Evaluation of Nano-Composite Coatings as Environmentally Acceptable Alternatives to Hard Chromium Plating

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The U.S. Air Force Research Laboratory has been working with Concurrent Technologies Corporation to evaluate alternatives to electroplated hard chromium (EHC) for a variety of applications. One effort focused on performing screening tests on numerous nano-structured coatings or amorphous coatings containing nano- or micro-particles. Electrodeposited nano-crystalline cobalt, with and without tungsten carbide particles, electroless nickel (ENi-P) coatings with various sizes of diamond particles (150, 1,000, 2,000 and 150 + 1,000 nm), and electroless nickel-cobalt-phosphorous (ENi-Co-P), cobalt-phosphorous (ECo-P), and cobalt-boron (ECo-B) coatings - all with and without codeposited diamond particles - were tested and compared to EHC, polycrystalline cobalt, and electroless nickel coatings without occluded particles. The intent was to elucidate the improvements in performance that can be obtained with decreasing grain or particle size.

Preliminary results, suggested that all of the ENiP, ENi-Co-P, and ECo-P processes with occluded diamond particles have the potential to impart the required tribological properties, while reducing the environmental impact of chromium plating processes. To conclude this phase of evaluation, additional studies were performed to obtain thicker (*e.g.*, 5-10 mils) ENi-P coatings with 150 nm diamond particles and 2-mil thick ECo-B and electrodeposited nano-structured nickel-cobalt coatings, both with 150nm diamond particles occluded. This paper discusses the adhesion, thickness compliance, hardness, and abrasive wear resistance results that were obtained during screening tests. Plans for follow-on work in nano-structured coatings and nano-particulate occlusion plating are described briefly.

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BACKGROUND AND OBJECTIVE

As is stated in a previous paper, hexavalent chromium is used extensively to finish surfaces within the Department of Defense (DoD) and private industry due to its properties and decorative appeal (1). Because environmental, health and safety (EHS) issues associated with hexavalent chromium have led to stringent regulations regarding its use, the search for viable alternatives to electroplated hard chromium (EHC) has become a high priority, especially in the DoD. The Air Force Research Laboratory (AFRL) has been active in addressing near- and long-term EHC replacements for both line-of-sight (LOS) and non-line-of-sight (NLOS) applications, respectively.

The Air Force has been working with the Hard Chromium Alternatives Team (HCAT) to address near-term replacements for LOS applications and has initiated several efforts focusing on near-term replacements for NLOS applications. As part of the NLOS effort, the Air Force found that nano-structured coatings or nano-particle composite coatings might offer promise as mid-term replacements for both LOS and NLOS applications. (2) Nano-structures have been shown by many 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 (1, 3-5). Because nano-structured coatings offer the promise to improve the hardness and wear properties of conventional, softer, protective coatings, the AFRL established an effort to investigate the suitability of nano-particle composite plating processes as long-term replacements for EHC (5).

PROOF-OF-CONCEPT STUDY

It was reported earlier, that a Proof-of-Concept study was conducted under the Air Force effort to identify and evaluate commercially available, or near commercial, nano-composite coatings. (2, 6, 7) Additional studies were performed that were based on the findings from this study. This paper provides an update that includes additional testing that has been performed on additional nano-composite coatings.

Additional Coatings Selected

In the earlier report, it was noted that vendor A deposited nano-crystalline cobalt (Nano-Co) electrodeposits both with and without 2,000 nm tungsten carbide (WC) particles incorporated within the nano-crystalline cobalt matrix. Vendor B deposited pseudo-amorphous coatings with micron- and nano-sized particles incorporated into the matrices. Vendor B 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., 2,000 nm). Vendor B deposited electroless nickel, mid-phosphorous (ENi-P) coatings with and without diamond particles (2,000 nm, 1,000 nm, and 150 nm in diameter), and electroless nickel-cobalt-phosphorous (ENi-Co-P), electroless cobalt-boron (ECo-B), and electroless cobalt-phosphorous (ECo-P) coatings, with and without occluded, 1,000 nm diamond particles.

A third vendor, Vendor C, was sought to deposit polycrystalline cobalt (Poly-Co) coatings as baseline specimens against which the Nano-Co coatings could be compared. Data from previous studies were used to provide the EHC benchmark for comparison. Upon completing the initial testing and analyses, which were discussed in the previous paper (2), the team decided to have Vendor B deposit another set of coatings that included ECo-B with 150 nm diamond particles occluded, ENi-P with 150 nm particles occluded (with a target coating thickness between 5-10 mils), and a nano-structured Ni-Co (Nano Ni-Co) coating with 150 nm diamond particles occluded. A summary of all the coating systems that were testing under this study is presented in Table 1.

Table 1. Proof-of-Concept Coatings Evaluated

Category	Baseline/Coating Applied†	Vendors
Baselines	EHC	- -
	ENi-P (mid-phosphorous)	B
	Nano-Co without particles	A
	Poly-Co without particles	C
Nano-structured Matrix and Occluded Micro- and Nano-Particles	Nano-Co with 2,000 nm tungsten carbide particles	A
	Nano Ni-Co with 150 nm diamond particles	B
	ENi-P with 150 (2 mil and 5-10 mil target thickness), 1,000, 2,000 and 150 + 2,000 nm diamond particles	B
	ENi-Co-P with 1,000 nm diamond particles	B
	ECo-P with 1,000 nm diamond particles	B
	ECo-B with 150 and 1,000 nm diamond particles	B

† Vendor B heat treated their coated samples at 350 °C for two hours, with the exception of the Nano Ni-Co coating.

Coating Application

Each vendor prepared nine, flat, 1010 cold-rolled steel (CRS) panels (3 each with dimensions of 4 x 4, 1 x 4, and 1 x 1 inches), and then applied their coatings (see Table 1). The requested target coating thickness was a minimum of 0.002 inch (2 mils), with the exception of the 0.005 to 0.010 inch (5-10 mils) requirement imposed on the second ENi-P coating with 150 nm diamond particles. Vendor B used a heat treatment (350°C for two hours) for all their coated samples, except the Nano Ni-Co deposits, to improve their properties. Companies A and C did not use a heat treatment, but supplied their samples “as plated”.

Coating Testing

All coating testing was performed at Concurrent Technologies Corporation (*CTC*) and is outlined in Table 2. In some instances, the vendors performed some additional characterization of their coatings. These data have been incorporated into the discussion of the results, where appropriate.

Table 2. Evaluation Test Matrix

Test	Test Method	Panel Sizes (inches)	No. of Panels per Test
Metallographic Thickness	ASTM B487	1 x 1	3†
Bend Adhesion	ASTM B571	1 x 4	3
Micro-hardness	ASTM B578	1 x 1	3†
Taber Wear Resistance	ASTM D4060	4 x 4	3

† The same panels were used for both these tests.

Test Results

A summary of the results of testing is presented below, and correlations between composition, structure, and properties have been made, where possible, in the "Summary of Findings" section of this paper. Typical EHC data, per "Federal Specification Chromium Plating Electrodeposited) QQ-C-320B for Class II Engineering Plating," are included in Table 3 so that the alternative coatings may be compared to the currently used coating. A more detailed presentation and discussion of test results is given in Reference (6).

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".

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 inch (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 knife test or bend test.
Hardness	850 Vickers Hardness Number at 100-gram load, 10-15 seconds: measure each specimen at five locations and take the average of results.

Coated samples were mounted, ground, and polished then inspected at a magnification of 100 to 1,000 times, using a metallographic microscope. The cross-sections of each coating were photographed. To support the thickness testing performed at *CTC*, Vendor B used an instrument with commercial software to determine the approximate distributions of particles within the coating cross-sections on their first set of samples.

Table 4 summarizes the thickness data obtained for the various samples. It can be seen that only a few of the processes met or were relatively close to the 2-mil thickness requirement (or 5-10 mil requirement for the case of the thicker ENi-P with 150 nm diamond). In most cases, the deposition processes require additional optimization to control plating rate or increase the plating rate so that thicker coatings can be obtained in reasonable processing times. Most of the ENi-P coatings met or came close to meeting the specified thickness. In addition, the ECo-B, ECo-B (with 150 nm diamond particles), Nano-Co (with 2,000 nm WC particles, set #2) and nano Ni-Co (with 150 nm diamond particles) marginally met the specified value. However, the remaining electrolytic processes and electroless processes, with and without particles, varied significantly from the requested 2-mils coating thickness.

Table 4. Thickness Measurement Test Results

Coating Type	Thickness (inch)
EHC	**(0.0020)**
ENi-P (no diamond)	**0.0020**
ENi-P + 150 nm diamond	**0.0018**
ENi-P + 1,000 nm diamond	**0.0018**
ENi-P + 2,000 nm diamond	**0.0021**
ENi-P + 150 & 2,000 nm diamond	0.0017
ENi-P + 150 nm diamond (5-10 mil thickness)	**0.0075**
Poly-Co	0.0012
Nano-Co	0.0016
Nano-Co + 2,000 nm WC (Sample Set #1) †	0.0014
Nano-Co + 2,000 nm WC (Sample Set #2) †	**0.0027**
ECo-P	0.0007
ECo-P + 1,000 nm diamond	0.0010
ECo-B	**0.0019**
ECo-B + 1,000 nm diamond	**0.0010**
ECo-B + 150 nm diamond	**0.0022**
ENi-Co-P	0.0016
ENi-Co-P + 1,000 nm diamond	0.0017
Nano Ni-Co + 150 nm diamond	**0.0031**

† The first set of samples contained approximately 10% WC by volume; the second set contained approximately 30% by volume.

It should be noted that many of these plating baths were not yet in commercial production and that bath chemistry and operating parameters have yet to be optimized. Consequently, relatively less weight was placed at this time on the thickness data 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.

All coatings passed the adhesion bend test, except two of the ECo-B coatings, with 1,000 nm diamond particles and the ECo-B matrix only. As was stated in the earlier paper (2), it was believed that there was a high level of tensile stress in these coatings due to the extremely fast plating rate, which could result in reduced coating adhesion. Both of these coatings also were deposited on substrates that were previously coated and stripped. Therefore, inconsistencies in pretreatment combined with high stress may have resulted in the reduced level of adhesion.

Table 5. Adhesion Test Results

Coating Type	Adhesion
EHC	**Pass**
ENi-P (no diamond)	**Pass**
ENi-P + 150 nm diamond	**Pass**
ENi-P + 1,000 nm diamond	**Pass**
ENi-P + 2,000 nm diamond	**Pass**
ENi-P + 150 & 2,000 nm diamond	**Pass**
ENi-P + 150 nm diamond (5-10 mil thickness)	**Pass**
Poly-Co	**Pass**
Nano-Co	**Pass**
Nano-Co + 2,000 nm WC (Sample Set #1) †	**Pass**
Nano-Co + 2,000 nm WC (Sample Set #2) †	**Pass**
ECo-P	**Pass**
ECo-P + 1,000 nm diamond	**Pass**
ECo-B	Fail
ECo-B + 1,000 nm diamond	Fail
ECo-B + 150 nm diamond	**Pass**
ENi-Co-P	**Pass**
ENi-Co-P + 1,000 nm diamond	**Pass**
Nano Ni-Co + 150 nm diamond	**Pass**

† The first set of samples contained approximately 10% WC by volume; the second set contained approximately 30% by volume.

It is promising that the second set of ECo-B coatings that were produced, this time with 150 nm diamond particles occluded, displayed no adhesion problems. Vendor B modified the stabilizing agents in this bath, which may have resulted in the improved adhesion (i.e., reduced stress). Otherwise it suggests that the etching and reapplication of the coating was the cause of the poor adhesion displayed in the initial samples.

Micro-hardness 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. Figures 1 and 2 provide the results of the average hardness for each coating type.

As can be seen from Figures 1 and 2, all of the electroless-deposited coatings, both with and without particles, met the EHC specification with the exception of the ECo-B films.

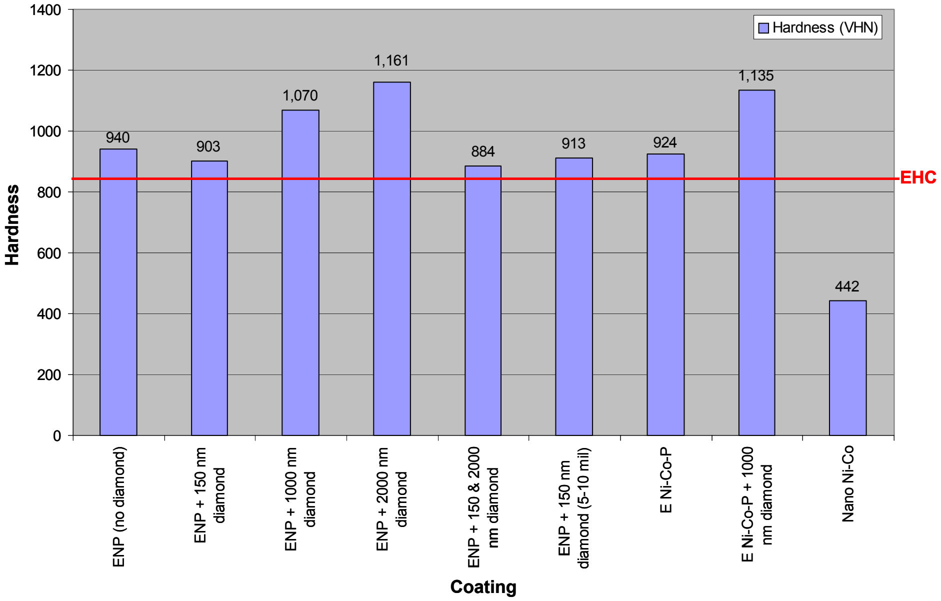

Figure 1. Coating hardness – Set 1

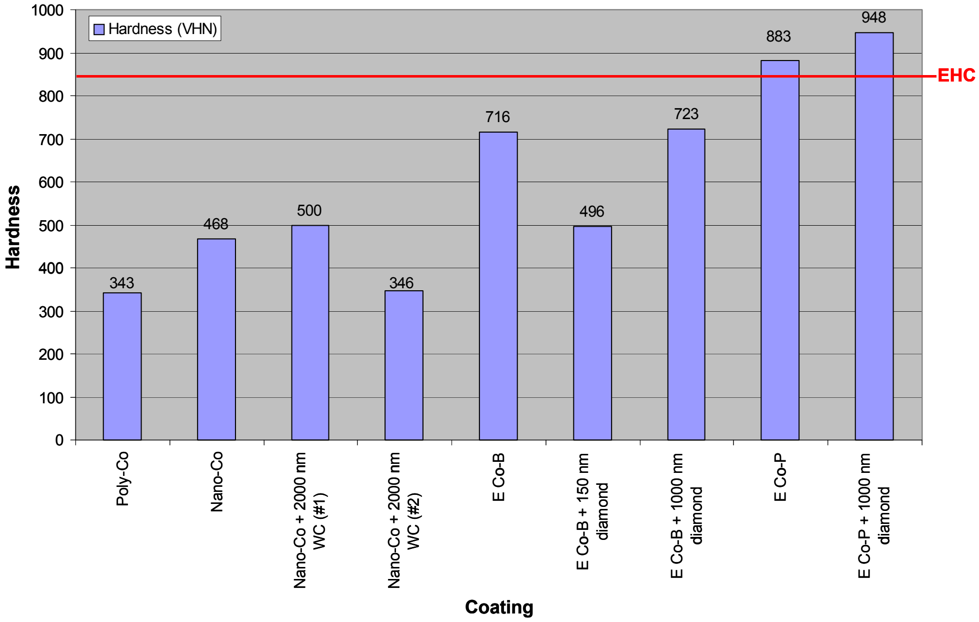

Figure 2. Coating hardness – Set 2
[The first group of Nano-Co samples contained about 10% WC,
and the second group contained about 30% WC by volume]

None of the electrolytically deposited coatings met the specification. However, upon comparing the poly-Co hardness to that of the Nano-Co, it can be seen that there is an increase in hardness with decreasing grain size, which follows the Hall-Petch theory.

As expected, all coatings with incorporated particles, regardless of whether they were WC or diamond, provided further increases in hardness. The only exceptions were the ENi-P and ECo-B coatings, both with occluded 150-nm diamond particles, which experienced decreases in hardness ranging from 37 to 220 VHN units, respectively.

Taber Wear Resistance

Wear testing was performed on the vendor-coated, 4 x 4-inch 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 1,000-gram load were used over 10,000 cycles.

The Taber wear data are presented as a “wear index” in Figures 3 and 4. Lower weight loss (lower Taber Wear Index) indicates a more wear resistant coating material. 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, Figures 3 and 4 also include the index values calculated by subtracting the weight losses in the first 1,000 cycles from the 10,000 cycle total weight loss data.

EHC exhibits a weight loss of between 0.004 and 0.0021 gram over 1,000 cycles, and it was decided to use the lower value for a more rigorous comparison in this evaluation of alternative coatings. The 0.004 gram loss was extrapolated over 10,000 cycles to give an estimated wear loss of approximately 0.04 gram (equivalent to a Taber Wear Index value of 4.0).

From Figures 3 and 4 it is clear that all of the electroless-deposited coatings that contained diamond particles provided superior wear resistance to hard chromium, except the ECo-B with 150 nm diamond particles. The low hardness value of this specimen could be responsible for the poor abrasive wear properties. None of the electrolytically deposited coatings met the most stringent wear requirements
(0.04 g loss).

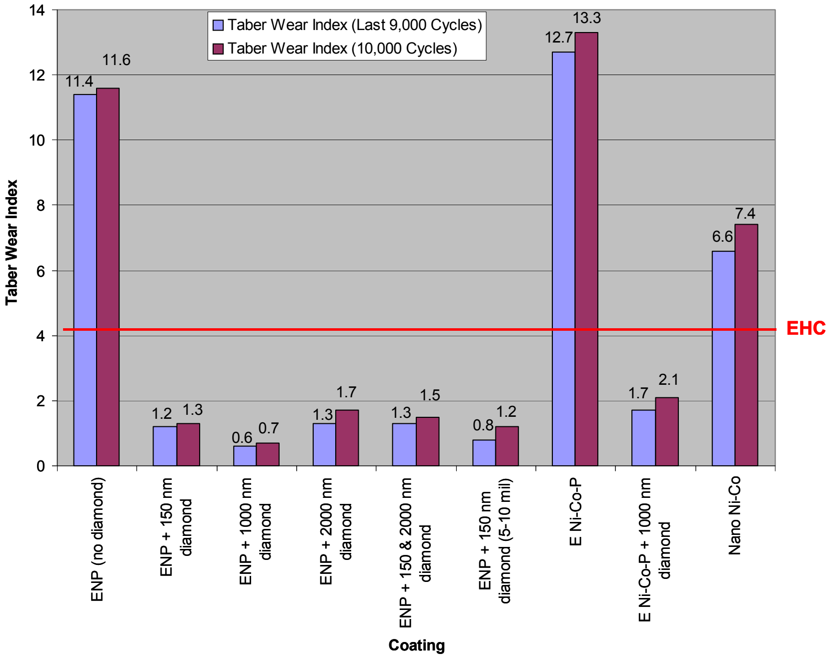

Figure 3. Taber Wear Data – Set #1

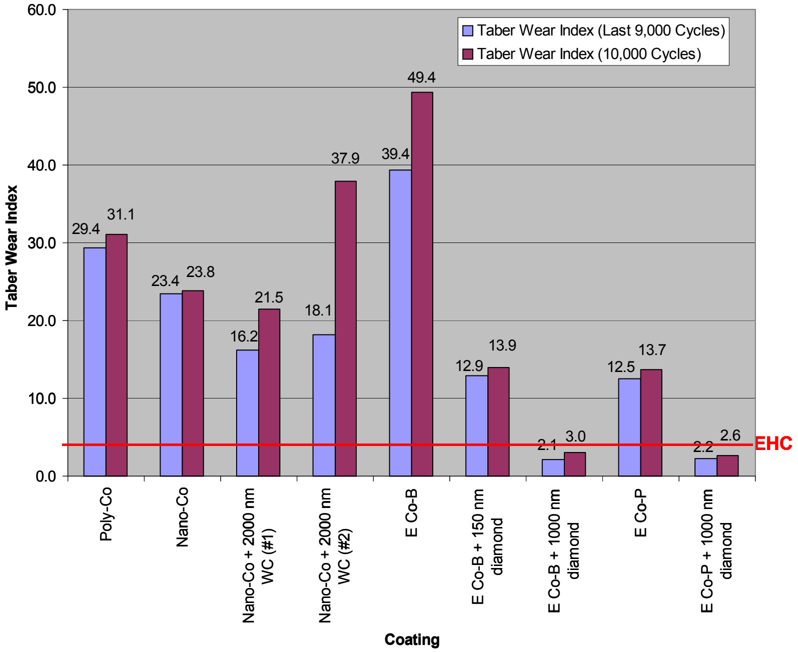

Figure 4. Taber Wear Data – Set #2

[The first group of Nano-Co samples contained about 10% WC, and the second group contained about 30% WC by volume]

Similarly, all of the electroless deposited coatings that contained diamond particles (other than the ECo-B + 150 nm diamond) provided better wear results than the baseline ENi-P coating without particles. However, none of the other electroless-deposited coatings without particles performed better than the ENi-P coating in wear testing. The ECo-P and ENi-Co-P matrices performed similarly to the ENi-P coatings, and therefore, may offer promise as composite coatings when optimized.

It also is evident that the Nano-Co coatings were significantly less wear resistant than the ECo-P coatings, but more wear resistant than the ECo-B coatings. It was expected that both sets of coatings would provide harder and more wear resistant surfaces due to the precipitation of cobalt phosphide and cobalt boride during heat treatment. However, it was not known that the ECo-B coating would be highly stressed in a tensile manner. A high level of tensile stress often evolves from poor coating structure (e.g., open columnar microstructure). The poor coating structure also contributes to lower hardness, as well as reduced wear resistance.

However, this same ECo-B coating with 1,000 nm diamond particles provided superior wear resistance to EHC, and even the coating with 150 nm diamond particles provided superior wear resistance over that of the Nano-Co with WC particles. Figures 5 and 6 show cross-sections of these two types of coatings. It is possible that the very rough, irregular coating of the Nano-Co with WC contributed to a greater weight loss through particles being pulled out or dislodged from the coating surface during the wear test.

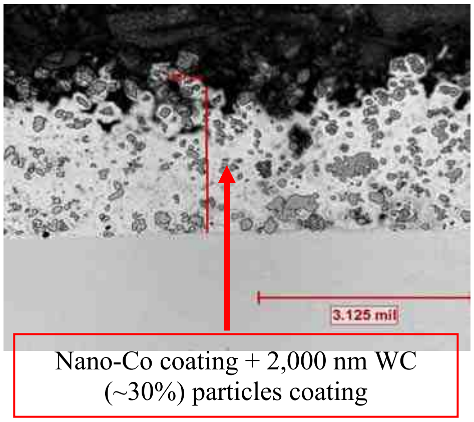

Figure 5. Cross-section of a Nano-Co + WC Coating

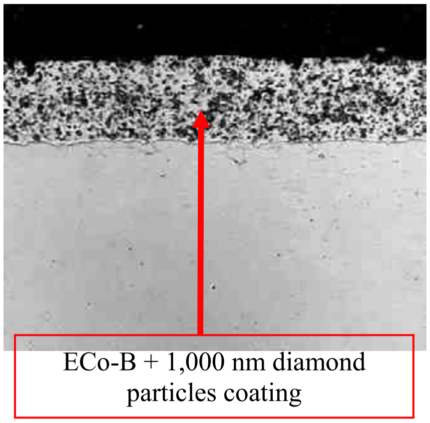

Figure 6. Cross-section of an ECo-B + Diamond Coating

Overall, it is clear that particle incorporation can provide significant improvements in abrasive wear resistance, regardless of the matrix. Variations in improvements are thought to be due to the amount and type of particles that were incorporated and the uniformity of dispersion within the film. However, a detailed metallographic characterization was not performed for all films to confirm this broad claim.

SUMMARY OF FINDINGS

The following table provides a summary of the test results and insight into the conclusions that were made based on test results, literature that was reviewed during this project, and data gathered from the vendors.

Table 6. Summary of Coating Performance Data

Coating Type	**Heat Treated**	**Thickness**	**Adhesion**	**Hardness**	**Wear**
EHC	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 + 1,000 nm diamond*	Yes	Marginal	**Pass**	**Pass**	**Pass**
ENi-P + 2,000 nm diamond*	Yes	**Pass**	**Pass**	**Pass**	**Pass**
ENi-P + 150 + 2,000 nm diamond*	Yes	**Marginal**	**Pass**	**Pass**	**Pass**
*ENi-P + 150 nm diamond (5-10 mils)**	*Yes*	***Pass***	***Pass***	***Pass***	***Pass***
Poly-Co	No	Fail	**Pass**	Fail	Fail
Nano-Co	No	Marginal	**Pass**	Fail	Fail
Nano-Co + 2,000 nm WC (Sample Set #1)†	No	**Pass**	**Pass**	Fail	Fail
Nano-Co + 2,000 nm WC (Sample Set #2)†	No	**Pass**	**Pass**	Fail	Fail
ECo-P*	Yes	Fail	**Pass**	**Pass**	Fail
*ECo-P + 1,000 nm diamond**	*Yes*	*Fail*	***Pass***	***Pass***	***Pass***
ECo-B*	Yes	Marginal	Fail	Marginal	Fail
ECo-B + 1,000 nm diamond*	Yes	Fail	Fail	Marginal	**Pass**
*ECo-B + 150 nm diamond**	*Yes*	***Pass***	***Pass***	*Fail*	*Fail*
ENi-Co-P*	Yes	Marginal	**Pass**	**Pass**	Fail
*ENi-Co-P + 1,000 nm diamond**	*Yes*	*Marginal*	***Pass***	***Pass***	***Pass***
Nano Ni-Co	No	**Pass**	**Pass**	Fail	Fail

† The first set of samples contained about 10% WC, the second set about 30% WC by volume.
* Heat treated at 350 °C for 2 hours.

During this study, a variety of matrices with a range of particle sizes were evaluated, including pseudo-amorphous, nano-structured, and micro/macro-crystalline (poly-crystalline) matrices, all with and without micro- and nano-sized particles. In general, the testing carried out suggested that (1) the micron-sized particles provided better wear properties than the nano-sized particles; (2) the pseudo-amorphous, electroless deposited films provided better properties than the nano-structured films; and (3) the Hall-Petch effect is valid (i.e., nano-crystalline hardness and wear properties showed improvements over the polycrystalline analogs).

Despite the promising results obtained, it is clear that the coatings/processes evaluated are not yet robust enough for Air Force implementation. All of the processes require further development and/or optimization prior to implementing the bath in a military or industrial production setting. This optimization should be accomplished with additional characterization to confirm coating structure, the concentration and dispersion of particles, and the overall coating quality (uniformity, density, etc.). Characterizing particle distribution and coating structure and quality are essential when trying to determine the specific reasons for one coating being more wear resistant than another.

Of the coatings tested, the ENi-P with 1,000 nm diamond particles coating appears to have the most promise in terms of being a short-term replacement for EHC, based on the test results and the bath maturity. The Taber wear results indicated superior performance to EHC. The ENi-P chemistry with other sizes of diamond particle also performed better than EHC, and may only require minimal process optimization to move the process towards implementation. With any of the ENi-P processes, it is suggested that differences in pre- and post-treatments should be studied as well as slight modifications in coating deposition parameters to achieve uniform and thicker coatings (e.g., 10 mils) in a reproducible manner.

Also very promising were the ENi-Co-P, ECo-P, and ECo-B processes - all co-deposited with 1,000 nm diamond particles. Each process performed better than typical EHC coatings. However, each of these coatings was developed by Vendor B using approaches outlined in the literature, and these are not commercially available processes. Therefore, further development is required to ensure process repeatability and the deposition of much thicker coatings that display adequate adhesion and good surface finish.

Lastly, although the Nano Ni-Co with diamond particles did not perform better than EHC, it did show promise in terms of wear resistance, being only slightly less wear resistant than EHC. It is interesting that this coating performed significantly better than the Nano-Co with WC particles, and it might be interesting to deposit the Nano-Co with diamond particles to determine whether it is the incorporation of diamond particles that enhances the performance of the film or the presence of nickel in the matrix.

Following additional characterization and selection of chemistries for further investigation, the incorporation of the 150 nm particles may be re-evaluated to try to elucidate whether particle agglomeration is an issue with the diamond particles as it was with the 2,000 nm WC particles. If particle agglomeration is an issue, but can be reduced to ensure that the particles are evenly dispersed within the film and particle incorporation is maximized, it is thought that the 150 nm diamond may be able to provide greater wear resistance. It is believed that the smaller particles are less likely to be extracted from the coating surface in an abrasive environment, leading to less coating damage. It is theorized that the coating would experience a more gradual wear process.

In addition, the smaller particles, while abrasive, are less likely to cause dramatic three-body wear, such as might be experienced by larger particles that can severely score a coating when subjected to movement under load. However, preliminary characterization work must be conducted to determine particle dispersion and concentration. ENi-P chemistry is considered the most ideal and mature process for such a study.

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