# Technical Article

# Nano-particle Composite Plating as an Alternative to Hard Chromium and Nickel Coatings

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As part of an effort to evaluate alternatives to electroplated hexavalent chromium coatings, screening tests were performed on numerous nanostructured coatings or amorphous coatings containing nano- or micro-particles. The objective was to determine if improvements in performance could be obtained with decreasing grain and/or particle size. Electrodeposited, nanocrystalline cobalt, with and without tungsten carbide particles, and electroless, mid-phosphorus nickel (ENi-P) coatings with various sizes of diamond particles (150, 1000, 2000 and 150+1000 nm) were selected for investigation. Preliminary results suggested that additional electroless composites should be investigated. These coatings included nickel-cobalt phosphorus (ENi-Co-P), cobalt phosphorus (ECo-P) and nickel boron (ENi-B), all with and without codeposited diamond particles. For baseline comparisons, electrodeposited polycrystalline cobalt and electroless nickel coatings were deposited without occluded particles. This paper discusses the results obtained from the screening tests, which included adhesion, thickness analysis, hardness and abrasive wear resistance. The results suggest that all of the ENi-P, ENi-Co-P and ECo-P processes with occluded diamond particles have the potential to impart the required adhesion, hardness and tribological properties, while reducing the environmental impact of chromium plating processes.

## Nuts & Bolts: What This Paper Means to You

This work is an ambitious evaluation of hexavalent chromium replacements. In particular, a number of electroless composites, with and without codeposited nano- and micro-particles, were studied. The goal was to find coatings with the same, or better, adhesion, hardness and wear properties, when compared to hard chromium obtained from hexavalent baths.

# **Background & Objective**

Hexavalent chromium is used extensively to finish surfaces within the Department of Defense (DoD) and private industry due to its properties and decorative appeal. However, the environmental, health and safety issues associated with hexavalent chromium have led to stringent regulations regarding its use. Reductions in permissible exposure limits and public owned treatment works discharge limits have escalated the burdens associated with using hexavalent chromium. Therefore, the search for viable alternatives to electroplated hard chromium has become a high priority. In response, various DoD agencies have directed efforts towards identifying and evaluating viable alternative processes. The Hard Chrome Alternatives Team (HCAT) and the Air Force Research Laboratory (AFRL) are addressing near-term solutions to replacing hard chromium for both line-of-sight and nonline-of-sight applications, respectively.

For many years, HCAT has been investigating and validating high-velocity oxygen fuel (HVOF) technology as a potential hard chromium alternative. While HVOF technology may be able to meet the required performance characteristics of hard chromium, it cannot replace it in all applications because it is a line-of-sight process. Therefore, even with the implementation of HVOF coatings, users would need to use hard chromium for some components. For example, non-line-of-sight requirements comprise about 20 to 40% of all hard chromium applications within the Air Force.

To address the non-line-of-sight need, the AFRL and Concurrent Technologies Corporation (CTC) established the "NLOS Hard Chromium Alternatives" project. This

\* Corresponding Author: Melissa Klingenberg Concurrent Technologies Corporation 100 CTC Drive Johnstown, PA 15904 Telephone: (814) 269-2545/6415 Facsimile: (814) 269-6847 E-mail: klingenb@ctcgsc.org project established hard chromium needs and requirements per Air Force Air Logistic Center (ALC) operations and identified over 100 possible alternatives. Included among these were electrochemical deposition processes that enabled the production of nanostructured coatings, as well as processes that produced amorphous structures and enabled the codeposition of nanoparticles. Inclusion of nanoor micron-sized particles into a metal matrix is sometimes called composite plating, and is a type of occlusion plating.

Nanostructures have been shown to exhibit interesting properties. Typically, as the grain size of a material decreases, its hardness, fracture toughness and yield strength increase. This effect is known as the Hall-Petch effect.<sup>1.4</sup> Because nanostructured coatings offer the promise of improving the hardness and wear properties of conventional, softer, protective coatings, the AFRL established an effort to investigate the suitability of nanoparticle composite plating processes as long-term replacements for hard chromium.<sup>5</sup>

# **Proof-of-concept study**

A proof-of-concept study was conducted to identify and evaluate commercially available, or near commercial, nanocomposite coatings. A literature search, vendor search and personal contacts were used to identify processes that could be used to create nanocrystalline matrices, or codeposit nanoparticles within a metal matrix (microcrystalline, nanocrystalline or amorphous).<sup>6</sup> Information regarding these processes and the testing performed on the coatings for the proof-of-concept study are outlined in the following sections.

#### Coatings selected

A vendor ("A") of nanocrystalline electrodeposits suggested that their nanocrystalline cobalt coating be tested. They also recommended that a second coating be developed to incorporate tungsten carbide (WC) particles within the nanocrystalline cobalt (Nano-Co) matrix. However, they were unable to obtain WC particles less than 2000 nm in diameter. Consequently, they focused their attention on producing coatings with nanostructured grains rather than occluded nanoparticles. However, they did produce a set of samples with the 2000-nm WC particles embedded in the cobalt matrix.

A second vendor ("B") was found that could either deposit nanocrystalline coatings or codeposit nanoparticles within a microcrystalline, nanocrystalline or amorphous matrix. This company was selected because of its existing knowledge of occlusion plating, its willingness to accommodate special processing requests based on their commercial baths and its willingness to adapt their process to accommodate smaller particles than what they currently used (*i.e.*, 2000 nm). The coating matrix selected was based on an electroless nickel, mid-phosphorus (ENi-P) process. Diamond particles (2000 nm, 1000 nm and 150 nm in diameter) were selected to be occluded in this matrix. In addition, samples with electroless nickel-cobaltphosphorus (ENi-Co-P), electroless cobalt-boron (ECo-B), and electroless cobalt-phosphorus (ECo-P) coatings, with and without occluded, 1000-nm diamond particles were prepared.

Baseline data were established with polycrystalline cobalt (Poly-Co) and ENi-P coatings, both without occluded particles, to try to determine the level of improvement imparted by nanostructured grains and particle incorporation, respectively. A third company ("C") provided the Poly-Co coatings, and Company ("B") provided the mid-phosphorus ENi-P baseline coatings. Data from previous studies were used to provide the electrolytic hard chromium benchmark for comparison. A summary of the various coating systems selected for study is presented in Table 1.

#### Table 1 Proof-of-concept coatings evaluated

Category	$\textbf{Coating(s) Applied}^{\dagger}$	Vendors
Baselines	EHC ENi-P (mid-phosphorus) Nano-Co without particles Poly-Co without particles	B A C
Nano- structured matrix and	Nano-Co with 2000 nm tungsten carbide particles ENi-P with 150, 1000, 2000 and 150+2000 nm diamond particles	A B
occluded micro- and	ENi-Co-P with 1000 nm diamond particles	В
nano- particles	ECo-P with 1000 nm diamond particles	В
	ECo-B with 1000 nm diamond particles	В

 $^{\dagger}$  Company B heat treated their coated samples at 350°C (662°F) for two hr.

# Coating application

The vendors prepared nine flat, 1010 cold-rolled steel panels (three each with dimensions of  $4 \times 4$ ,  $1 \times 4$ , and  $1 \times 1$  in.), and then applied their coatings. The requested target coating thickness was a minimum of 2 mil (0.002 in.). Company B used a heat treatment of 350°C (662°F) for two hr for all their coated samples to improve their properties. However, Companies A and C did not use a heat treatment, but supplied their samples "as plated." Once vendor processing was complete, the panels were returned for inspection, testing and evaluation.

#### Coating testing

The testing performed on the coatings is outlined in Table 2. In some instances, the vendors performed additional characterization of their coatings. These data have been incorporated into the discussion of the results, where appropriate.

### **Test results**

A summary of the test results is given here, and correlations between composition, structure and properties have been made, where possible, in the "Summary of findings" section of this paper. Typical hard chromium data are included so that the alternative coatings evaluated may be compared to the currently-used coating. A more detailed presentation and discussion of test results is available.<sup>5</sup>

For any of the evaluated processes to be considered a viable alternative to hard chromium, it must meet or exceed various performance characteristics. Namely, the alternative processes must meet or exceed all guidelines outlined in "Federal Specification Chromium Plating (Electrodeposited) QQ-C-320B for Class II Engineering Plating." Table 3 provides the desired properties, per QQ-C-320B, for the tests conducted during proof-of-concept activities.

# Table 2 Evaluation test matrix

Test	Test Method	Panel Sizes (in.)	No. of Panels per Test	
Metallographic thickness	ASTM B487	1 × 1	3†	
Bend adhesion	ASTM B571	1 × 4	3	
Microhardness	ASTM B578	1 × 1	3†	
Taber wear resistance	ASTM D4060	4 × 4	3	

<sup>†</sup> The same panels were used for both these tests.

# Table 3 Electroplated hard chromium property requirements

Parameter	QQ-C-320B Requirements		
Quality	Plating shall cover all specimen surfaces. Plating shall be free from beads, nodules, jagged edges and other irregularities. Plating shall be smooth and uniform, dull matte or bright, as required. Plating shall be smooth; fine-grained; free from blisters, pits, nodules, excessive edge build-up, contamination, excessive contact marks and contain minimal staining or discoloration.		
Thickness	For Class II Engineering Plating, a minimum of 0.002 in. (or as agreed upon by contract) shall be measured at several locations on accessible surfaces.		
Adhesion	At a magnification of 4X thickness, no separation of the plate from basis metal at interface shall be evident when using a knife or bend test.		
Hardness	850 <sub>VHN</sub> at 100-g load, 10 to 15 sec. Measure each specimen at five locations and take the average of results.		

### Thickness data

Coating thickness was measured in accordance with ASTM B487, "Standard Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross-section." Coated samples were mounted, ground and polished, and then inspected at a magnification of 100 to 200X, using a metallographic microscope. The cross-sections of the ENi-P+diamond particle coatings were photographed. Company B used an instrument with commercial software to determine the approximate distribution of particles within the coating cross-sections. Table 4 summarizes the thickness data obtained for the various samples. Most of the ENi-P coatings supplied were of, or close to the required 2 mil (0.002 in.) thickness. The Nano-Co+2000 nm WC, Set #2, and the ECo-B coatings also were close to the requirement. However, the ECo-P coating was only 0.7 mil thick, and several of the other coatings supplied were only about 1.0 to 1.5 mil thick (*e.g.*, Poly-Co, ECo-P+1000 nm diamond, ECo-B+1000 nm diamond and Nano-Co+2000 nm WC, Set #1). The remainder of the coatings exhibited a marginally acceptable coating thickness. It should be noted that many of these plating baths were not yet in commercial production and that bath chemistry and operating parameters had yet to be optimized. Consequently, at this time, relatively less weight was placed on the thickness data as compared to the adhesion, hardness and wear resistance data.

# Adhesion data

Coating adhesion was analyzed in accordance with ASTM B571, "Standard Practice for Qualitative Adhesion Testing of Metallic Coatings." The findings of the adhesion testing are shown in Table 5 for each coating type.

Of all the specimens tested, only the ECo-B coatings (with and without particles) did not meet the requirements. However, the panels used for this coating were coated once, stripped and etched, and then recoated by the vendor. Such operations may have contributed to the lack of adhesion. In addition, Company B believes that these films were highly stressed, leading to reduced adhesion. However, stress was not measured for these films in this study.

> Table 4 Thickness measurement test results

Coating Type	Thickness (in.)
EHC	0.0020
ENi-P (no diamond)	0.0020
ENi-P + 150 nm diamond	0.0018
ENi-P + 1000 nm diamond	0.0018
ENi-P + 2000 nm diamond	0.0021
ENi-P + 150 + 2000 nm diamond	0.0017
Poly-Co	0.0012
Nano-Co	0.0016
Nano-Co + 2000 nm WC (Sample Set $#1$ ) <sup>†</sup>	0.0014
Nano-Co + 2000 nm WC (Sample Set $#2)^{\dagger}$	0.0027
ECo-P	0.0007
ECo-P + 1000 nm diamond	0.0010
ECo-B	0.0019
ECo-B + 1000 nm diamond	0.0010
ENi-Co-P	0.0016
ENi-Co-P + 1000 nm diamond	0.0017

<sup>†</sup> The first set of samples contains about 10 vol% WC, and the second set contains about 30 vol% WC.

### Table 5 Adhesion test results

Coating Type	Adhesion
EHC	Pass
ENi-P (no diamond)	Pass
ENi-P + 150 nm diamond	Pass
ENi-P + 1000 nm diamond	Pass
ENi-P + 2000 nm diamond	Pass
ENi-P + 150 + 2000 nm diamond	Pass
Poly-Co	Pass
Nano-Co	Pass
Nano-Co + 2000 nm WC (Sample Set $\#1)^{\dagger}$	Pass
Nano-Co + 2000 nm WC (Sample Set $\#2)^{\dagger}$	Pass
ECo-P	Pass
ECo-P + 1000 nm diamond	Pass
ECo-B	Fail
ECo-B + 1000 nm diamond	Fail
ENi-Co-P	Pass
ENi-Co-P + 1000 nm diamond	Pass

 $^{\dagger}$  The first set of samples contains about 10 vol% WC, and the second set contains about 30 vol% WC.

#### Microhardness data

Coating hardness was measured in accordance with ASTM B578, "Standard Test Method for Microhardness of Electroplated Coatings," using the Knoop hardness test. Various loads were used depending on the coating thickness. Figure 1 provides the results of the average hardness for each coating type for Set #1 (upper bar graph) and Set #2 (lower bar graph).

On comparing the test results to the accepted value for hard chromium, it is clear that most electroless deposited films met the requirement. However, none of the electrodeposited coatings, or the ECo-B coatings (with or without particles), achieved



Figure 1—Coating hardness results. The first set of samples contains about 10 vol% WC, and the second set contains about 30 vol% WC.

the required hardness. It does appear that phosphide formation, whether in a cobalt or nickel matrix, was instrumental in improving hardness.

### Taber wear resistance

Wear testing was performed on the vendor-coated,  $4 \times 4$ -in. panels using a Taber wear apparatus in accordance with modified ASTM D4060, "Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser." A CS-10 wheel and a 1000-g load were used over 10,000 cycles.

The Taber wear data are presented as a "wear index" in Figure 2. Lower weight loss (Taber Wear Index) indicates a more wear-resistant coating. Typically, Taber wear evaluations do not include



the initial 1,000 cycles as part of the final analysis. This is largely because nodules and other surface imperfections (loosely bound particles, etc.) are removed during the initial 1,000 cycles and can provide seemingly large wear loss. Consequently, Fig. 2 also includes the index values calculated by subtracting the weight losses in the first 1,000 cycles from the 10,000 cycle total weight loss data.

There were no dramatic differences in Taber Wear Indices for these coatings when comparing the data after 10,000 cycles or 10,000 cycles minus the first 1,000 cycles, with the exception of the Nano-Co+2000 nm WC sample, which contained the greater amount of occluded WC particles. This observation might be attributed to the removal of a relatively larger number of WC particles at the beginning of the test.

Hard chromium displayed a weight loss of between 0.004 and 0.021 g over 1,000 cycles. Accordingly, it was decided to use the lower value for a more rigorous comparison. The 0.004-g loss was extrapolated over 10,000 cycles to give an estimated wear loss of approximately 0.04 g, equivalent to a Taber Wear Index value of 4.0.

As can be seen from Fig. 2, none of the electroplated cobalt coatings (with or without particles) or the electroless coatings without particles provided adequate wear resistance. However, all of the electroless coatings with diamond particles, regardless of their diameter, exhibited wear properties superior to those of hard chromium.

# Summary of findings

The performance data that were obtained for the various coatings supplied by the three companies are summarized in Table 6. The Pass/Fail criteria are based on comparison to the performance of electrolytic hard chromium coatings (see Table 3). As mentioned earlier, the coatings applied by the three companies were not optimized. Consequently, the performance data obtained may not represent the best obtainable. However, these data represent the only information on which to base the conclusions at the present time.

The coatings that were studied represented a mixture of matrices with and without occluded micro- and nanosize particles. The types of matrices studied were as follows:

- Amorphous and pseudo-amorphous matrix: electroless nickelphosphorus, electroless nickel-cobalt-phosphorus, electroless cobalt-phosphorus, electroless cobalt-boron
- Nanocrystalline matrix: cobalt
- · Micro- and macro-polycrystalline matrix: cobalt.

Coating Type	Heat Treated	Thickness	Adhesion	Hardness	Wear Resistance
ЕНС	No	Pass	Pass	Pass	Pass
ENi-P (no diamond)	Yes	Pass	Pass	Pass	Fail
ENi-P + 150 nm diamond	Yes	Marginal	Pass	Pass	Pass
ENi-P + 1000 nm diamond	Yes	Marginal	Pass	Pass	Pass
ENi-P + 2000 nm diamond	Yes	Pass	Pass	Pass	Pass
ENi-P + 150 + 2000 nm diamond	Yes	Marginal	Pass	Pass	Pass
Poly-Co	No	Fail	Pass	Fail	Fail
Nano-Co	No	Marginal	Pass	Fail	Fail
Nano-Co + 2000 nm WC (Sample Set #1) <sup><math>\dagger</math></sup>	No	Pass	Pass	Fail	Fail
Nano-Co + 2000 nm WC (Sample Set $#2)^{\dagger}$	No	Pass	Pass	Fail	Fail
ECo-P	Yes	Fail	Pass	Pass	Fail
ECo-P + 1000 nm diamond	Yes	Fail	Pass	Pass	Pass
ECo-B	Yes	Marginal	Fail	Marginal	Fail
ECo-B + 1000 nm diamond	Yes	Fail	Fail	Marginal	Pass
ENi-Co-P	Yes	Marginal	Pass	Pass	Fail
ENi-Co-P + 1000 nm diamond	Yes	Marginal	Pass	Pass	Pass

# Table 6Summary of coating performance data

<sup>†</sup> The first set of samples contains about 10 vol% WC, and the second set contains about 30 vol% WC.

Any hard chromium alternative used in aerospace applications must be able to:

- 1. Be deposited to the required thickness (typically 1 to 20 mil, depending on the application);
- 2. Adhere well;
- 3. Have high hardness, good corrosion resistance and good wear and abrasion resistance and
- Not cause a fatigue debit in the substrate material because of phenomena such as hydrogen embrittlement or hydrogen reembrittlement.

In the concept evaluation phase described here, some relatively low cost, preliminary screening tests were performed to identify candidates for further study. The results are summarized below.

#### Thickness (Table 4)

Based on the ability to deposit thick coatings, the electrodeposited coatings certainly have potential, but the electroless coatings, in general, are hampered either by a slow deposition rate or an inability to provide the required thickness. Nevertheless, the ENi-P coatings with diamond particles warrant further study to optimize the deposition parameters to obtain thicker coatings, as do some of the ENi-Co-P coatings. With the large WC particles, it was difficult to keep them in suspension because of their mass. As a result, there was some difficulty in obtaining a uniform concentration and distribution within the composite coatings. Further optimization efforts are required with this type of coating, and particles with smaller diameters might help to alleviate this problem.

#### Adhesion (Table 5)

Adhesion does not appear to be a limiting factor with any of the candidate coatings studied, with the exception of the ECo-B coatings. However, the company that applied the coatings experienced some problems with panel preparation and coating application and had to strip and recoat the panels. They felt that this could have contributed to the poor results. In addition, this type of coating may require the use of suitable bath additives to control internal stress. However, this type of coating did not meet all of the other property requirements, so further development work does not appear to be justified.

#### Hardness (Fig. 1)

In general, the nickel-based coatings had no problem in exhibiting the required hardness. By contrast, the Nano-Co and ECo-Bbased coatings were much softer, and the lattice strain that was introduced by the occluded particles was insufficient in improving the hardness of the matrix materials. Company A indicated that heat treating the Nano-Co-based coatings would not significantly improve hardness and even could have a detrimental effect. Contrary to expectations, with the ENi-P coatings, increasing the size of the occluded diamond particles increased the hardness values obtained. However, the volume percentage of the occluded particles was less for the nano-sized particles (~23%) than with the micron-size particles (~35 to 40%). Lattice strain may have contributed to the results. Decreasing the grain size of the cobalt matrix did have the desired effect in making the coatings slightly harder (via the Hall-Petch effect).

#### Wear resistance (Fig. 2)

Generally, the nickel-based coatings exhibited satisfactory wear resistance. The small differences observed in wear resistance may be attributed to non-uniformity of the particle dispersion or the different percentages of occluded particles in the matrices. As expected, the softer, cobalt-based coatings failed this test unless they contained occluded 1000 nm diamond particles.

#### Conclusions

The electroless nickel coatings with occluded diamond particles warrant further development and investigation as an alternative to hard chromium plating. These coatings have the potential to meet the four criteria used in this proof-of-concept study and, as a group, they are the best understood in terms of commercial maturity. The electroless nickel-cobalt coatings did not perform as well in this study, suggesting that there was no benefit in substituting cobalt for some of the nickel. However, this observation needs to be investigated further with optimized coatings.

The electroplated, polycrystalline and nano-structured cobaltbased coatings, although they adhered well, in general failed the other requirement criteria. The electroless cobalt-boron based coatings, like the electroplated cobalt-based coatings, also failed most of the requirements criteria. The electroless cobalt-phosphorus based coatings exhibited mixed results. Although thick coatings were not deposited, when diamond particles were occluded in the coatings they provided satisfactory adhesion, hardness and wear resistance.

Further development work and additional testing are required before any of the candidates evaluated can be considered as being sufficiently robust to replace electroplated hard chromium coatings.

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