# Non-Line-of-Sight (NLOS) Hard Chromium Alternatives Status Report

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**ABSTRACT**

Since 1998, Concurrent Technologies Corporation (*CTC*) has been working with the U.S. Air Force Research Laboratory (AFRL) to evaluate drop in alternatives to hexavalent chromium electroplating for non-line-of-sight (NLOS) applications. To date, the NLOS effort established hard chromium baseline data at the Air Force Air Logistics Centers (ALCs), identified potential alternatives, completed screening tests, and conducted additional activities to evaluate new processes that have been developed since the initial alternatives were identified. Based on the test data obtained, the NLOS Team selected three, nickel-based coating processes that will be subjected to advanced performance testing. The testing that will be completed includes block-on-ring wear resistance, adhesion, fatigue, corrosion resistance, strippability, and machinability. The alternative coatings that successfully pass these tests will undergo on-site demonstration/validation (dem/val) activities at a selected ALC. Pending dem/val results, at least one process may be pursued for implementation.

In addition to the testing efforts, the NLOS Team also intends to conduct activities to identify and evaluate new processes that have the potential to fulfill long-term Air Force goals of eliminating hazardous materials in general, which includes nickel. To support this initiative, AFRL and *CTC* will conduct market surveys and literature reviews to identify and evaluate new, developmental, and research and development (R&D)-type processes that can replace hard chromium for NLOS applications. To ensure that all possibilities are considered, wet and dry chemistries, commercially available, R&D processes, as well as coatings that are nano-structured or contain nano- particles, will be evaluated.

This paper will provide a status of the NLOS activities that are being performed, present test results that are available at the time the paper is written, and highlight the conclusions that have been made to date.

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**BACKGROUND AND OBJECTIVE**

The U.S. Air Force is currently implementing high velocity oxygen fuel (HVOF) technology in place of some of its electroplated hard chromium (EHC) processes. The potential benefits of HVOF coatings have been investigated for many years; however, one limitation of HVOF is the fact that it is a line-of-sight technology. In other words, HVOF is applicable to simple-shaped parts, but it cannot coat non-line-of-sight (NLOS) geometries, such as blind holes, crevices, and internal diameters. Therefore, even with the implementation of HVOF technology, the Air Force would still need to maintain some EHC baths to accommodate NLOS components. To address this issue, a complementary technology (to HVOF) must be validated and implemented to completely eliminate EHC.

As discussed in earlier papers, the U.S. Air Force Research Laboratory (AFRL) and Concurrent Technologies Corporation (*CTC*) initiated efforts to identify and evaluate EHC alternatives for NLOS applications. The overall project was initially designed as a series of four phases. Phase I is complete, Phases II and III are in progress, and Phase IV is a planned effort[1], pending positive Phase III results. To date, all of the potential EHC alternatives that the NLOS initiative has focused on contain nickel. Nickel is considered better than chromium from environmental and occupational health standpoints, but it is listed on the Environmental Protection Agency's (EPA's) 17 chemicals required for reduction. Recent regulations have not targeted nickel, but future mandates will likely impact its use and disposal. Therefore, the potential alternatives that have been identified and evaluated during the NLOS project are considered to be only short-term solutions. To address this issue, the AFRL established another NLOS task to identify, evaluate, and assess NLOS EHC alternatives that do not contain chromium or nickel. This separate, new task is called "Advanced NLOS (ANLOS)" and was established in August 2003. The ANLOS task will focus on the demonstration/validation (dem/val) and transition of chromium-free and nickel-free coating technologies as alternatives to the ALCs' NLOS hard chromium plating activities. To ensure that all possibilities are considered, wet and dry chemistries, commercially available and R&D processes, and domestic and foreign processes will be investigated during this task, as well as coatings that are nano-structured or contain nano-particles. The primary focus of this task will be on environmentally friendly processes that are viable for ALC production implementation.

Past papers have presented the results of Phase I of the NLOS project and have provided some screening data on Phase II activities. This paper will elaborate on test data that have been obtained and provides a status report of the current work as well as highlight future activities that are related to the subject matter. At present time, Phases II and III of the initial NLOS task and the ANLOS effort are in progress.

[1] Phase IV is currently not funded.

 

## REVIEW OF INITIAL NLOS EFFORTS AND RESULTS

In 1998, the AFRL and *CTC* established the "NLOS Hard Chromium Alternatives" task under the National Defense Center for Environmental Excellence (NDCEE) Program. At that time, AFRL and *CTC* personnel designed an overall plan for the NLOS effort that consisted of four phases, as depicted in Figure 1. The scope of the NLOS project was to identify, demonstrate, validate, and implement alternatives to EHC processes for NLOS applications at the ALCs. At the time this paper was developed, the NLOS initiative had:

- Established the ALCs' EHC performance requirements and additional considerations that an alternative must be able to meet
- Identified potential EHC alternatives for NLOS applications
- Developed of a user community-based decision tool to prioritize viable alternatives
- Subjected six potential NLOS EHC alternatives to Level 1 screening test
- Completed screening level 2A testing for three alternatives
- Established support efforts to evaluate new EHC alternatives that were developed since the start of the NLOS initiative in 1998
- Initiated the completion of Phases II and III and the ANLOS effort.

<u>*ALC Requirements Summary*</u>

*CTC* personnel performed two site surveys at Oklahoma City Air Logistics Center (OC-ALC), Ogden Air Logistics Center (OO-ALC), and Warner Robins Air Logistics Center (WR-ALC) to identify NLOS chromium plated parts, the coating requirements for those parts, and relevant processing methods for each part. Through these investigations, it was determined that 20-40% of the chromium-plated parts are NLOS components. All identified NLOS candidates were catalogued and the processing methods were defined for each component. The individual components ranged in size, geometry, and substrate composition.

In addition to obtaining NLOS part information, current ALC processing methods and part characteristics were identified. In general, it was found that most current rework processes follow similar sequences, and that part characteristics were a combination of the requirements identified in the Federal Specification for Chromium (Electrodeposited) (QQ-C-320B) and the functional, production, and environmental needs and concerns, as identified by ALC production engineers and equipment operators.

Federal Specification QQ-C-320B is referenced by most process instructions for the components identified at the ALCs. Therefore, the criteria outlined in this specification were used to evaluate each of the alternative processes. Other characteristics and considerations that were deemed to be important by ALC personnel and the NLOS team, included anti-galling characteristics, removal and processing times, coating properties (i.e., quality, rms finishes), hazardous/toxic nature of the coating, processing and capital costs related to the coating, hydrogen embrittlement elimination, and compatibility with existing plating equipment.

 

Additional requirements identified by the NLOS team included 1) the ability to remove or grind the coating, 2) the reproducibility of the process, and 3) property data such as wear and corrosion resistance, coefficient of friction, and fatigue life of the coating. The baseline data used for comparison is listed in Table 1.

**Table 1. Performance Characteristics**

| Characteristic | Value |
|---|---|
| Microhardness (per ASTM B-578) | 950-1050 VHN<br>68-74 RCH<br>900 KHN [1] |
| Corrosion Resistance (per ASTM B117) | 24 hours (per ASTM B-117 salt spray testing) [2] |
| Wear resistance (per ASTM D 4060) | 0.004 g loss/1,000 cycles [1] |
| Coefficient of Friction | 0.5 [2] |

*Identification of Alternatives and Process Selection Summary*

Prior to the start of the identification effort, four primary requirements were identified that each alternative process must meet to be considered for ALC use. They were:

1. The alternative must be readily available and easily implemented at the ALCs.
2. The alternative must adhere to steel substrates.
3. The alternative cannot contain any form of chromium (e.g. trivalent chromium).
4. The alternative must be able to plate to a thickness of two mils or greater.

Any alternative that did not meet any one of these four requirements was eliminated from further consideration.

A market survey, including a literature search and discussion with vendors and researchers, was conducted to identify available alternatives to hard chromium plating. Vendors were contacted to obtain information related to their respective processes. Information also was obtained through material safety data sheets, technical data sheets, and a survey requesting specific process and product data. In addition, articles that focused on hard chromium alternatives were reviewed for their applicability to NLOS issues. Those articles that offered pertinent information were summarized and efforts to retrieve additional information from the authors were made.

After reviewing the potential alternatives, the alternative processes were separated into three categories: (1) commercially available alternatives, (2) alternatives approaching commercialization, but require some development, and (3) alternatives in the research phase. Alternatives were categorized based on information obtained from the vendor/researcher of the process/coating. Information was gathered about each process/coating, the physical and

[2] The corrosion resistance of hard chromium, with an underlayer of nickel, is 96 hours, per QQ-C- 320B.

mechanical properties of the coating, the environmental impacts of the process, ability of the coating to be reworked, the ability of the coating to restore dimensions, and the process limitations and/or advantages. Capital and operating cost information also was gathered when available.

The findings of the alternatives search were assembled into the Identification of Alternatives Report. It discussed each alternative process and provided comparisons of each alternative to that of hexavalent chromium plating. To evaluate and compare the alternatives, a matrix of the characteristics of the alternatives was developed. The matrix highlighted data voids. Where no data were present for a particular alternative, the vendor(s) were contacted again to attempt obtain the missing data. The matrix provided a searchable database of the alternatives and their engineering characteristics.

Based on the remaining information in the matrix, a tool was developed to analyze the remaining alternatives and determine the most viable alternative(s) for this task. The engineering data were given ratings of 3, 2, or 1, where 3 equated to "exceeds requirements," 2 equated to "meets requirements," and 1 equated to "does not meet requirements." For example, an alternative that displayed a hardness value that was lower than hard chromium would receive a rating of 1.

The characteristics being evaluated were then weighted to reflect the importance of each criterion; i.e., a multiplier was assigned according to the importance of the criterion. The importance of each criterion was established by input, quantified by surveys, from key personnel involved with this project, which included members of the HCAT and the PEWG, and representatives from the ALCs, OEMs, and AFRL.

A final score for each alternative was determined by multiplying the rating of each characteristic by the ranking of that characteristic. The alternative processes were then ranked from high to low. The findings were submitted to AFRL to select those processes that will be evaluated during demonstration activities.

The coatings identified were primarily nickel-based processes, and specifically electroless nickel (EN) coatings. The majority of the non-nickel alternatives that were identified are in the research and development stage. The nickel-based alternatives included conventional, electroless nickel phosphorous and electroless nickel boron coatings as well as composite and alloy coatings. Many of the composite or alloy coatings involved the codeposition of polytetrafluorethylene (PTFE), tungsten, silicon, silicon carbide, diamond, boron nitride, inorganic powders, boron and phosphorous, $CF_x$ (a product formed by reacting coke with fluorine), and combinations thereof. In addition, two commercially available non-nickel alternatives were identified, which included a polymer-based product and a cobalt-tungsten alloy.

The NLOS team met in January 2000 to select the technology (ies) that would be evaluated in Phases II and III of the NLOS initiative. The Air Force set minimum requirements that each alternative needed to demonstrate to be considered a viable alternative for NLOS applications. The technologies that were selected for Phase II evaluation include two (2) electroless nickel phosphorous processes, one (1) electroless nickel boron process, one (1) electrolytic nickel-tungsten technology, one (1) electroless nickel silicon carbide composite, one (1) electroless nickel composite diamond coating, and one (1) nickel-based nanoparticle electrodeposition process.

*Level 1 Screening Tests Results Summary*

Beginning in late 2000, screening test activities commenced for the NLOS project being completed under the NDCEE Program. The activities were initiated with the confirmation of vendor processing efforts. All of the vendors, excluding one, of the abovementioned processes agreed to deposit their coatings on test specimens in support of this effort. The electrolytic nickel-tungsten process was not deposited by the vendor, but rather by an original equipment manufacturer (OEM). The vendors were asked to plate one-half of the samples to a thickness of 2 mils and the other half to a thickness of 20 mils, and each group was exposed to three heat treat scenarios. Scenario one involved no heat treatment, scenario two involved a heat treatment of 375°F for 24 hours[3], and scenario three involved the vendor's recommended optimum heat treatment to achieve the desired properties.

*CTC* personnel attended each vendor processing visit to observe the process as well as record optimization tactics. The vendor visits occurred between February and June 2001. As the samples were coated, they were returned to *CTC* for independent testing and analysis. *CTC* completed all of the Level 1 Screening tests for the vendor-coated samples, which included evaluations of adhesion, hardness, composition, thickness, quality, stress, and profile. A summary of the results obtained from Level One testing is located in the following table (P = complete pass; F = complete fail; M = mixed results). For a complete copy of the results obtained during Level One, please see the NLOS "Level One Screening Tests Results Report" (dated February 21, 2002) document, as well as the "Supplemental Information to the Level One Screening Tests Results Report" (dated May 6, 2002) document.

[3] This heat treatment was per the federal specification QQ-C-320B.

**Table 2. Level One Screening Test Results Summary**

| Coating | Requested Thickness Values | Adhesion | Hardness | Composition | Thickness | Quality | Stress [1] | Profile |
|---|---|---|---|---|---|---|---|---|
| EN-high P | 2 mil | M | M | P | M | M | T | F |
| | 20 mil | F | M | P | M | M | | M |
| EN-mid P-SiC | 2 mil | M | F | P | M | P | C | P |
| | 20 mil | P | F | P | M | P | | P |
| EN-low P | 2 mil | M | F | P | M | M | C | F |
| | 20 mil | P | F | P | M | M | | F |
| EN-mid P-BN | 2 mil | P | F | P | P | M | T | F |
| | 20 mil | F | F | P | P | M | | F |
| Electrolytic nano-composites | 2 mil | F | F | M | P | M | C | F |
| | 20 mil | P | F | M | P | M | | F |
| Electrolytic NiW[2] | 2 mil | P | P | P | F | P | T | F |
| | 20 mil | P | P | P | F | P | | F |

EN-high P stands for electroless nickel-high phosphorous.
EN-low P stands for electroless nickel-low phosphorous.
EN-mid P-BN stands for electroless nickel-mid phosphorous with boron nitride particles.
NiW stands for nickel-tungsten.
EN-mid P-SiC stands for electroless nickel-mid phosphorous with silicon carbide particles.
[1] T = tensile; C = compressive.
[2] Panels processed by OEM.

At the conclusion of testing, vendors was provided with a Test Report for their respective process. *CTC* personnel contacted each of the vendors to obtain input and feedback related to the performance of their process. Based on the test results and vendor feedback, AFRL and *CTC* personnel selected four of the processes to proceed to Level 2A Screening.

*Level 2A Screening Tests Results Summary*

The processes that were subjected to Level 2A Screening efforts included the EN-high P, EN-mid P-SiC, EN-low P, and electrolytic NiW coatings. Again, the vendors of these processes were asked to deposit their coatings such that the best coating would be evaluated. Level 2A Screening tests included quality inspections and Taber wear resistance analysis.

Only three of the four selected processes completed Level 2A tests. The OEM that had earlier processed the electrolytic NiW coating could not fit the request into their production schedule in time for Level 2A tests; therefore, the process was not evaluated for Taber wear resistance. Taber wear resistance was selected as the wear test that would be completed for the NLOS initiative based on ALC input. Per ALC personnel, Taber wear was stated as the most generic type of test to run. Pending AFRL approval, Taber wear resistance was performed by *CTC* in accordance with modified ASTM D4060, Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser. The plated specimen was subjected to wear using a CS-10 wheel under a 1,000-gram load for 10,000 cycles. The specimen was initially weighed and then tested in 1,000 increment cycles, with the weight of the specimen being recorded after each cycle. Upon completion of the 10,000 cycles, a final specimen weight is obtained. From this data, wear index values (1,000 times the loss in weight in milligrams per cycle) was calculated.

Table 2 (located on the following page) provides the results of the Taber testing for the three selected EHC alternatives. At the end of Taber testing, the NLOS effort under the NDCEE Program concluded.

*Support Efforts*

Following the completion of the NDCEE NLOS task, the AFRL and *CTC* reviewed the types of technologies that were investigated during the effort. It was found that while viable processes were evaluated, other technology advancements had been made and were continuing to evolve as the task progressed. In an effort to ensure that the NLOS task was considering and evaluating suitable processes, the Air Force and *CTC* initiated a series of low-level evaluations of some newly developed processes. These evaluations were conducted in the same manner as the NLOS effort, to allow for direct comparisons. Additionally, AFRL and *CTC* solicited ALC personnel feedback to determine what types of processes they would be interested in evaluating (recall, the ALCs constitute the user community that ultimately must support and commit to implementation).

The processes that the AFRL and *CTC* evaluated under the support efforts included one electroless nickel-boron (ENB), two electrolytic nickel-cobalt (EN-Co), a nickel alloy, and two nickel composite (diamond and silicon carbide, respectively) coatings. These processes were (and in some cases are) subjected to the same NLOS testing that was completed during Screening Levels 1 and 2A such that all of the potential NLOS EHC alternatives are comparable and have the proper justification to warrant their inclusion in future NLOS activities.

 

**NLOS PHASES II AND III**

This task will complete the initial NLOS work established and finalize the remaining screening tests and conduct validation evaluations of up to three potential alternatives, as selected by the Air Force. The three processes that will be evaluated during this task may be any of the processes previously tested and/or recommended by the ALCs (pending AFRL concurrence).

This task will be a joint effort between the Air Force and *CTC*. The two organizations will share task responsibilities and work together to accomplish this task's goals. The testing that will be

 

| Coating | Average Taber Results | | | | | | |
|---|---|---|---|---|---|---|---|
| | No. of Cycles | No Heat Treatment | | Standard Heat Treatment | | Optimum Heat Treatment | |
| | | **Average Cumulative Weight Loss (mg)** | **Average Wear Index** (for last 9,000 cycles) | **Average Cumulative Weight Loss (mg)** | **Average Wear Index** (for last 9,000 cycles) | **Average Cumulative Weight Loss (mg)** | **Average Wear Index** (for last 9,000 cycles) |
| EN-high P | 2,000 | 68.1 | 33.4 | 67.3 | 32.8 | 33.1 | 18.2 |
| | 3,000 | 100.8 | | 100.3 | | 51.7 | |
| | 4,000 | 134.6 | | 132.7 | | 70.2 | |
| | 5,000 | 168.1 | | 165.5 | | 88.7 | |
| | 10,000 | 335.1 | | 329.0 | | 178.5 | |
| EN-mid P-SiC | 2,000 | 15.2 | 3.0 | 15.5 | 2.7 | 13.5 | 2.0 |
| | 3,000 | 18.9 | | 19.2 | | 16.1 | |
| | 4,000 | 21.8 | | 22.1 | | 18.4 | |
| | 5,000 | 24.9 | | 25.1 | | 20.3 | |
| | 10,000 | 37.6 | | 36.0 | | 27.8 | |
| EN-low P | 2,000 | 48.5 | 23.0 | 50.7 | 23.8 | 57.5 | 22.3 |
| | 3,000 | 74.0 | | 74.5 | | 80.3 | |
| | 4,000 | 97.3 | | 98.6 | | 103.2 | |
| | 5,000 | 120.1 | | 122.0 | | 132.6 | |
| | 10,000 | 232.3 | | 240.6 | | 226.0 | |

conducted during Phase II includes advanced adhesion, block-on-ring wear resistance (in accordance with ASTM G77-98), neutral salt fog exposure, electrochemical analyses of corrosion potentials, grindability, strippability, fatigue, hydrogen embrittlement, and quality inspections. Similar to the initial NLOS effort, the vendors of the selected processes will be requested to deposit their coatings such that the "best" coating is evaluated. Pending positive results, the AFRL and *CTC* will complete ALC on-site dem/val activities for up to two processes. The on-site demonstration will be performed by ALC operators in the presence of AFRL, additional Air Force (as appropriate), *CTC*, and vendor personnel. The ALC operators will coat a specified number of test specimens with the process. As part of the demonstration, transferability of the process as well as repeatability aspects will be evaluated. The ALC-coated specimens will then be taken back to AFRL and *CTC* facilities for testing and analysis. The testing that will be completed includes tests that were conducted during Phase II, screening efforts. The dem/val test results will be compared to those obtained during Phase II, screening efforts as yet another measure of process transfer ease. If the vendor-applied coatings exhibited far superior performance than those processed by the ALCs, technology transfer will need to be reviewed and fully assessed if a process is deemed viable.

At the conclusion of Phase II, Air Force and *CTC* personnel will meet to review the results and determine the future NLOS work, which may include implementation planning, qualification testing, and/or technology transfer efforts.

**ADVANCED NLOS WORK**

As earlier noted, all of the commercially available processes that were identified during the NDCEE NLOS task were wet chemistry alternatives that contained nickel. The types of commercially available alternatives that were identified included variations of electroless nickel products (low, mid, and high phosphorus contents and composite coatings), electrolytic alternatives (such as nickel-tungsten), and nanocomposite coatings. Because nickel has been identified as a hazardous material, implementation of any of these alternatives would be only an intermediate solution, from an environmental point of view. Therefore, other solutions that can fulfill long-term goals of eliminating hazardous materials need to be identified and subjected to screening and dem/val testing. To support this initiative, *CTC* will work with the AFRL to identify and evaluate newly available, developmental, and R&D-type processes that can replace hard chromium for NLOS applications. For this effort, the Air Force's plating requirements that were identified during the original NLOS project will be applicable, and an understanding of this information will be necessary for proper execution of this new task.

Similar to the other NLOS efforts, the ANLOS task will be completed through a series of sequential phases, which are briefly outlined below. At the completion of each Phase, AFRL and *CTC* will review the progress to determine if the following phase is warranted. A brief description of the planned approach is outlined below.

**Phase I**

- Identify candidate processes and materials
- Select the most viable processes based upon vendor data, proof-of-concept evaluations, and laboratory screening testing

**Phase II**

- Conduct extensive performance testing of the best candidate(s)
  - ➢ Establish coating engineering properties
  - ➢ Identify processing capabilities and any limitations

**Phase III**

- Demonstrate the ability of the best process(es) to provide a suitable coating on representative aircraft components
- Develop scale-up requirements based on ALC needs
- Complete optimization efforts to facilitate the transition of the selected technology into ALC operations

The ANLOS task also will be a joint effort between the Air Force and *CTC*. The two organizations will share task responsibilities and work together to accomplish this task's goals.

**SUMMARY**

The NLOS initiative was established in 1998 as a collaborative effort between the AFRL and *CTC*. The project's goals were to establish ALC EHC requirements and needs, identify potential alternatives applicable for NLOS applications, and evaluate the viability of the alternatives through screening tests, ALC demonstrations, and dem/val testing activities. The initial NLOS project was set-up as a series of iterative phases, of which the following phase was only conducted pending positive results from the previous phase.

At the time this paper was developed, Phase I was successfully completed and Phase II efforts were in progress. Additionally, the Air Force and *CTC* established two types of complimentary efforts to support the NLOS initiative. The first effort was one that allowed the AFRL and *CTC* to evaluate potential NLOS EHC alternatives that were developed after the initial NLOS' alternatives search was completed in 1999. The second task (ANLOS) was designed to identify emerging and innovative processes that have the ability to offer the Air Force long-term solutions to eliminating EHC.

 