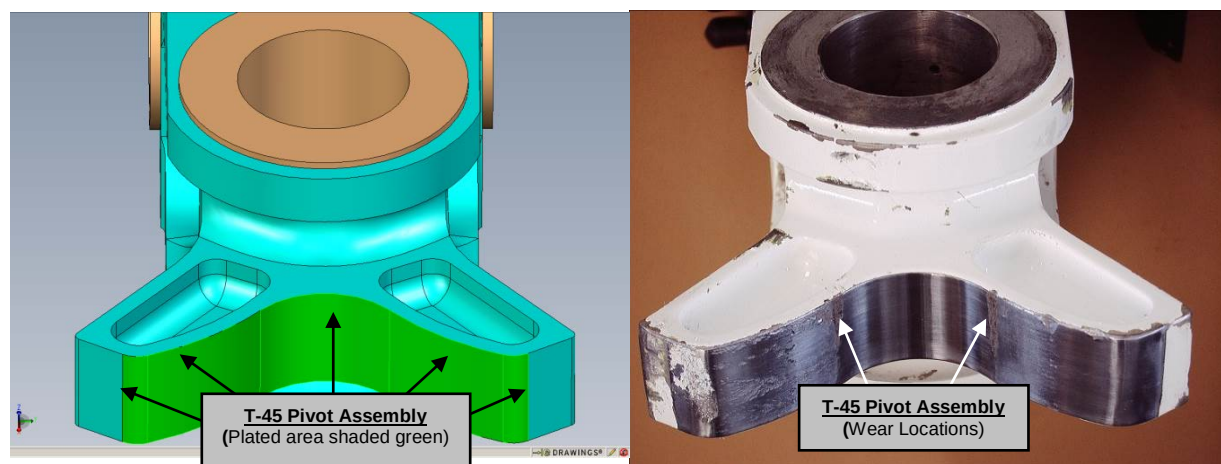


Figure 6-69. Solid model (Elsyca, Inc.) illustrating plating area (left) and images of component removed from service showing wear locations (right) on T-45 Pivot Assembly.

6.2.1.2 Establishing Plating Parameters for T-45 Pivot Field Pivot

To ensure the above thickness requirements and uniformity of the nCoP deposit were achieved, several test coupons (3” x 6” x 040, AISI 4130 steel) of similar geometry and area representing the pivot cam surface were initially plated with the nickel/nCoP duplex coating and evaluated. Figure 6-70 illustrates how the test coupons were racked, masked, plated and tested to validate deposition rate and coating thickness which would assist in establishing the plating parameters needed for plating the actual Dem/Val part. Use of computational analysis aided in determining the optimal distance between the anode-cathode in order to obtain a uniform coating thickness build-up. However, due to the high risk and complexity of the proposed Elsyca tooling design, a simpler approach to tooling design was employed. The resulting design led to exceptional results using a practical approach, which is ultimately more compatible with the existing depot practices. To facilitate plating into the throat area of the cam surface, a cobalt plated titanium rod

was used as an auxiliary anode. The total area plated including “thieving” areas, used by conductive tapes, was approximately 5.0 in². Following the above coupon testing, a surveyed (prototype) pivot was also racked, masked and plated with nCoP to better understand challenges with processing the subject part and to understand actual deposition conditions prior to plating the actual Dem/Val part.

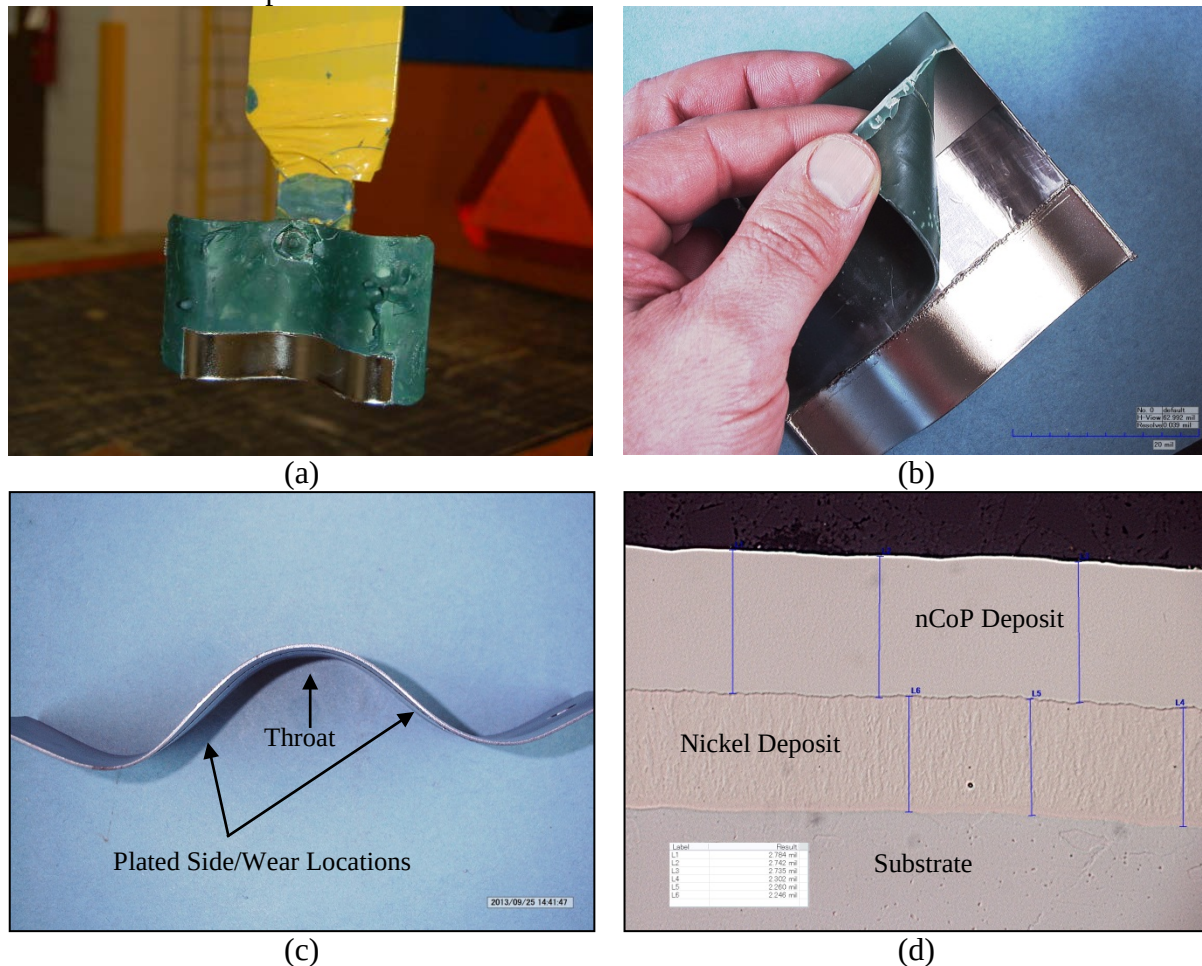


Figure 6-70. Image of (a) geometry test coupon following plating, (b) removal of maskant from test coupon, (c) side view of test coupon and (d) cross-section of test coupon for thickness verification.

6.2.1.3 Preparing the T-45 Pivot for Plating

Prior to plating the Dem/Val pivot, and to prepare the part for plating, all bushings and existing coatings (i.e., Ni/Chromium duplex plating, cadmium plating of external surfaces and primer/top coat) had to be removed. Figure 6-71 shows the Dem/Val pivot in the as received condition and following removal of all existing coatings/bushings.

Before proceeding with the plating process, the cam surface was non-destructively inspected for defects and other imperfections that may interfere with plating and/or the function of the part in service.

In parallel to preparing the pivot for plating, new bushings were manufactured and cadmium plated for later installation.

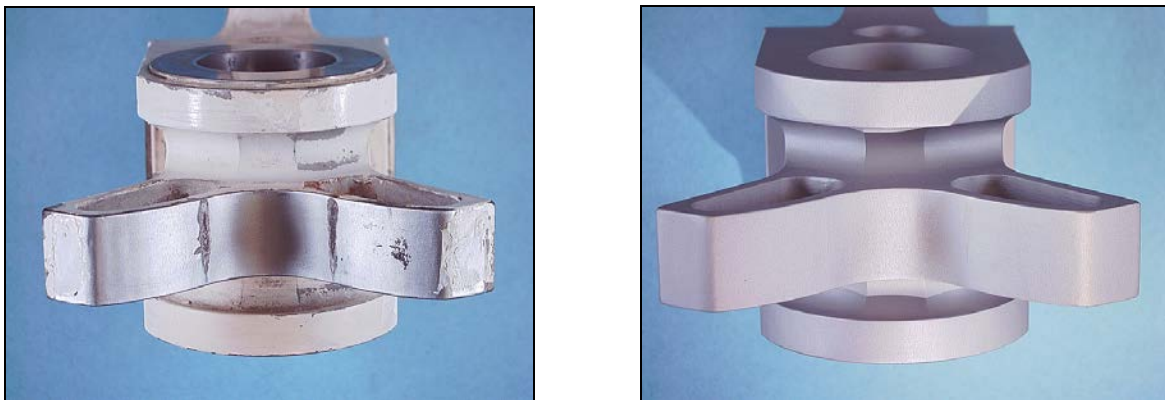


Figure 6-71. Image of DemVal part as-received (left) and following coating removal and media blasting (right).

6.2.1.4 Masking

Because the cam surface is the only surface required to be plated, all remaining surfaces of the pivot including the plating fixture were needed to be properly protected from the plating process. Masking of the pivot and fixture was applied using Enthone, Inc. Enplate Stop-Off #1 as shown in Figure 6-72. The masked pivot is shown in Figure 6-72 in the racked configuration with an auxiliary cobalt plated titanium anode positioned for ensuring coverage within the throat area of the cam surface. Yellow electroplater's tape, traditionally used for conventional masking for plating at FRCSE, was also used for facilitating masking of the fixture and to expose a section of the cobalt anode for plating the cam surface. However, it was later found that this particular tape was not desirable, due to break down of the adhesive backing of the tape in the plating bath. Other, more compatible tapes were identified during the course of the project development that would prove to be more compatible with the bath temperature and chemistry eliminating risk of pitting in the deposit and thus minimizing the frequency of carbon treatments required in the bath.

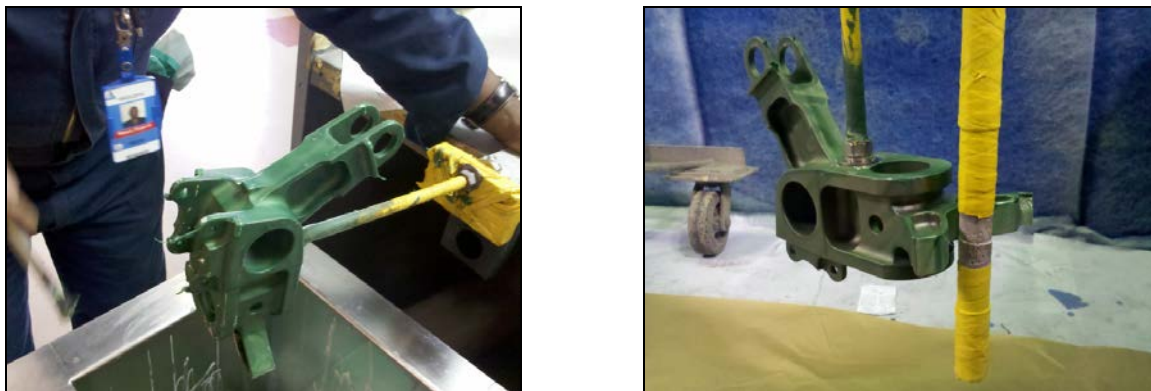


Figure 6-72. Image of DemVal part being masked for selective plating (left) and fixturing prior to plating with positioned auxiliary cobalt anode (right).

6.2.1.5 Detailed Plating Process Steps of T-45 Pivot

Based on initial coupon testing and plating trials, specific plating parameters/process steps were established for plating the actual Dem/Val component. Detailed process steps utilized in plating the T-45 Dem/Val part including a run sheet of the actual plating process are provided in **Table 6-44** and Table 6-45, respectively. In Figure 6-73, the process flow diagram 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.

Table 6-44. Plating Process Steps Ni/nCoP duplex on T-45 Pivot Assy

Step No.	Description	Time	Remarks	Temp (F°)	Comments	Entry Log
1	Degrease as necessary		Cleaning Shop (not required if part has been blasted)			24 May 11
2	Inspect surface for defects					24 May 11
3	Abrasive Blast		Glass or Grit (size 220)		Hand Sanded (80/320 grit)	24 May 11
4	Remove excess blast media					N/A
5	Verify pre-plate dimensions					N/A
6	Tape, Rack & Mask		Enthone Stop Off #1 Maskant		150°F cure for 1hr, trim to expose cam surface	24 May 11
7	Alconox/pumice scrub area	As req.	1 part Alconox to 100 parts pumice			25 May 11
8	Rinse		Check for water breaks	Room		
9	Electroclean (Anodic)	1 – 5 min	4 to 6 volts anodic (45 – 55 ASF)	125-135	1.3 – 1.5 Amps @ 6V	In: 9:53 Out: 9:56
10	Rinse			Room		
11	Acid activation H2SO4	15 – 60 sec	Reverse current (0.69 to 2.8 amps/sq. in) (If smut is present, pumice scrub and repeat activation step for 5 sec)	Room	2.8 – 11.2 Amps	In: 10:09 Out: 10:10
12	Rinse			Room		
13	Ni Strike (Current on)	30 – 60 sec	0.07 – 0.7 amps/in2 (keep surface wet prior to next step)	80 - 100	0.28 – 2.8 Amps (2 amps @ 1.2V)	In: 10:10 Out: 10:13
14	Ni Plate (Current on)	1 hr 20 min	0.17 – 0.52 amps/in2 Target 0.002" /side	90 - 140	0.68 – 2.1 Amps (1.0 A @ 1.3V)	In: 10:14 Out: 11:34
15	Rinse			Room		
16	nCoP Plate (Current on)	1 Hr	116 ASF (50% Duty Cycle) Target 0.003" /side	180 - 195	7.0 peak Amps (NOTE: went in w/o current for 20 sec)	In: 11:35 Out: 12:35
17	Rinse			Room		
18	Inspect plating		Inspect for conformance		Bright Uniform Deposit (verification panel plated)	Passed Adhesion
19	De-mask/ De-Rack					25 May 11 13:30
20	Bake for HE Relief	min 23 hr		375±25		In: 14:35 (25 May 22) Out: 14:30 (26 May 11)

Table 6-45. Run Data Sheet.

General Information			
S/N: 0002PG-19DP		Qty:1	
Project No: WP-0936		Batch No: 01	
Description: Pivot Assy, P/N: DA327A5213-17		Bath ID: Tank A-11	
Configuration			
Plating Area (in ² or ft ²)	4.38 in ² including robber or 0.030 ft ²		
Anode (Basket/Rod)	Auxiliary Cobalt Plated Rod		
Agitation/Flow	3000 GPH		
Shielding/Thieving	Lead Tape on edges of cam surface		
Other	Yellow plater's tape/Enplate Stop Off #1		
Run Data			
Parameter	Target	Actual	Notes
Temperature (°F)	185	185	
Current Density, ASF	116	116	
Current Average, A	3.5	4.0	Including Robber
Current Peak, A	7.0	7.0	" "
Duty Cycle, %	50	50	
Voltage, V		0.6	
Duration, min, hr, etc.		1 hr	
Amp/min	240	252	Before: 601779 After: 602031 (252 total Amp/min)
Thickness, mils	2/3	2.1/2.8	Target: 0.002"/side Ni, 0.003"/side nCoP
Tank Analysis Data			
pH @ 25°C	1.1	1.2	
Density (g/cm ³)	1.14	1.14	
Co (g/L)	65	63	
P (g/L)	13	12	
Cu (mg/L)	-	-	
Fe (mg/L)	-	-	
Notes: Some observations during processing of the component are noted below: 1. Smut formation was noted following the electroclean step. This was rectified byalconox/pumice scrubbing and reactivating in acid etch step. 2. Component was immersed in nCoP plating tank without current for approx. 20 sec before applying current to part. A verification coupon was plated under the same conditions and was tested for adhesion. Verification coupon passed bend adhesion test.			
Run Sheet Code Designation: 			

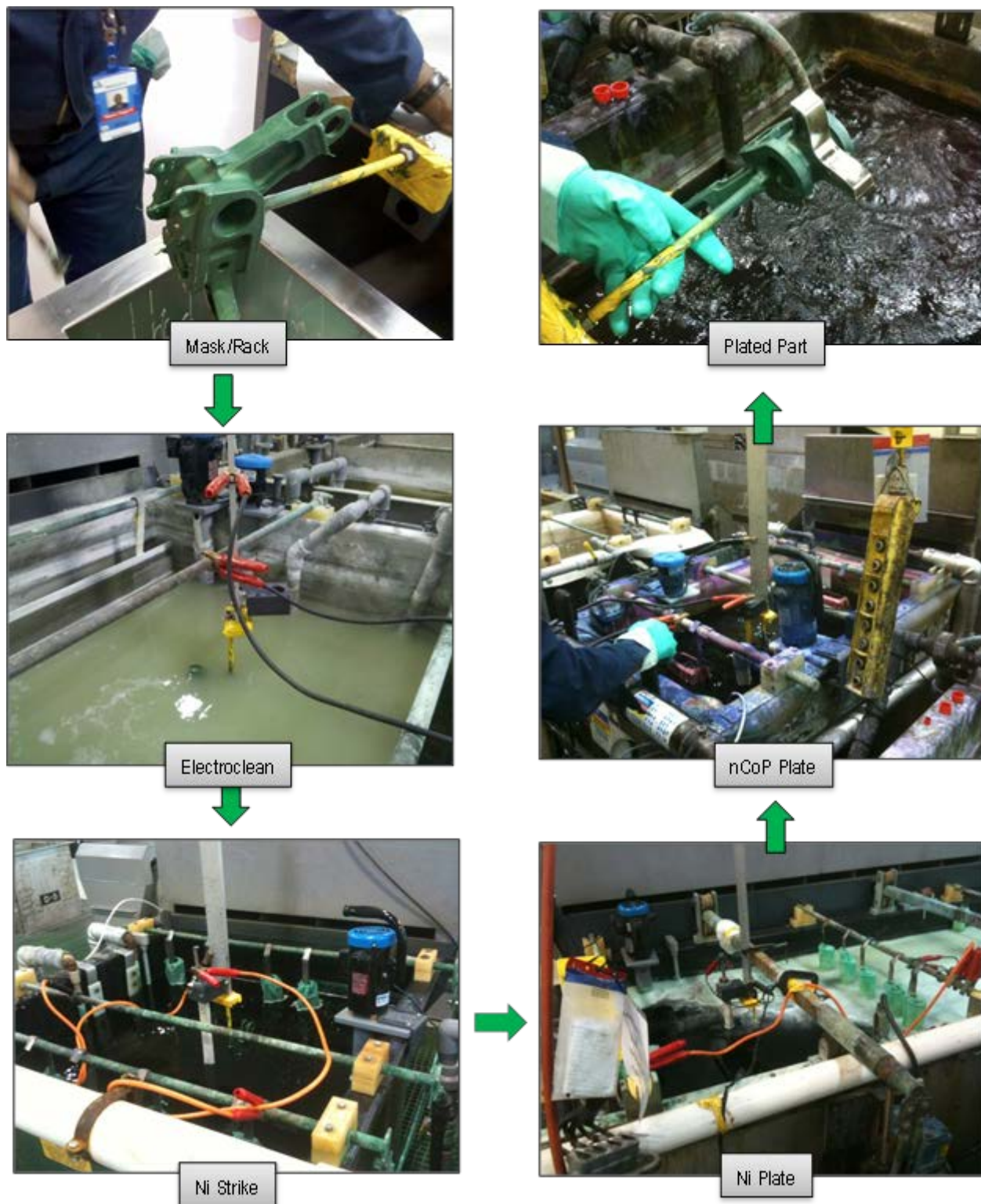


Figure 6-73. Visual illustration of the general plating process flow.

6.2.1.6 IVD Aluminum Plating Remaining Surface of the Pivot Assembly

Upon completion of the nCoP plating process, the part was de-masked, HE relief bake for 24 hrs and prepared for IVD plating of remaining surfaces. The edges of the cam surface were lightly blended/buffed to remove the “treeing” or buildup of plating which naturally occurs on the high current density areas. Both the nCoP plated pivot and the baseline EHC plated pivot are shown in Figure 6-74 and Figure 6-75, respectively in the as-plated condition prior to edge blending.

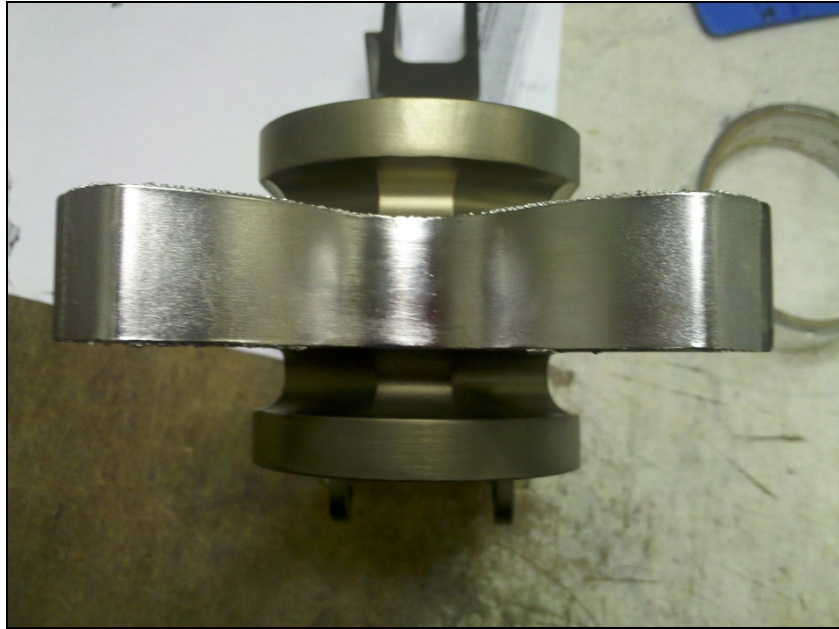


Figure 6-74. Image of T-45 nCoP plated pivot shown in the “as-plated” condition.



Figure 6-75. Image of T-45 EHC plated pivot shown in the “as-plated” condition.

IVD aluminum plating of the pivot body was applied IAW FRCSE local process specifications followed by supplemental chromate treatment. The nCoP plated Dem/Val pivot following the IVD process and blending/buffing of the cam edge/surface is shown in Figure 6-76.

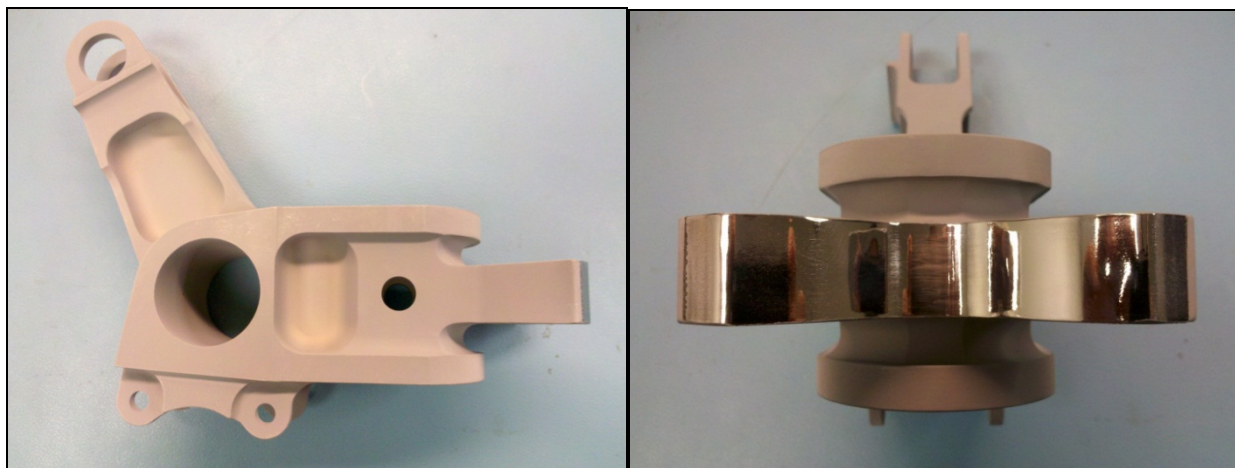


Figure 6-76. Image Pivot Assembly side view of IVD aluminum coated and front view following IVD blending/buffing of cam surface.

6.2.1.7 Paint Application and Identification of Dem/Val Pivots for Field Demonstration Tracking

Following the IVD coating process and bushing installation, both the nCoP plated and EHC plated pivots were routed for painting. Each pivot was uniquely identified by painting a series of black stripes along the fitting with the project number and coating type stenciled on side of the pivot body to identify them in the field. The nCoP plated pivot was identified by three black stripes painted on the fitting with the words “WP-0936 nCoP” and “ENG. Prototype” stenciled in black on the side (Figure 6-77). The EHC plated pivot was identified by two black stripes painted on the fitting with the words “WP-0936 Cr” and “ENG. Prototype” on the side (Figure 6-78).

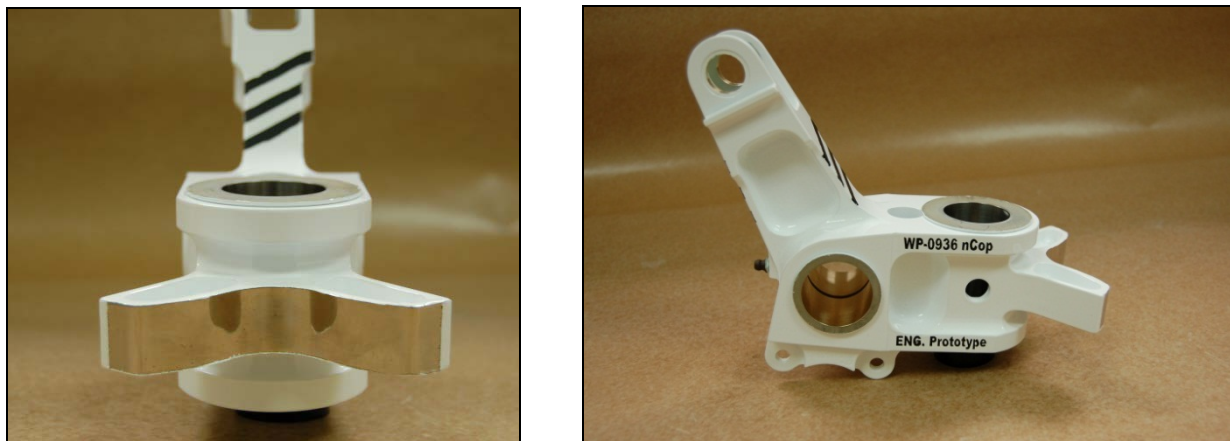


Figure 6-77. Image of nCoP coated Pivot Assembly following painting front view (left) and side view (right). Stenciling of three stripes is used as identifier for field inspection.



Figure 6-78. Image of EHC coated Pivot Assembly following painting front view (left) and side view (right). Stenciling of two stripes is used as identifier for field inspection.

To provide supplemental field data with the nCoP coating, the pivot assembly tie down rings were also plated. Only half the ring required a wear coating (plated with nCoP) while the remaining surfaces were IVD aluminum coated and painted with similar identification markings for field demonstration purposes (Figure 6-79).



Figure 6-79. Image of nCoP and IVD Plated Tie down rings (left) and following painting Tie down rings ready for installation (right).

6.2.1.8 Installation and Tracking of Field Components

The nCoP plated Dem/Val pivot was successfully installed on aircraft as shown in Figure 6-80. Components processed were tracked by serial number and identified within the applicable scheduled removal component card (SRC) as a T-45 engineering study (Figure 6-81 thru Figure 6-84). 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 time of this report, only the nCoP plated pivot assembly had reached sufficient amount of required arrestments for a full off aircraft inspection of the plated cam surface. A record of field history data extracted from the SRC cards for both nCoP and EHC plated pivots is also provided in Table 6-46 and Table 6-47.

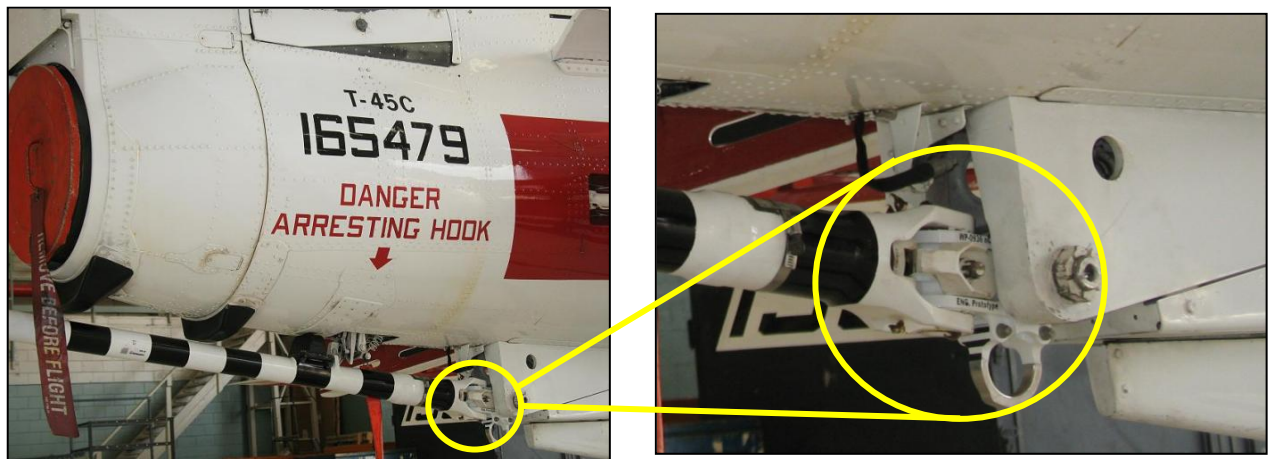


Figure 6-80. nCoP plated pivot installed on A/C BUNO 165479.

[illegible]

Figure 6-81. Scheduled Removal Component Card (Page 1 of 2), Section I-III for EHC Plated Pivot (-204DR).

Interval inspection data listing the number of arrestments, the aircraft BUNO number, associated flight hours and date of inspection for each Dem/Val pivot is provided in Table 6-48 &

Table 6-49.

Table 6-48. Field Inspection Log of nCoP Plated Dem/Val Pivot (S/N: 0002PG-19DP).

ARRESSTMENTS	FLT HRS	BUNO	DATE	REMARKS
Installed	--	165479	Mar 2012	--
20	--	165492	Aug 2012	--
39	--	165492	Mar 2013	--
72	--	165492	Oct 2013	--
72	705 ± 5	165492	Dec 2013	Inspected
72	--	165089	Mar 2014	
97	825 ± 15	165089	Jul 2014	Hook shank/pivot inspected
116	900 ± 15	165629	Nov 2014	Still in the field

Table 6-49. Field Inspection Log of EHC Plated Dem/Val Pivot (S/N: 0006PG-204DR).

ARRESSTMENTS	FLT HRS	BUNO	DATE	REMARKS
Installed	--	165482	May 2012	--
33	--	165463	Aug 2012	--
63	--	165463	Mar 2013	--
63	--	165463	Oct 2013	--
63	--	165463	Dec 2013	--
63	--	165463	Mar 2014	--
63	--	165463	Jul 2014	--
63	795 ± 5	165463	Nov 2014	Still in the field

6.2.1.9 Field Inspection of nCoP Plated Pivot Assembly

Because an inspection within intermediate level maintenance was due on the arresting hook shank which was attached to the field demo pivot, the pivot was also visually inspected. A spot check on the cam surface of the pivot is normally performed to identify if the protective wear coating has worn to the base metal. This is done by cleaning the cam surface of any grease/oil or debris followed by adding a few drops of copper sulfate solution to the worn area to determine the presence of iron or exposed substrate metal. Copper is typically plated out of solution when in contact with iron or ferromagnetic materials providing a positive indication of exposed ferrous metal. Because, nCoP has ferromagnetic properties, the traditional spot test method was unreliable as it gave a false positive when inspected in the field. As such, alternate field inspection methods are being explored to provide the field maintainers a quick and reliable indication of the amount of wear damage in service. Exposed substrate due to wear damage or compromised coating in service will require that the pivot be completely overhauled and reprocessed at Depot level maintenance.

6.2.1.10 *Field Performance Summary*

To date, field performance of the nCoP plated pivot assembly has performed equivalent or better than the baseline coating. Figure 6-85 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 NAVAIR Jacksonville for refurbishment if plating damage is observed beyond the scope of repair by the operating site.



Figure 6-85. Images of cam surface of nCoP plated pivot following 97 arrestments.

6.2.2 Spotting Dolly Lifting Pins

6.2.2.1 Spotting Dolly Dem/Val

A/S32A-32 Spotting Dolly lifting pin demonstration consisted of field testing within a carrier based operational environment aboard the USS Harry S. Truman (CVN-75). Technical points of contact for initiating, organizing and performing field demonstrations are presented as Table 6-50.

Table 6-50. Demonstration Points of Contact.

POC	Organization	Location
Doug Kilgore	SD-2 Project Engineer	NAWC-AD (Lakehurst)
Mike Firth	Materials Engineering	NAWC-AD (Lakehurst)
Billy Ostrum	TYCOM	COMNAVAIRLANT
ASCS VanSteinburg	IM4 LCPO	CVN 75

In preparation for demonstration, Lifting Pins were procured from the national supply system via NSN: 5315-01-081-6481, Part No. 1117AS243-1 during May 2011. Upon receipt, pre-existing electroplated coatings were removed via chemical striping and mechanical blasting. Items were then packaged for shipment to Integran Technologies for application of nCoP electroplate.

6.2.2.2 Processing

Electroplating was performed Oct 2012 in accordance with the Nanovate R3010 Process Specification Guide. Custom racking and anodes were designed to ensure uniform electroplating thickness throughout the component. Figure 6-86 illustrates racking and anode design features utilized for electroplating. Following application of nCoP items were returned to the Fleet Readiness Center Southeast for application of IVD-Al. IVD-Al was applied in accordance with Local Process Specification 300 followed by supplemental chromate treatment. IVD-Al was applied to the large diameter areas of the lifting pin for corrosion protection of Spotting Dolly's Lifting Arm. The small diameter and flange face of the lifting pin were fielded without IVD-Al.

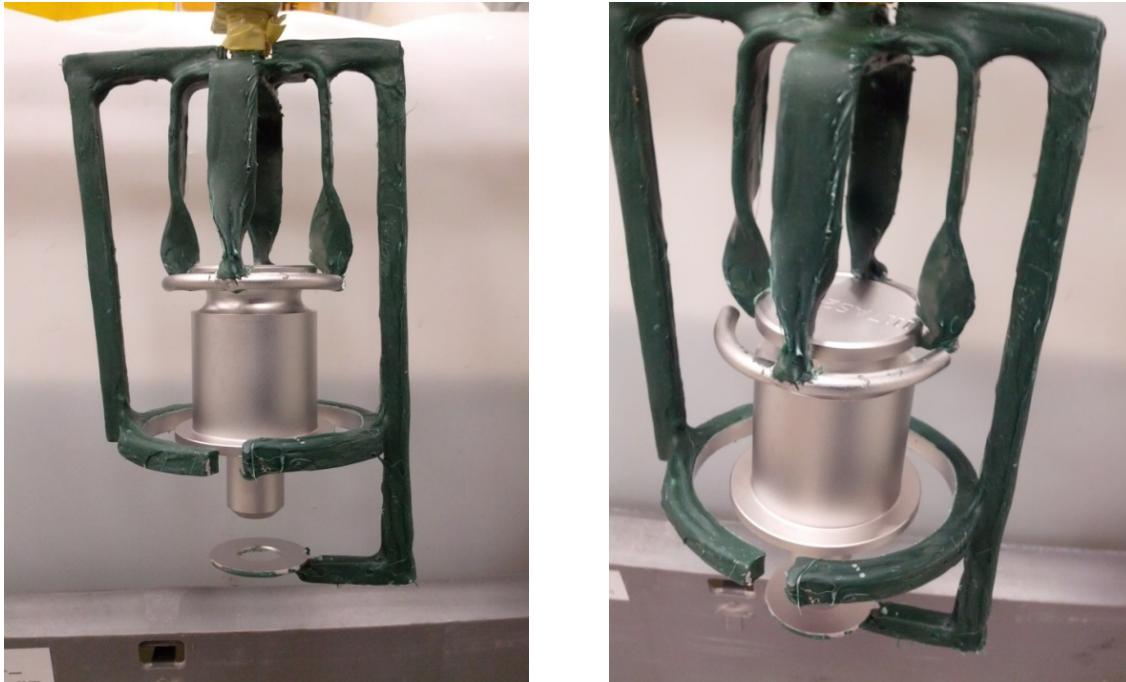


Figure 6-86. Custom racking and anode design for application of Nanovate R3010 electroplate.



Figure 6-87. Image of Nanovate R3010 electroplated part. Grey areas are contact points which will be protected by sacrificial coating.

6.2.2.3 Fielding

Following completion of electroplating operations by Integran Technologies and IVD-AI post processing by FRCSE, demonstration components were transferred to 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 USS Harry S. Truman (CVN 75). While onboard, the CVN 75 deployed to the Mediterranean Sea and Persian Gulf regions supporting Operation ENDURING FREEDOM.

As of January 2014, an estimated 672 aircraft moves were performed without failure or defect. Figure 6-88 summarizes the demonstration background timeline.



Figure 6-88. Lifting Pin demonstration background timeline.



Figure 6-89. Lifting Pin demonstration components following processing at Integran Technologies and FRCSE.

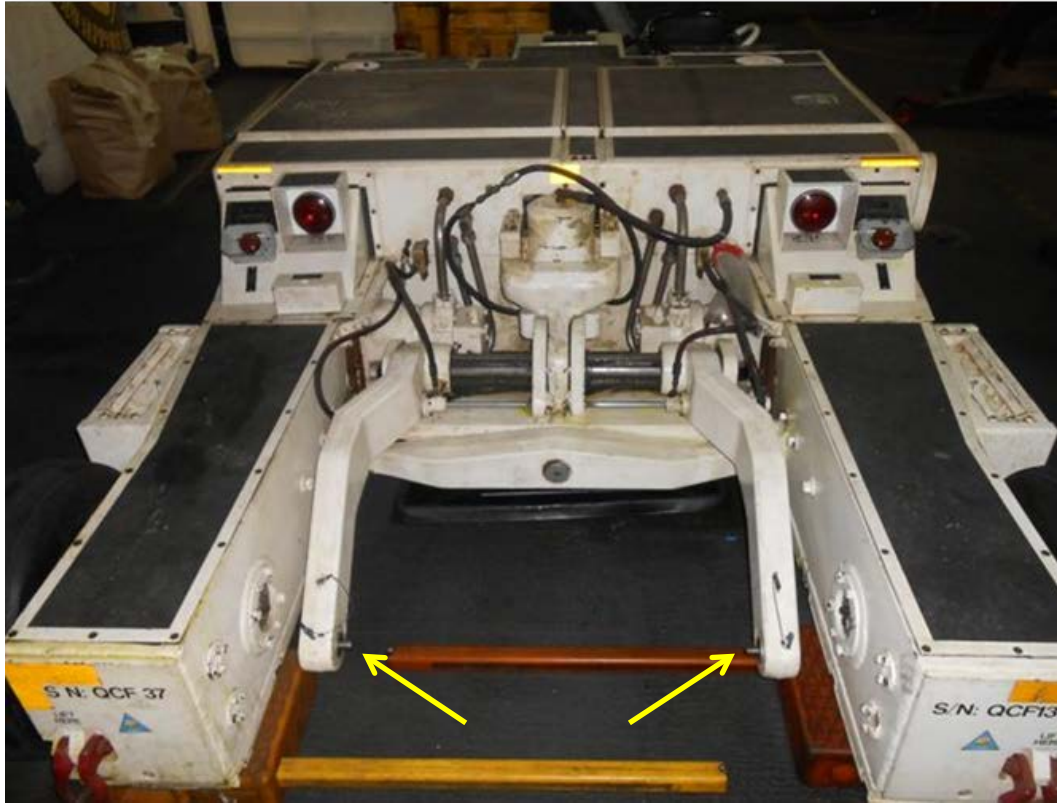


Figure 6-90. A/S32A-32 Spotting Dolly (S/N QCF137) with lifting pins installed at indicated areas of the lifting arm. Spotting Dolly located onboard the USS Harry S. Truman (CVN 75).

6.2.2.4 Inspection

Demonstration components were evaluated during regularly scheduled NDI inspections established at a 91 day interval (see Table 6-51). Feedback reports were provided to NAWC-AD Lakehurst via email by the ASCS VanSteinburg of AIMD for the CVN 75. Most reports were received as qualitative assessment to minimize impact upon fleet operations. Notable comments included:

- nCoP lifting pins exhibited no defects
- Several legacy lifting pins were replaced due to failed NDI inspection.
- nCoP lifting pins were easier to prepare for NDI as compared to legacy components thus reducing inspection man-hrs by eliminating the need for bead blast.

Field photos were obtained during Sept 2013 and Jan 2014 and are presented as Figure 6-89 and Figure 6-90.

Table 6-51. Summary of inspection results received from USS Harry S. Truman (CVN 75).

Dem/Val Inspection	Date	Criteria	Observation	Conclusion
Installed	Mar 2013	N/A	New Installation	N/A
1	Jun 2013	No visual presence of corrosion or cracking	No Defects Observed	PASS
2	Sept 2013	No visual presence of corrosion or cracking	No Defects Observed	PASS
3	Dec 2013	No visual presence of corrosion or cracking	No Defects Observed	PASS
4	Mar 2014	No visual presence of corrosion or cracking	No Defects Observed	PASS
Final	June 2014	End of Demo	End of Demo	Remain in Service



Figure 6-91. Visual appearance of demonstration component following 91 Day NDI examination aboard CVN-75 Sept 2013 (left) and Jan 2014 (right).



Figure 6-92. USS Harry S. Truman reported that approximately 672 aircraft moves have been performed at time of photo.

6.2.2.5 Field Performance Summary

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

According to ASCS Charles 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 [turnaround time] (TAT) 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 NDI inspection to remove a corrosion preventative coating, and then reapplication, after 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 inspection 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 NSWPCD. Technical points of contact for initiating, organizing and performing field demonstrations are presented as Table 6-52.

Table 6-52. Demonstration Points of Contact.

POC	Organization	Location
Denise Aylor	Corrosion Engineering	NSWCCD (Bethesda)
Jeff Dinges	M9ACE	NSWC (Panama City)

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 OD surface selectively as shown in Figure 6-93.



Figure 6-93. 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 exposure. The M9 ACE vehicle selected for the Dem/Val was NSWCCD's Program Management (PM) 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 6-94 shows the prototype Dem/Val nCoP coated apron cylinder after installation on the M9 ACE vehicle, and Figure 6-95 shows the unassembled hydraulic cylinder after the field test exposure on the M9 ACE.



Figure 6-94. Image of installed prototype nCoP Dem/Val cylinder assembly on the M9 ACE.

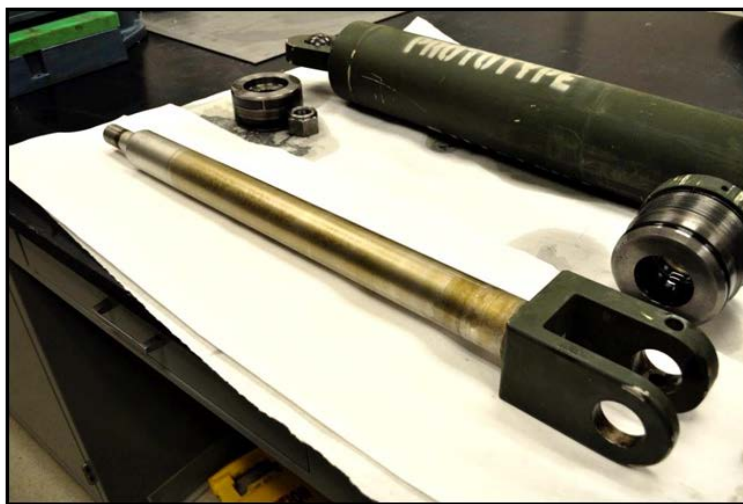


Figure 6-95. Image of unassembled prototype Dem/Val cylinder assembly following two months of field exposure on the M9 ACE.

7.0 COST ASSESSMENT

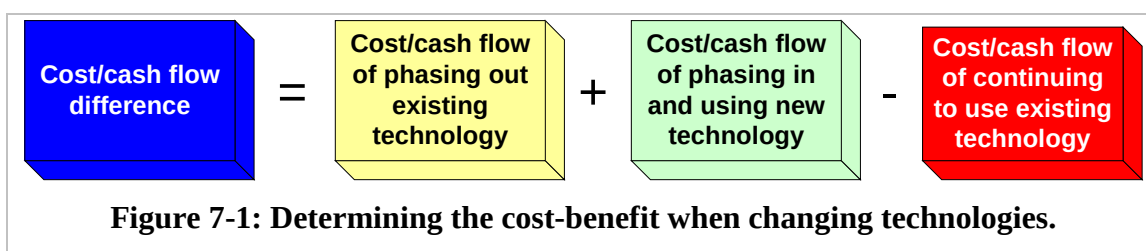
7.1 COST MODEL

7.1.1 Overview

C-MAT (Calculation for Material Alternative Technologies) is an Excel-based cost analysis decision tool developed by Rowan Technologies, Inc. to determine the costs and benefits of re-engineering weapons systems. It can be used for analyzing any engineering change, including redesign, applying new materials or coatings, upgrading avionics, etc.

It was developed under funding from SERDP for analysis of the cost benefit of re-engineering aircraft landing gear with a new high strength stainless steel. We have since modified and used it for a variety of projects, including analyzing the cost of adopting new coatings in the depots, re-engineering gun barrels and analyzing the relative costs of using chromates versus clean alternatives throughout the weapons system life of the Joint Strike Fighter. Because it was developed specifically for DoD using DoD funding, it is of course available to DoD free of license fees.

Most cost-benefit analysis compares the total life cycle costs of using different engineering designs or technologies. The C-MAT tool, on the other hand, compares the costs of using the current engineering design or technology with the cost of both *adopting and using* an alternative (Figure 7-1). However, C-MAT can be used equally well to decide between two alternatives prior to adopting either of them. The output is given in both cost and cash flow so that it can be used commercially as well as in DoD. In fact, as DoD depots increasingly operate as business units, they are likely to find cash flow analysis more useful than cost analysis since it factors in customer pricing.



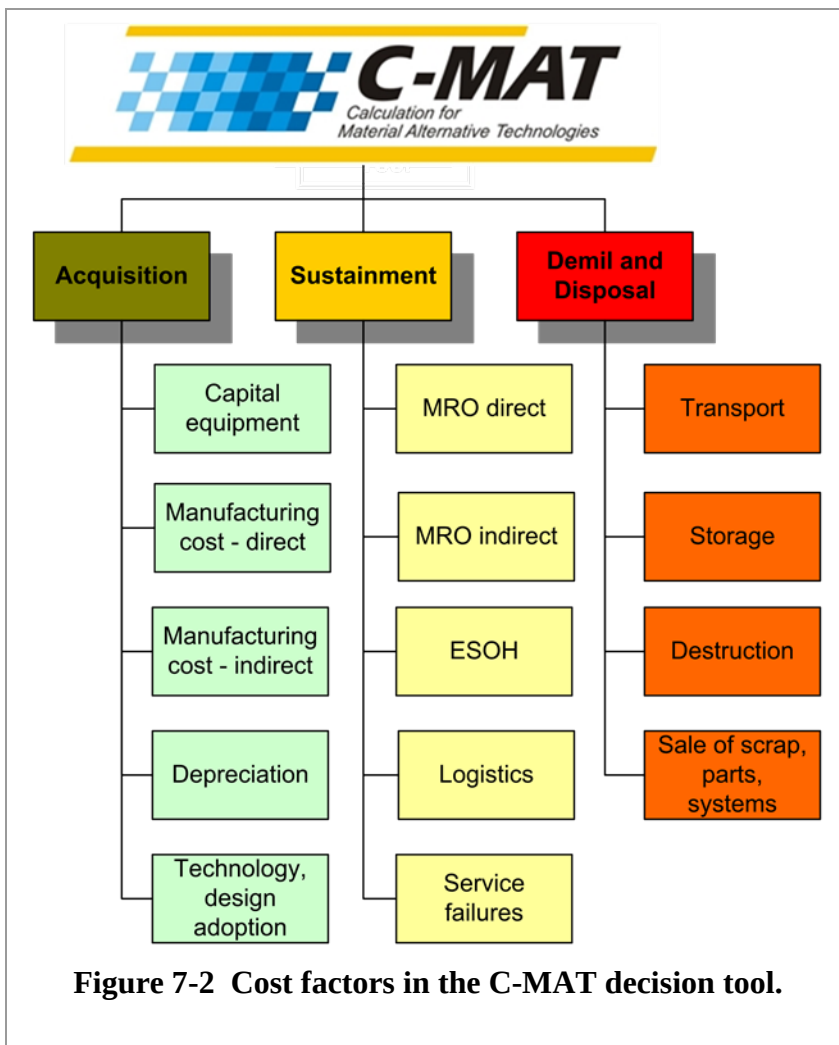
The heart of the tool is the Scenario, a Scenario being a model of the cost structure for the life cycle use of a particular technology or material, or for operations at a particular site. The tool has been designed to facilitate decisions by making it easy to vary scenarios for different design decisions or for different ways of adopting a new technology. Once the first scenario is built, modifying it into other scenarios is usually fairly easy, after which comparing the costs of different approaches is a simple matter of choosing which scenarios to compare.

Financial performance is provided both in tabular and graphical form, and includes Return on Investment (ROI), Net Present Value (NPV), Internal Rate of Return (IRR), and Payback Period. In addition, the costs are broken out by source, making it easy to see where the costs come from in each scenario. The user can provide the estimated accuracy of each input value to derive the

accuracy of the overall calculation. This often provides a necessary reality check on how good our information really is.

7.1.2 C-MAT Details

Figure 7-2 shows the cost factors that are included in the decision tool. Each type of cost (capital, manufacturing cost, etc.) is entered on a sheet that includes all the most common cost details, with the capability of entering other costs peculiar to any particular system. The model covers both OEM manufacturing and life cycle sustainment, and is designed for military or commercial use. Basic data such as inflation, cost of money and type of depreciation (if any) are entered as global variables. The model for each scenario is then built by inputting the ways in which costs will vary under that scenario. Below we briefly summarize the most important aspects of each type of cost factor.



Capital costs – Capital costs can be expended over a number of years, and can be undepreciated or depreciated using the standard Straight Line, Sum-of-years' Digits, Declining Balance or Double Declining Balance methods.

Manufacturing costs (direct) – These costs, which can be used for original manufacture and for overhaul, include materials, labor, finishing, QC, utilities, etc. Values can be input for a specific component or subsystem, an assembly of components, a whole weapons system, or a manufacturing or overhaul site such as a depot. Also included, if needed is cost of money for time in process, which allows us to account for the value of faster turnaround time.

Indirect manufacturing costs – These include such items as inventory cost, training, cost of money, legal costs, depreciation of plant and equipment, and cost of sales for commercial

operations. Inventory can be modeled in a number of ways to accommodate different methods of determining how much inventory to hold and when to put items into inventory or take them out.

Environmental costs – This includes regulatory costs, permitting, licensing, hazardous waste disposal and the costs of running pollution control equipment. Because environmental costs vary widely with each weapons system and material the model allows the user to input a wide variety of annual and per-item costs.

Field failure – Service failures are a major cost item for some weapons systems. The model allows service failure costs to be gathered and input from historical records, including all direct and collateral costs and the cause of each failure. This permits the user to predict how an alternative technology that changes failure mechanisms would be expected to change the probability of failure and hence it's probable cost and its variance. This has been used, for example, to analyze the cost-benefit of replacing Cd-plated landing gear steel with more expensive stainless steel.

Fleet build, replacement and overhaul rates – The model permits the user to input a model for the rate at which an entire fleet of systems will be put into service, maintained and retired. Overhaul and replacement rates are calculated from expected system or component lives. Thus, if a change is expected to increase the time between overhauls or permit a larger number of overhauls before replacement, those changes can be modeled and will automatically enter the cost-benefit calculation.

Logistics – Logistics costs include packaging, shipping and storage for US and overseas deployment, as well as shipping items back and forth for maintenance and repair.

Demil and disposal – The costs incorporated here include transport, storage and destruction based on the model for fleet retirements and the number of items replaced over the life cycle. In addition it allows for some costs to be recouped through system or subsystem reclamation or sale, as well as sales for scrap.

7.1.3 Typical Use Cases for C-MAT

New weapons system decisions – The model can be used to compare the life cycle cost of choosing different options, factoring in differences in R&D cost, capital cost, overhaul cycle, and the probability and cost of service failures.

Changes to legacy weapons systems – The tool calculates the cost of continuing to use the current technology versus the cost of phasing out that technology over time, while phasing in and then using an alternative. It therefore includes the cost of development, training, drawing and Tech Order changes, as well as the effect on maintenance cycle time, service reliability and inventory requirements.

Evaluating alternative ways of making a change – For currently fielded systems a change might be made in various ways, including immediate wholesale replacement, gradual replacement over time, replacement at overhaul, or replacement at failure,. Each of these has its costs and risks. Since the tool continues to calculate costs due to overhaul frequency and service

failure for all systems currently in service with the existing technology as well as doing so for its alternative, it is possible to build each scenario and compare the risks and costs of each approach to find the lowest cost option.

Risk reduction – Because the tool incorporates the effect of data accuracy on life cycle cost, it is possible to determine the probability of different financial outcomes. In addition it is possible, not just to carry out sensitivity analysis, but also to see how the probability of a particular outcome is affected by the accuracy with which the costs are known. This makes it possible to determine where better information is needed.

7.2 COST ANALYSIS AND COMPARISON

7.2.1 Environmental Assessment

Eliminating chrome plating at FRCSE, Jacksonville will remove the major use chromic acid and hexavalent chromium air emissions, which must be removed by a scrubber:

- Cr^{6+} -contaminated wastewater – 300,000 gal/yr
- Sanitary water discharge – 300,000 gal/yr
- Chrome wastewater disposal due to upsets – 12,000 gal/yr
- Chrome plating tanks – 3 tanks, 1,500 gal each

Chrome plating also contributes to contamination of shoes and clothing, which raises the risk of carrying hexavalent chromium from the regulated work areas into break rooms and other unregulated areas contributing to OSHA housekeeping problems.

In 2006 OSHA promulgated new rules on “Occupational Exposure to Hexavalent Chromium” [28]. This rule not only defined a new, lower PEL for Cr^{6+} , but also instituted rules on personal protection, regulated areas and housekeeping.

These rules include separation of hex Cr-contaminated work areas (where Cr^{6+} may be generated by sanding, cleaning, chromating, plating, painting, depainting, etc.) from other work areas and from offices, break rooms, etc. It also includes provision for changing to and from street clothes, and showering at the end of the work day. These changes have required facilities modifications in many depots and maintenance shops. In many ways, however, the most expensive change has proved to be a requirement for maintaining non-regulated areas in a clean state with no detectable hexavalent chrome as determined by surface wipes. This requires daily cleaning of all surfaces that can be touched by workers, including tables, door knobs, refrigerator handles, etc. in break rooms.

The rules were kept in abeyance for several years by legal challenges, but since these legal proceedings were resolved the rules have been aggressively enforced, leading to numerous citations at several depots. FRCSE Jacksonville has kept track of these compliance costs, providing cost data that is expected to be typical of the cost to depots of continuing to use all Cr^{6+} processes under the new OSHA requirements.

7.2.2 General Inputs and Assumptions

1. With the adoption of OSHA housekeeping rules, the most important cost for depots has become the cost of compliance with these rules. These costs comprise:
 - a. A one-time cost for equipment and facilities modifications.
 - b. The annual labor and materials costs for showering, building cleaning and production meetings.

Because these costs cannot be eliminated, or even substantially reduced, until most of the chromate processes and materials are removed from the depot, these costs have been captured but not included in the CBA.

2. The plating workload is based on the average annual ampere-hours, taken from shop records from Jan 2010 to end of July 2013. Most calculations in the present analysis are provided in terms of annual cost and cost per sq.ft. plated.
3. Note that all current OD plating that can be done by HVOF is being migrated to HVOF, and eventually only the ID workload (all of which is J52 engine parts) will remain in JAX chrome plating operations.
4. The calculations assume plating tank sizes of 1500 gals for each of the stripping, activation, and plating operations in both the Cr and nCoP process lines. The masking tanks (wax for Cr, peel coat for nCoP) are assumed to be smaller at 700 gals, and the Co anode tank for nCoP only 10 gals.
5. It is assumed that the plating tanks are dummied once a month for Cr and once a week for nCoP, and that each dummie requires 1 hr of labor.
6. 24/7 operation is assumed for:
 - Plating tank heating (by steam) for both Cr and nCoP plating
 - Rinsewater and makeup water use
 - The Cr^{6+} scrubber, and also EPA NESHAP monitoring of Cr^{6+} (monitoring actually required only when Cr plating operations occur)
7. Plating parameters for nCoP are 116A/sq.ft., 20V, 185°F and for Cr 432A/sq.ft., 28V, 140°F.
8. Power required for the activation step is ignored, because it is small.
9. For nCoP ID plating, it is assumed that the anodes will be Ti rods, Co plated in a special Co anode tank (rather than electrolytic Co squares in a basket). The rods will be plated to a thickness of 20 mils, enabling 10 ID parts to be plated from each anode rod in 1-2 runs.
10. Hazardous waste:
 - At JAX, Cr^{6+} waste from all Cr stripping and chromating operations is combined with waste from the Cr plating line. But the Cr^{6+} waste numbers used in this analysis are estimates for plating operations only.
 - The scrubber, with its \$50,000 annual cost for energy and maintenance, is used solely for the chrome plating line.
11. EPA NESHAP non-compliance fines are not included in the environmental costs for Cr plating, since such fines have never been levied on JAX. However, the penalties are substantial - \$32,500 per day per violation.
12. OSHA monitoring costs for Cr plating are not included in the cost model. This is because it has been found that the exposure level of plating personnel to Cr^{6+} under typical plating conditions are low enough that ongoing monitoring is not required. An annual skin test is carried out, however.

7.2.3 Cost-Benefit Analysis for FRCSE

7.2.3.1 Cost of continuing to use Cr⁶⁺

In most cost-benefit calculations the baseline is simply the historical cost associated with continuing to use the current process. With the advent of the new OSHA rules, the cost of continuing to use Cr⁶⁺ processes must now include the additional capital and annual compliance costs imposed by the new OSHA standard, which are very high. At FRCSE Jacksonville the initial cost of compliance was about \$1.5 million, or the annual compliance cost is \$1.15 million, primarily for the labor time and consumables required to the housekeeping requirements (see Table 7-1).

Table 7-1. Cost of OSHA hexavalent chrome regulations.		
Cost item	One-time	Annual
Capital expenditures:		
Installation of facilities (showers, clothing storage, etc.)	\$0	
Building modifications (walls and barriers, relocation of hex Cr producing processes, relocation of offices, etc.)	\$350,000	
Facilities/equipment for dust and fume control	\$840,000	
Heavy Metals Cleaning: One time cost: (Equipment, etc)	\$264,749	
Other		
Labor costs:		
Time for changing, showering, etc. per worker per day		\$140,000
Labor cost of housekeeping (cleaning, decontamination)		\$640,000
Labor hours for meetings, etc		\$18,000
Other		\$5,000
Recurring Annual Cost: (Consumables, Materials, Laundry)		\$350,697
Total	\$1,454,749	\$1,153,697

Note, however, that although they can be reduced, these costs cannot be eliminated all the while there is some usage of Cr⁶⁺ in the depot and while corrosion control and repaint operations continue to be required with chromated paint systems. Although some of the annual cost of OSHA hexavalent chromium compliance doubtless derives from chrome plating operations, there is no way to assign a portion of the annual cost to this source. **Therefore, we have not included any portion of this cost in this analysis.**

7.2.3.2 Environmental cost

In addition to the very high regulatory costs in Table 7-1 imposed by the OSHA hexavalent chrome regulations, the use of hexavalent chromium imposes the costs we more commonly referred to as environmental regulatory cost that is summarized in Table 7-2. These include hazardous waste management, wastewater disposal, etc. However the primary cost is the energy cost for running the scrubber that is required to remove Cr^{6+} mist from the air stream to meet the stack emission limits. This equipment is used only for hard chrome emissions. Therefore replacing hard chrome will eliminate this cost.

Another cost that is frequently not taken into account is the annual cost associated with cleanup of hexavalent chrome spills. This cost is of course highly variable, but it cannot be ignored, and it does of course increase the risk of dragging Cr^{6+} contamination from regulated areas into other areas of the plant.

Table 7-2. Environmental cost.

Cost item	Quantity					Unit
	EHC Cost	EHC \$/yr	EHC \$/ft2	nCoP \$/yr	nCoP \$/ft2	
Scrubber energy cost	\$50,000	\$50,000				\$/yr
Scrubber maintenance cost	\$4,000	\$4,000				\$/yr
Stack testing, inspection	\$5,000	\$5,000				\$/yr
NESHAP monitoring of employees	\$0	\$0				\$/yr
NESHAP reporting, training	\$2,400	\$2,400		\$2,400		\$/yr
Haz waste management	\$5,000		\$52.63		\$52.63	\$/yr
Haz waste disposal	\$54,000		\$568.42			\$/yr
Wastewater treatment	\$1,752		\$18.44		\$18.44	\$/yr
Chrome waste water disposal due to upsets	\$14,400		\$151.58			gal/yr
Sanitary Discharge	\$3,693		\$38.87		\$38.87	\$/yr
Environmental total	\$140,245	\$61,400	\$830	\$2,400	\$110	\$/yr
Annual environmental cost @ sq ft/yr =	95	\$78,845		\$10,445		
Environmental total	\$140,245			\$12,845		

7.2.3.3 Capital and adoption cost

Capital cost (Table 7-3) depends on whether an existing line is to be modified or a new line is to be installed. In either case the pulse plating rectifier and carbon filtration are required. The process also requires an etching tank for etching prior to plating.

Note that we assume that the anode will typically be in the form of Co chips in a basket, although a plated rod may be used. This is because for many ID coating applications plated rod is the only viable approach, which requires the installation of a Cobalt plating tank even if it is used for only some of the part production.

Note that, because the nCoP has about 6x the current efficiency of hard chrome, a single nCoP tank can replace several chrome tanks – in the case of FRCSE the single nCoP tank replaces 3 chrome plating tanks.

Table 7-3. nCoP capital and adoption costs.

Cost item	EHC	nCoP
nCoP capital cost		
Stripping tank, rectifier & wiring		\$130,000
Maskant tank		\$50,000
Activation tank, rectifier & wiring		\$130,000
Plating tank:		\$50,000
Plating rectifier & wiring		\$300,000
Tank liner		\$10,000
Carbon filtration equipment		\$20,000
Co anode tank		\$30,000
Nickel or Cobalt or Woods Nickel Strike Tank		\$30,000
Total		\$750,000
nCoP adoption cost (initial M &S)		
Stripping chemicals		\$29,600
Maskant		\$15,000
Plating chemicals		\$18,100
Chemicals for anode tank		\$3,000
Total		\$65,700

7.2.3.4 Production cost

Table 7-4 summarizes the present hard chrome plating actuation at FRCSE Jacksonville and the changes in process time that would occur with the adoption of nCoP.

nCoP plating is far more current-efficient than EHC for two primary reasons:

1. Cobalt in the bath is divalent, so that each Co atom requires only 2 electrons to carry it to the cathode, while Cr atoms require 6.
2. EHC baths are highly inefficient, with most of the power dissipated in hydrolysis, with only about 15% of the power resulting in plating. nCoP plating is about 90% efficient.

As a result of these two factors the deposition per A-hr for Co is about 18 times that of Cr.

The cost of nCoP vs hard chrome plating is summarized in Table 7-3 (general process data) and Table 7-6 (processing costs). Most of the labor cost of electroplating is involved with masking, demasking, setup, insertion and removal from the plating tank, and maintaining the plating system. The shorter plating cycle for Co-P has little effect on the labor hours. Because the nCoP bath requires more maintenance for carbon filtration, and in some cases anode rods must be plated, the labor hours are somewhat higher for nCoP.

Table 7-4. FRCSE general cost data.

Item	Quantity	Unit	EHC \$/yr	nCoP \$/yr
Annual amp-hrs (Jan 2010- July 31 2013)	727,875	A-hr		
Typical plating thickness	0.020 - 0.025	in		
Rebuild thickness after grind	0.010 - 0.015	in		
Area plated/yr	95	sq ft		
Max component size (dimensions)	54" x 6" x 22"			
Max component size (plated area)	4.5	sq ft		
Tank size (gal)	1,500	gal		
Tank size (dimensions)	144"x36"x72"			
Number of plating tanks	3			
Annual man-hrs for Cr plating	726	hr	\$56,628	
Annual man-hrs for nCoP plating	766	hr		\$59,748
Annual Labor cost			\$56,628	\$59,748
Average production time	EHC	nCoP	Unit	
Number of plating tanks	3	1		
Current efficiency (%)	15%	90%		
Current density	0.25	0.81	A/sq in	
Deposition rate	0.75	5.48	mils/hr	
Batch size	1 to 2	1 to 2		
Stress relief bake	4.00	4.00	hr	
Mask	2.00	2.00	hr/item	
Etch	0.08	0.08	hr	
Plate	30.0	4.1	hr	
Demask, clean up	1.00	1.00	hr/item	
H relief bake	23	23	hr	
Grinding	1.50	1.50	hr	
Average total production time	61.6	35.7	hr	

The cost of nCoP plating chemicals is higher, as is the cost per bath of utilities because of the higher bath temperature, which requires more steam power for bath heating and more make-up water to replace evaporant. However, since we are replacing three chrome baths with a single nCoP bath the total utilities consumed is far lower.

In order to evaluate the cost of maintaining a higher electroplating bath temperature, the evaporation rates at the hard chrome bath temperature (140°F) and the Co-P bath temperature (185°F) were estimated from the NMFRC Plating Tank Evaporation Calculator²⁹, and these estimates were used to determine the total replacement water needed and the energy required to bring that water back to the plating temperature. The calculations show that maintaining a single nCoP tank at 185°F costs almost precisely the same as maintaining three chrome tanks at 140°F (see Table 7-5).

The nCoP direct M&S cost is about 60% higher than hard chrome plating (Table 7-6). This is because, as shown in Table 7-4, the plating volume is only 95 sq ft in the base year, spreading all

the fixed costs over a very small production volume. However, the direct production cost is an order of magnitude lower than that of hard chrome primarily because of the far lower plating power requirements (see Table 7-6). Note, that the chrome plating workload at FRCSE is very low, and the unit area cost would be significantly lower at a depot with a higher workload such as OO-ALC where the fixed environmental and capital costs would be spread over a far larger workload.

Table 7-5. Calculation of plating bath evaporation and energy use – all plating tanks (3 EHC, 1 nCoP).

	EHC	nCoP
Tank T (°F)	140	185
# tanks	3	1
Tank surface area	5,184	5,184
Incoming water T (°F)	50	50
T differential (°C)	50	75
T differential (°F)	90	135
Evaporation rate (gal/hr)*	11.58	12.75
Evaporation rate (gal/yr)	101,441	11,690
Evaporation rate (liter/yr)	384,461	423,305
Spec heat water (kJ/liter/degC)	4.2	4.2
Spec heat water (kWh/liter/degC)	0.00117	0.00117
Spec heat water (Btu/gal/degF)	8.3	8.3
Energy to raise water to bath T (kWh)	22,427	37,039
Energy to raise water to bath T (MBtu)	76	126

7.2.3.5 Effect of Rework

Hard chrome plating is a very forgiving process that continues to plate even when tank chemistry or plating conditions are not optimum. It has been found that nCoP, on the other hand, is much more sensitive to contamination in the bath, particularly in the form of hydrocarbons.

Contamination causes defects in the coating, necessitating stripping and replating. While contamination can be controlled through proper bath maintenance, including carbon filtration and periodic dummieing, we would expect that the incidence of rework would increase to some extent with the use of a more sensitive process.

At this point there is insufficient experience with production nCoP plating to assess the expected cost of rework.

Table 7-6. Materials, supplies and utilities.

Materials and supplies	EHC/yr	nCoP/yr	Unit	EHC \$/yr	EHC \$/ft2	nCoP \$/yr	nCoP \$/ft2
Plating tank chemicals	\$4,800	\$4,690	\$/yr		\$50.53		\$49.37
Etch tank chemicals		\$2,500	\$/yr				\$26.32
Other materials	\$4,690		\$/yr		\$49.37		
Masking materials		\$6,500	\$/yr				\$68.42
Anodes and anode materials		\$1,700	\$/yr				\$17.89
Total M&S (annual) at sq ft/yr=95	\$9,490	\$15,390			\$99.89		\$162.00
Utilities	EHC/yr	nCoP/yr	Unit	EHC \$/yr	EHC \$/ft2	nCoP \$/yr	nCoP \$/ft2
Plating bath temperature	140	185	deg F				
Water use (dragout)	118,239	118,239	gal/yr		\$9.58		\$9.58
Water use (evaporation)	101,441	111,690	gal/yr	\$781		\$860	
Electricity (plating power)	727,875	44,363	kWh		\$1,080.32		\$65.84
Tank heating - energy to heat makeup (dragout)	88.75	133.13	Mbtu		\$28.76		\$43.15
Tank heating - energy to heat makeup (evaporation)	76.14	125.75	Mbtu	\$2,344		\$3,872	
Rinse water use/waste water treatment	25,000	25,000	gal/yr		\$5.27		\$5.27
Total				\$3,125	\$1,124	\$4,732	\$124

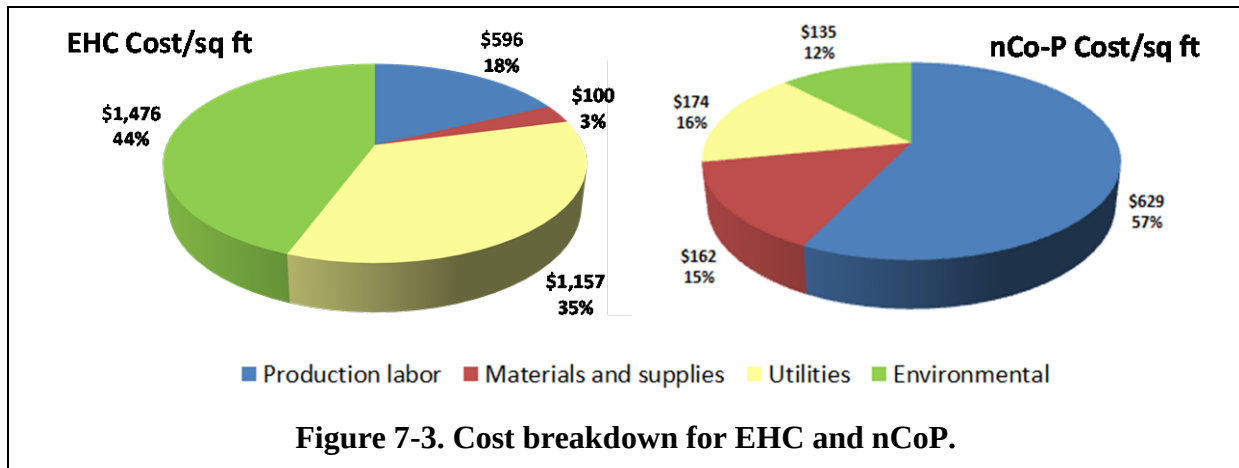
7.2.3.6 Cost breakdown summary

Taking all of the above costs into account, the cost breakdown is shown in Table 7-7. Although the cost for nCoP supplies is significantly higher, the utility and environmental costs for nCoP are very much 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 7-3. 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 we were to include the OSHA compliance costs in Table 7-1, of course, the cost of chrome plating would be completely overwhelmed by them. We have not done so because the savings will only be realized when almost all the Cr^{6+} -producing materials and processes are eliminated.

Table 7-7. 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



7.2.3.7 Cost-benefit for replacing hard chrome plating with nCoP

As noted above, the cost of continuing to use Cr^{6+} processes (Section 7.2.3.1, Table 7-1) 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 7-8 assuming an accuracy of 20% in most of the inputs.

Table 7-8 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 7-8. 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 7-9. 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 FRC-SW (North Island) is approximately 5 times the FRCSE (Jacksonville) workload. Simply substituting the higher workload into the model produces the right-hand column of Table 7-9 (i.e. we take that same cost structure as

FRCSE but substitute a workload comparable with FRC-SW). This doubles the IRR, but does not make a large difference in NPV or ROI. However, it produces such a large annual cost change that the payback occurs within the first year. This is shown in Figure 7-4, where the cumulative cost is shown for workloads typical of FRCSE and FRC-SW.

Table 7-9. 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, equal to FRC-SW
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 (Jacksonville); the numbers do not reflect the cost structure at FRC-SW 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.

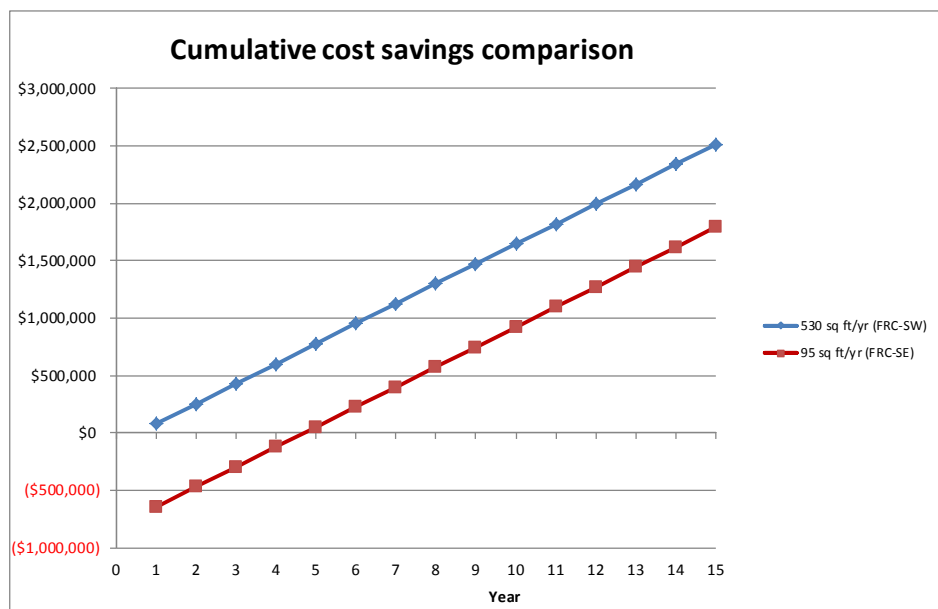


Figure 7-4. Comparison of cumulative cost savings for nCoP implementation at a low volume depot (such as FRCSE) and high volume depot (such as FRC-SW).

Note that the cost-benefit impact of any changes in performance of nCoP vs hard chrome have not been taken into account in this analysis. This is because the laboratory results show similar wear and corrosion performance for most applications, with the exception of abrasive wear, where the nCoP performance is lower.

7.2.3.8 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 hexavalent chromium compliance, replacement of hard chrome plating will be an essential component of hexavalent chromium elimination.

Electrochemical data taken in the course of this program show that the galvanic potential (open circuit potential, OCP) of nCoP is very close to that of aerospace aluminum alloys such as 2024 Al. This raises the possibility of using nCoP in direct contact with aluminum airframes (e.g. for bushings) without the need for Cd plating, chromate conversion, or other galvanic protection schemes. Of course this also means that bushings made of this material would be galvanically incompatible with stainless steels and nickel-based alloys, both of which are commonly used for fasteners, pins and shafts that run through bushings, but it is generally preferable to remove and replace bushings than to repair corrosion in airframes themselves. Therefore it might be viable to consider adoption of this coating on bushings in place of Cd or similar cathodic materials (or equivalently the use of electroformed nCoP bushings).

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 CoP 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 maintain solution level control and also impacts the ability to utilize conventional electroplating wax formulations for selective electroplating. Use of a level control sensor may be used to maintain solution level thus mitigating risk. For selective masking, as a result of elevated temperatures, the use of thermoplastic 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 to meet the following operating conditions:

- Average Current Density:
 - o 107 – 188 mA/cm²
 - o 100 – 175 A/ft²
- Duty Cycle = 50%
- Pulse Frequency = 25 Hz
- Voltage = 20V Maximum

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 Joint Test Protocol for WP-0936.

Another approach to improve abrasive wear performance is by incorporation of hard ceramic particles. Integran has developed Nanovate CoP-X coating process which behaves similarly to Nanovate CoP (commercial name of 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 CoP-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 nanosized 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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