

A Comprehensive Test of the Effect of a Fume Suppressant Used in Hard Chromium Electroplating on Emissions Quality, Worker Exposure, and Product Quality

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Uncontrolled atmospheric emission tests were conducted on hard chromium electroplating baths, while a fume suppressant was in use. Tests were also conducted without using fume suppressant. During all tests, industrial hygiene samples were also taken to measure the effect of the fume suppressant on occupational exposure. Additionally, parts electroplated during the use of the fume suppressant, and without suppressant, were tested for six electroplating quality parameters.

A 20- to 70-fold reduction in chromium atmospheric emissions was achieved with fume suppressant. It was also demonstrated that occupational exposure to hexavalent chromium was reduced with fume suppressant use. Further, no significant effects from suppressant use were noted on the physical quality of the electroplated parts.

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INTRODUCTION

This project was funded by the Department of Defense's (DoD's) Environmental Security Technology Certification Program (ESTCP). DoD and numerous commercial electroplaters perform hard chromium electroplating to produce hard surface finishes, typically for use as bearing surfaces. Hard chromium electroplating is conducted using a bath containing warm (about 130°F) chromic acid. The electroplating activity causes bubbling in the bath. As the bubbles break the surface of the bath they emit particles of chromic acid mist. Chromic acid contains chromium in its more toxic "hexavalent" form. Because of the toxicity of hexavalent chromium, these baths are required by the Occupational Safety and Health Administration (OSHA) to be ventilated through an exhaust system. Such exhaust systems remove almost all the chromium from the workplace. However, the exhaust system deposits the chromium in the ambient environment surrounding the plating facility. Because of the toxicity of hexavalent chromium, the U.S. Environmental Protection Agency (EPA) limits the amount of chromium that may be emitted to the environment (See Table 1 for emission standards under 40 CFR 63, Subpart N).

	Emission Limitations					
Type of Bath	Small Facility (<60 million amp- hrs/yr)	Large Facility				
All existing baths	0.03 milligrams/dry standard cubic meter (mg/dscm)	0.015 mg/dscm (6.6 x 10 ⁻⁶ gr/dscf)				
	(1.3 x 10 ⁻⁵ grains/dry standard cubic foot) (gr/dscf)					
All new baths	0.015 mg/dscm	0.015 mg/dscm				
	$(6.6 \text{ x } 10^{-6} \text{ gr/dscf})$	(6.6 x 10 ⁻⁶ gr/dscf)				
Decore	Decorative Chromium Plating Baths Using Chromic Acid					
All new and existing baths	0.01 mg/dscm (4.4 x 10	⁻⁶ gr/dscf)				
	Surface Tension of <45 dynes/centimeter (3.1 x 10^{-3} pounds/foot (lbf/ft)					
Chromium Anodizing Baths						
All new and existing baths	$0.01 \text{ mg/dscm} (4.4 \text{ x } 10^{-6} \text{ gr/dscf})$					
	Surface Tension of <45 dynes/centimeter $(3.1 \times 10^{-3} \text{ lb}_{\text{f}}/\text{ft})$					

To comply with these limits, hard chromium electroplating emissions are usually passed through an air pollution control device (APCD), typically a wet scrubber/mist eliminator, to remove the chromium mist from the exhaust air stream. The chromium thus removed becomes part of a wastewater stream (wastewater is also regulated, but is not the subject of

this investigation). However, it is noted from Table 1 that *decorative* chromium electroplaters have the option of either meeting a stack emissions standard (0.01 mg/dscm [4.4 x 10^{-6} gr/dscf]) or complying with a bath surface tension requirement (less than 45 dynes/centimeter [3.1 x 10^{-3} pounds/foot]). Currently, *hard* chromium electroplaters do not have the option of meeting a surface tension standard.

The primary objective of this investigation was to test the effectiveness of a fume suppressant (FS)^{*} in minimizing the evolution of hexavalent chromium mist from the surface of a hard chromium electroplating bath. Ideally, this investigation would support allowing hard chromium electroplating operations to comply with a surface tension standard in the same way that decorative chromium electroplaters have this option available.

FS reduces the surface tension of the liquid in the bath. When the surface tension is reduced, gases escape at the surface of the solution with a diminished "bursting" effect, causing less mist formation (*i.e.*, smaller bubble size, less surface impact). Adding FS to the electroplating bath at a concentration of only 0.25 percent significantly reduces the surface tension. If chromium emissions can be reduced significantly using FS, there will also be a savings in the amount of chromic acid purchased, because less acid will escape the bath as a mist. The FS additive tested in this investigation is a perfluorinated material that is relatively soluble in water and produces very little foam. Active ingredients include organic fluorosulfonate and tetraethylammonium-perfluorocytyl sulfonate.

A further objective of this project was to quantitatively evaluate the effect that the FS has on the substrates being plated and on the chromium electroplate coating.

PROJECT DESIGN

The following sections discuss the project's specific performance objectives, the setup of the electroplating operation and the sampling equipment, and the sampling/monitoring procedures.

Performance Objectives

As noted above, the primary project objective was to provide data to support inclusion of FS addition as an alternative to an emission concentration standard for hard chromium plating. The intent was to show that if the FS kept the surface tension at or below about 30 dynes/cm $(2.1 \times 10^{-3} \text{ pounds/foot})$, atmospheric emissions from the hard chromium electroplating bath would remain below the most stringent hexavalent chromium regulatory limit of 0.015 milligrams per dry standard cubic meter (mg/dscm) [6.6 x 10⁻⁶ grains per dry standard cubic foot (gr/dscf)]. Consequently, FS additives may be an effective alternative to mechanical APCDs.

^{*} Fumetrol[®] 140, manufactured by Atotech USA, Inc., Rock Hill, South Carolina

A second objective was to demonstrate that there was a significant reduction in fugitive emissions from the bath (*i.e.*, those emissions that escape the bath's exhaust system, and enter the shop environment). Fugitive emissions increase the occupational exposures of workers in the shop. The permissible exposure level (PEL) to hexavalent chromium is currently 0.052 mg/m³ (as chromium) [2.3×10^{-5} grains/cubic foot (gr/ft³)], and compliance through ventilation is generally not difficult, but OSHA is considering reducing the PEL to as little as 0.0005 mg/m³ (2.2×10^{-7} gr/ft³).

A third objective was to certify that the FS does not negatively affect the integrity of the electroplating process, the hard chromium coating, or the functional properties of the plated components. Critical properties include: hydrogen embrittlement, hardness, porosity, adhesion, thickness, and fatigue. Hard chromium is plated on critical components at DoD facilities. Successful evaluation required that materials electroplated in hard chromium baths treated with FS performed as well as materials treated in baths without FS.

Physical Setup and Operation

A schematic of a typical hard chromium electroplating operation, including the emission control device is shown in Figure 1. The use of FS to control emissions of hexavalent chromium is quite simple. It consists of adding approximately 0.25 percent by volume of the FS to the bath, and allowing the bath contents to reach equilibrium (a few hours). Addition of FS effectively lowers the surface tension of the bath from above 70 dynes/centimeter (as measured by a De Nouy Ring Tensiometer) to below 30 dynes/cm. Additional FS is added over time as required to maintain the surface tension below 30 dynes/cm. There is essentially no capital equipment involved with the addition of FS to the electroplating process.

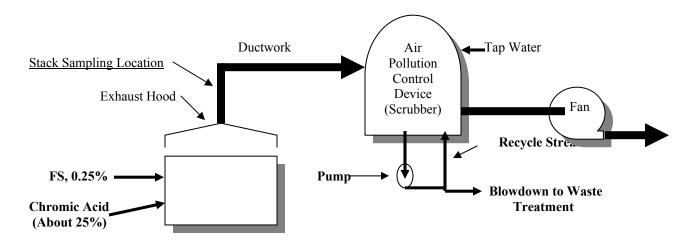


Figure 1 – Hard Chromium Electroplating Process Schematic

Sampling/Monitoring/Analytical Procedures

Testing was conducted at either the Oklahoma City Air Logistics Center at Tinker Air Force Base, Oklahoma City, Oklahoma, (Tinker) or at the Naval Aviation Depot, Cherry Point, North Carolina (Cherry Point). One day of testing was conducted at Tinker without FS (*i.e.*, baseline sampling), and five tests with FS. At Cherry Point, two days of testing were conducted without FS, and five with FS.

Stack emissions testing (*i.e.*, stack tests in the ductwork between plating bath and the APCDs) at each site was conducted using EPA Method 306, *Determination of Chromium Emissions from Decorative and Hard Chromium Electroplating and Anodizing Operations*. This is the conventional test protocol for total and hexavalent chromium analysis for point source air emissions. Each emissions test sample was taken during a two-hour period, using "isokinetic" sampling techniques mandated by Method 306. Three emissions tests were conducted during each sampling day. The results for each sampling day were calculated by averaging the data from each of the three tests taken that day. Samples from each of the three tests conducted during each sampling day were analyzed for total and hexavalent chromium.

Occupational health area sampling was conducted using OSHA Method 215 *Hexavalent Chromium in Workplace Atmospheres*. During each test day samples were taken in three locations: a few inches above the surface of the baths, in the breathing zone directly in front of the baths, and in the breathing zone a few feet in either direction from the baths. At each

of the sampling locations, two to four samples were taken during each sampling day. All samples were analyzed for hexavalent chromium.

Material quality testing, except for fatigue testing, complied with SAE Aerospace Material Specification (AMS) QQ-C-320B, *Chromium Plating Electrodeposited*. The standard test includes appropriate ASTM Methods. Limited equivalence fatigue testing was based on Navy requirements.

PERFORMANCE ASSESSMENT

There are three types of performance data that were developed in conjunction with this study:

- Stack emissions data: These are measurements of atmospheric emissions of chromic acid mist from the electroplating bath that are captured by the ductwork leading to the bath's air pollution control device (APCD).
- Industrial hygiene data: These are measurements of the concentration of chromium taken in the shop environment directly over the plating bath, as well as in the breathing zone in front of, and a few feet away from the bath.
- Plated parts quality data: To determine if the use of FS had any effect on the quality of the plated part. Parts that were plated with and without the use of FS were subject to six plating quality tests: fatigue, hydrogen embrittlement, hardness, porosity, adhesion, and thickness.

The summarized results of these performance data are described in the following sections.

Atmospheric Emissions Data

With respect to stack emissions (*i.e.*, samples taken in the bath exhaust ductwork), the use of FS reduced the concentration of chromium in the exhaust gases by between 20- fold and 70fold compared to operations without the FS. Typically, the exhaust emissions readily complied with EPA regulatory standards for existing, *small* (less than 60 million amperehours per year) hard chromium electroplating shops. However, it does not appear that the reduction in emissions is enough to comply with current regulatory emission standards for new hard chromium electroplating baths or existing baths for *large* shops.

A review of the summary data in Table 2 and Figures 2 and 3, confirms that FS causes a dramatic decrease in the concentration of total and hexavalent chromium from stack emissions to the atmosphere (or to an APCD). At Cherry Point the average reduction in concentration of total chromium was about 70-fold, and at Tinker it was about 20-fold.

As noted above though, when comparing the emissions data to the current EPA National Emissions Standards for Hazardous Air Pollutants (NESHAP) standard of 0.015 mg/dscm (6.6 x 10^{-6} gr/dscf), the Cherry Point average data with FS for total chromium was 0.0348

mg/dscm (15 x 10^{-6} gr/dscf), and the Tinker average data with FS for total chromium was 0.0245 mg/dscm (11 x 10^{-6} gr/dscf). Both would be out of compliance if they did not have APCDs installed downstream of the sampling points.

Data are also presented in graphs in the upper right-hand corners of Figures 2 and 3 for the emissions as a function of the electroplating load (*i.e.*, mg of chromium per ampere-hours (mg/amp-hr). The results are also dramatic with respect to the reduction in emissions with FS as compared to without FS. (At Tinker there was some question about whether the amp-hr meters were providing the correct readings. Therefore, some of the ampere-hours emissions data for Tinker may be incorrect.)

Sampling	Surf. Tension	Hexavalent Chromium				Total Chromium			
Date	(dynes/cm)	Sample # 1	Sample # 2	Sample # 3	Average	Sample # 1	Sample # 2	Sample # 3	Average
7/11/00	72	n/a	6.32	0.737	3.529	n/a	6.804	0.853	3.829
7/12/00	72	3.13	0.912	1.37	1.804	4.06	0.919	1.56	2.180
9/21/00	33	0.0418	0.0299	0.0216	0.0311	0.0482	0.0367	0.0237	0.0362
11/15/00	76	1.49	1.30	1.26	1.35	1.57	1.31	1.21	1.36
11/16/00	23.1	0.0446	0.0482	0.0678	0.0535	0.0431	0.0473	0.0678	0.0527
12/13/00	23.4	0.0170	0.0273	0.0233	0.0225	0.0193	0.0289	0.0243	0.0242
3/27/01	27	0.0313	0.0533	0.0276	0.0374	0.0356	0.0539	0.0349	0.0415
4/17/01	27	0.0215	0.0153	0.0204	0.0191	0.0218	0.0163	0.0209	0.0197

CHERRY POINT

Average Without FS:	2.228	2.457
Average with FS:	0.0327	0.0348

NOTE: n/a indicates that no parts were being electroplated during test number 1 on 11 July 00

Sampling	Surf. Tension	Hexavalent Chromium				Total Chromium			
Date	(dynes/cm)	Sample # 1	Sample # 2	Sample # 3	Average	Sample # 1	Sample # 2	Sample # 3	Average
9/12/00	72	0.516	0.286	0.347	0.3833	0.645	0.333	0.443	0.474
10/11/00	34	0.00818	0.0104	0.00624	0.0083	0.00890	0.0125	0.0111	0.0108
11/8/00	27	0.00870	0.00715	0.00295	0.00627	0.00896	0.00642	0.00299	0.00612
12/6/00	30.5	0.0234	0.0186	0.0106	0.0175	0.0240	0.0215	0.0125	0.0193
7/31/01	27.5	0.106	0.0204	0.0337	0.0534	0.109	0.0217	0.0397	0.0568
8/1/01	27.5	0.0242	0.0314	0.0242	0.0266	0.0271	0.0344	0.0262	0.0292

TINKER

Average without FS:	0.383	0.474
Average with FS:	0.0224	0.0245

NOTE: Italicized and shaded rows represent baseline sampling (i.e., without FS).

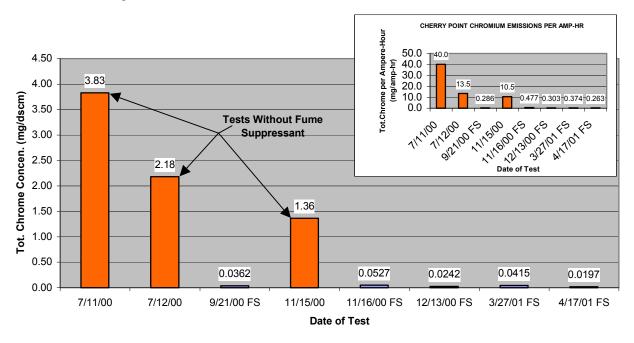
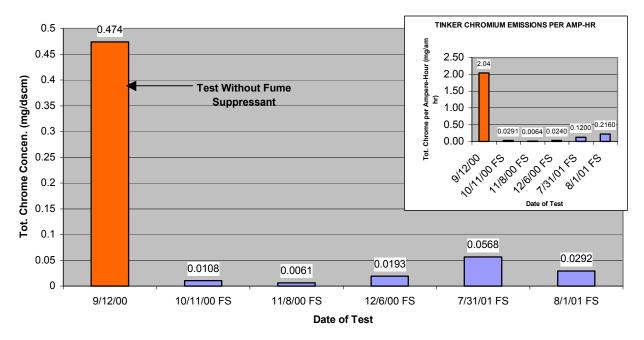


Figure 1 - CHERRY POINT TOTAL CHROMIUM EMISSIONS CONCENTRATION





Occupational Exposure Data

Table 3 presents the data from industrial hygiene (IH) sampling. IH samples were taken concurrently with the stack samples at Cherry Point and Tinker. Samples were taken in three locations: (1) a few inches directly above the sampled bath liquid surface ("In Tank"), (2) directly in front of the sampled bath in the breathing zone ("Near Tank Breathing Zone"), and (3) a few feet from the sampled bath in the breathing zone ("Remote Breathing Zone"). It would be anticipated that the most concentrated samples would be those taken above the liquid surface, and that the least concentrated would be those "remote" samples taken a few feet from the bath. In fact this was the general trend for all testing *except* at Tinker during the baseline tests (*i.e.*, tests without FS in the bath).

Each value in Table 3 represents an average of two data points, unless otherwise noted. Shaded values represent baseline samples (*i.e.*, when no FS was in the bath). Average concentrations for all testing are shown at the bottom of Table 3, both for the baseline condition, and when the baths contained FS. As noted above, the trend is clear from the averages that the hexavalent chromium concentrations decrease as the sampling location becomes more remote (except for the baseline testing at Tinker). The trend is even more dramatic if the excluded Table 3 "outlier" values, except for the 0.585 mg/m³ (2.6 x 10^{-4} gr/ft³) value, had been included (see notes 3, 4, 5, and 8).

It is also clear that the concentrations of chromium are lowest when the FS is in use (again with the exception at Tinker for samples taken in the breathing zone near the bath). In fact, for the samples taken a few inches from the liquid surface ("In Tank"), the improvement when FS is in use is greater than 20-fold. It is theorized that the improvement is not as dramatic in the breathing zone locations (and is in fact reversed for the noted Tinker "Near Tank" samples) because the concentrations are very low at those locations to begin with, and consequently the influence of other facility chromium-containing baths becomes significant.

CHE	TINKER						
	Hexavalent Ch	Hexavalent Chromium Concentration			Hexavalent Chromium Concentration		
Test Date	Remote Breathing Zone	Near Tank Breathing Zone			Remote Breathing Zone	Near Tank Breathing Zone	In Tank
7/11/00	0.041	0.038	1.450	9/12/00 am	0.115	0.014	0.201
7/12/00	0.033	0.077	1.250	9/12/00 pm	(note 6)	0.022	0.252
9/21/00 am	0.031	0.024	0.023	10/11/00 am	0.007	0.035	0.023
9/21/00 pm	(note 6)	0.043	0.043	10/11/00 pm	(note 6)	0.028	0.033
11/15/00 am	0.056	0.112	2.266	11/8/00 am	0.047	0.014	0.036
11/15/00 pm	(note 6)	(note 6)	2.400	11/8/00 pm	(note 6)	(note 6)	0.078
11/16/00 am	0.042	0.035	0.070	12/6/00	0.028	0.042	0.100
11/16/00 pm	(note 6)	(note 6)	0.120	7/31/01	0.023	0.038	0.053
12/13/00 am	0.014	0.030	0.113	8/1/01(note7)	0.050	0.018	4.23
12/13/00 pm	(note 6)	0.030	0.075				
3/27/01	0.014	0.186	0.073				
4/17/01	0.028	0.014	0.041				
Averages ⁹ :							
w/o FS:	0.043	0.076	1.68		0.083	0.018	2.23
with FS:	0.026	0.060	0.067		0.026	0.031	0.060

Table 3: INDUSTRIAL HYGIENE SAMPLING DATA - also see NOTES (concentrations in micrograms/cubic meter)

NOTES:

1 - Rows with shaded background represent baseline data (i.e., without fume suppressant [FS]).

2 - All values reported below various detection limits were averaged as the detection limit divided by the square root of 2 (*i.e.*, 1.414).

For example: if non-detect was less than 0.020 mic/cu.m. then it was reported as 0.014 (i.e., 0.020/1.414) - see reference 5.

3 - For Cherry Point, a value of 3.59 mic/cu.m. was considered an outlier from the 7/11/00 sampling for "Near Tank Breathing Zone", and was not included in the calculations.

4 - For Tinker, a value of 585 mic/cu.m. was considered an outlier from the 9/12/00 am sampling for "In Tank", and was not included in the calculations.

5 - For Tinker, 9/12/00 am, "Near Tank Breathing Zone", two locations were sampled. One of the locations had a concentration of 31.52 mic/cu.m.

This value was considered an outlier, and was not included in calculations.

6 - Only one set of samples was taken during the day, spanning the entire day (*i.e.*, am plus pm). The value shown for "am" represents the entire day.

7 - This baseline sample was taken on Tank 214. All other data were for Tank 222.

8 - For Tinker, 8/1/01, "In Tank", two locations were sampled. One of the locations had a concentration of 28.6 mic/cu.m. This value was considered an outlier, and was not included in the calculations.

9 - To calculate averages, concentrations based on a full-day sampling were given twice the weight as concentrations based on half-day sampling.

For REFERENCE:

1 - Occupational Safety and Health Administration (OSHA) Permissible Exposure Limit (PEL) is 100

micrograms per cubic meter (mic/cu.m.) as chromic oxide (52 mic/cu.m. as chromium).

- 2 Proposed OSHA PEL ranges between 0.5 and 5 mic/cu.m.
- 3 American Conference on Governmental Industrial Hygienists (ACGIH) Time Weighted Average (TWA) for

water-soluble hexavalent chromium compounds is 50 mic/cu.m. as chromium.

- 4 National Institute for Occupational Safety and Health (NIOSH) Recommended Exposure Limit (REL) for
- hexavalent chromium compounds is 1 mic/cu.m. as chromium.
- 5 Navy Environmental Health Center (NEHC), Industrial Hygiene Field Operations Manual, Chapter 4, Section 8a.(3), page 4-22.

Quality of Electroplated Parts

Hydrogen Embrittlement

Hydrogen embrittlement testing was performed on ASTM F 519 Type 1a.1 notched round bars made from 4340 steel. The bars were chromium plated at three facilities (Cherry Point, Tinker, and North Island [San Diego, CA]) while using FS and from Cherry Point and Tinker with no FS (controls). Two types of testing were performed. The first was the standard 200-hour sustained tensile load test per AMS QQ-C-320B as defined in ASTM F 519, that holds the specimen at 75 percent of ultimate tensile strength (UTS) for 200 hours. The second test was a developmental rising step load (RSL) test that holds the specimen at 75 percent of UTS test for 24 hours; followed by five percent step tensile increases each hour to failure.

All specimens from all sites and baths passed the 200-hour sustained tensile load test, indicating that FS has no deleterious effect on the embrittlement characteristics of high-strength steels plated with hard chromium. For comparison purposes, all test samples survived the initial 24-hour sustained load of the RSL test (not unexpected due to the success in the 200-hour test).

Hardness

Hardness testing was based on the Rockwell C method. Three samples from Cherry Point and Tinker with and without FS were chosen at random from a batch of 1" by 4" test coupons. Each of the samples had 10 hardness tests performed on it.

Based on the data, there appears to be no statistical difference between the results with or without FS. Therefore, the use of FS in hard chromium electroplating baths has no detrimental effect on the hardness of the plated part. An additional set of tests was run on three samples from the North Island facility, using FS. The results were similar to the Tinker and Cherry Point data.

Porosity

The pitting test detailed in AMS QQ-P-320B provides a relative measure of the quality of the electroplated chromium. Since previous generations of fume suppressants increased the porosity of the electroplated chromium, this is an important test to validate the performance of FS relative to previous products and the control baths.

Like the hydrogen embrittlement and the hardness tests, the pitting tests that were conducted showed no conclusive evidence of any detrimental effect from the use of FS. The six test coupons from Tinker showed from 5 - 40% of the coupon areas covered with pits. It was not possible to distinguish any difference in the pitting on coupons processed without FS versus those processed with FS. For the six Cherry Point coupons, the three coupons processed

without FS showed no pitting. The three coupons processed with FS showed some pitting, but also showed some markings that suggested that the coupons were mismanaged during processing and handling, unrelated to the use of FS. Two of the three North Island coupons that were processed with FS had no pits, and one had four pits. North Island does not hard chromium electroplate without FS, so no coupons processed without FS could be processed.

Adhesion

A bend-to-break adhesion test was used to evaluate the quality of adhesion of the chromium to the substrate. Five random samples of the original sets of 1-mil thick coatings from Cherry Point (with and without FS), Tinker (with and without FS), and North Island (with FS) were tested. All samples from Cherry Point and Tinker passed the test in that no loss of adhesion was noted after breaking. The North Island samples showed a small degradation in adhesion that was linked to a quality control problem and resolved. The test was repeated using five random 3-mil thick coatings from Cherry Point. No samples showed any degradation in adhesion. Based on the results, FS is considered not to have an effect on coating adhesion compared to the control coating.

Thickness

Thickness is a measurement of how close the plated coating is to the requested thickness and takes into consideration the regularity from sample to sample. There was no statistical difference in requested thickness with or without the use of FS.

Fatigue

The potential influence of FS on the fatigue characteristics of representative high-strength steels was evaluated using a Limited Equivalence Test. Three alloys were selected based on their use and importance to DOD in critical components. Fatigue specimens were designed and machined out of these alloys per ASTM E 466 and ASTM E 606. The coupons were sent to Cherry Point for electroplating of hard chromium in production baths with and without FS. Plated specimens were tested in the NAVAIR Materials Mechanical Test Laboratory. Analysis of the data indicates that the FS had no, or a slightly positive, effect on fatigue performance of the test specimens.

SUMMARY/CONCLUSIONS

With respect to atmospheric emissions, there is a 20- to 70-fold decrease in emissions from hard chromium electroplating baths when FS is used, as opposed to when FS is not used. However, it does not appear that the reduction in emissions is enough to comply with current regulatory emission standards for hard chromium electroplating baths.

With respect to occupational exposure, there is about a 20-fold reduction in the concentration of hexavalent chromium directly above the electroplating bath when FS is in use. In the breathing zone in front of the bath, and a few feet remote from the bath, concentrations of chromium are extremely low, compared to the OSHA Permissible Exposure Limit (PEL), regardless of whether FS is used or not. It appears that breathing zone concentrations are lower when FS is in use.

With respect to electroplated product quality, there appears to be no statistical difference in product quality whether or not FS is in use.

With respect to costs, there is essentially no capital cost involved in applying FS to hard chromium electroplating baths (perhaps less than \$1,000 to purchase a tensiometer to measure the surface tension of the bath). The cost of maintaining the appropriate amount of FS in the bath is typically less than \$500 per year. This maintenance cost is more than offset by the reduction in the amount of chromic acid that must be added to the bath to replace the chromic acid that is lost as mist through the ventilation system. Also, because there is less mist formed when FS is used, there is less chromium in the air pollution control scrubber wastewater discharge, therefore, less costs associated with wastewater treatment. In other words, there is a net cost savings when using FS, as opposed to not using FS.

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