



# Hexavalent Chromium Free Coatings Projects for Aerospace Applications



*TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.*

August 2012

**Chris Giesecking**  
Digital Fusion/Aviation and Missile RDEC  
Weapons Development and Integration Directorate  
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# Report Documentation Page

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Hexavalent Chromium Free Coating System  
for Magnesium Housings on Aviation Systems



# ***Hexavalent Chromium Free Coating System for Magnesium Housings on Aviation Systems Testing***

## Hexavalent Chromium Free Coating System for Magnesium Housings on Aviation Systems

### Requirement/Impact Statement

- To perform laboratory qualification testing of a completely hexavalent chrome free coating system by utilizing a hexavalent chrome free topcoat, primer, and pretreatment for magnesium parts used on Army rotorcraft.
- Specific weapon systems applications would be on any weapon system using coated magnesium components such as AH-64, CH-47, and UH-60.
- Exit criteria is a final report detailing the laboratory performance of a total hexavalent chromium free coating systems for use on magnesium aviation components.



### Description

- If successful, the end product would be laboratory qualification of a total hexavalent chrome free coating systems for use on magnesium transmission housings.
- This project will leverage work performed under the Tagnite Non-Destructive Inspection (NDI) for Aviation Systems and NDCEE Task 473-A2 Stripping of Tagnite Housings projects.
- The technical approach involves performance, materials testing, and outdoor exposure of magnesium substrates with control and alternative coatings. Recommendations will be made based on results.

#### ▪Schedule:

- Testing complete and final report distributed.

#### ▪ Performance:

- Completion of Wet Tape and Pull-Off Adhesion testing
- GMW 14872 and ASTM B117 accelerated corrosion testing complete.
- One year of exposure at beach and desert sites complete.
- Sulfur Dioxide (SO<sub>2</sub>) testing complete.

**TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.**

- **Hexavalent Chromium Free Coating System for Magnesium Housings on Aviation Systems Project Objectives:**
  - 1A-Evaluate MIL-PRF-23377 Class N primer over chromated depot and touch up conversion coatings (Dow 7 and 19).
  - 1B-Evaluate the use of MIL-PRF-23377 Class N primer over Tagnite/Rockhard based coatings as part of a **total** hexavalent chromium free coating system.
  - 2-Evaluate the use of hexavalent chrome free conversion coatings for depot and field applications.
    - Alodine 5700 and Alodine T 5900 immersion for depot applications and touch up for field applications

**Completed coating 576 coupons with six different pretreatments, three different resin coatings, two different primers, and one topcoat.**

- **Substrate:** ZE41A- T5 Magnesium
- **Pretreatments:** Alodine 5700 and T5900 Immersion and Touch Up; Tagnite 8200 Type I and Brush Tagnite 12, Dow 7 Immersion and Dow 19 Touch Up Controls (chromated)
- **Resins:** Low Temperature and High Temperature Rockhard, Touch Up Rockhard
- **Primers:** MIL-PRF-23377 Type I Class N and C2
- **Topcoat:** MIL-DTL-53039 Type I



## Hexavalent Chromium Free Coating System for Magnesium Housings on Aviation Systems

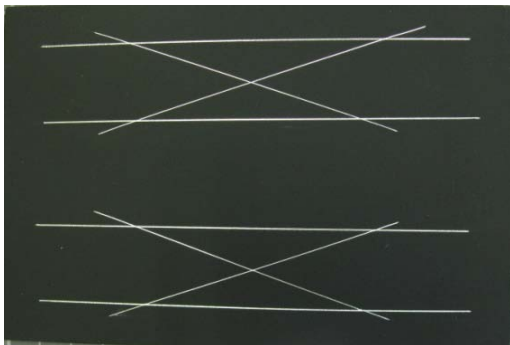


### Tests performed

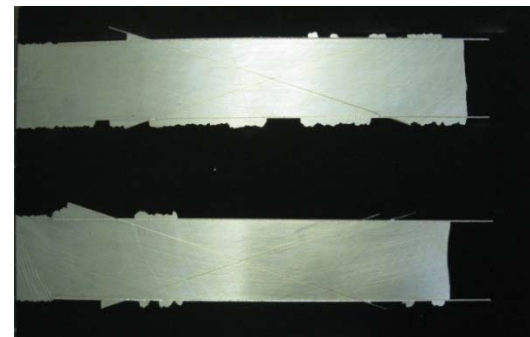
- Wet Tape Test Adhesion per ASTM D3359, Method A
- Pull-Off Adhesion per ASTM D4541-95, Method E
- Neutral Salt Fog per ASTM B117
  - 2000 hours or failure
- Accelerated Corrosion per GMW14872
  - 80 cycles or until failure
- SO2 Salt Fog per ASTM G85 Annex 4
- One Year Beach Exposure at Kennedy Space Center (KSC) Test Corrosion Site
- One Year Desert Exposure in Arizona

## Wet Tape Test Adhesion

- Performed per ASTM D3359, Measuring Adhesion by Tape Test, Method A. Immersed panels in deionized water for 24 hours.
- Alodine 5700 Immersion rated at 5A (best rating possible). This rating is equivalent to the Dow 7 Immersion Chromated Conversion Coating and the Tagnite Anodize controls.
- Alodine 5700 Immersion outperformed Alodine T 5900 Immersion.
- Alodine 5700 and Alodine T 5900 Field Touch Up Wipe applications did not perform as well as the chromated Dow 19 Touch Up Wipe control.
- For Dow 19 Touch Up Control, better adhesion with the MIL-PRF-23377 Class N primer than Class C2.



Dow 7 Control/LT Rockhard/23377N/53039  
on Mg



Alodine T 5900 Touch Up/Touch Up Rockhard/23377N/53039  
on Mg

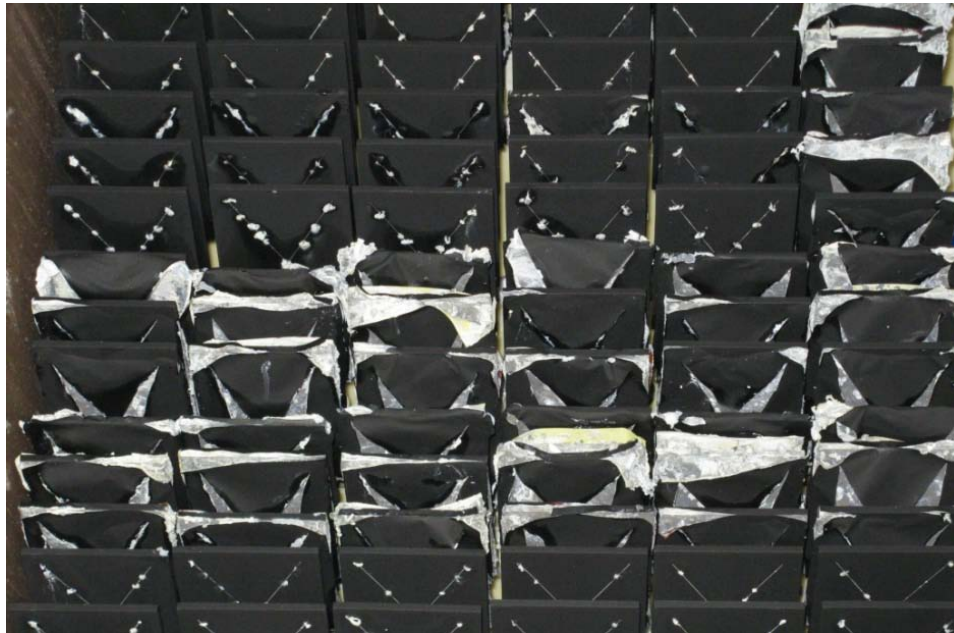


## Pull Off Adhesion Testing

- Testing per ASTM D4541.
- Pretreatment/primer/topcoat combinations that completely failed accelerated corrosion were not tested.
  - No hex chrome free immersion or touch up applications were tested.
  - Fully non chrome coating systems including Tagnite 8200 and Brush Tagnite 12/Low Temp and High Temp Rockhard/MIL-PRF-23377Type I Class C2 and N/MIL-DTL-53039 were tested as well as chromated controls.
- Most failures were cohesive in the MIL-DTL-53039 topcoat layer at 1000-1500 psi.



- Testing was performed per ASTM B117 continuous salt spray.
- All non chromated conversion coatings for magnesium (Alodine 5700 and T 5900, immersion and touch up) were total failures after 336 hours of continuous salt spray testing.
- Promising results indicated for a **total hex chrome free coating system** for Tagnite 8200 and Brush Tagnite 12 with Rockhard/MIL-PRF-23377 Class N/MIL-DTL-53039.
- GMW 14872 cyclic corrosion results were very similar to ASTM B117 continuous salt spray results.



# Hexavalent Chromium Free Coating System for Magnesium Housings on Aviation Systems Beach Exposure

- One year exposure at Kennedy Space Center Test Corrosion Site. Specimens assessed per ASTM D1654.
- Tagnite 8200 Type I and Brush Tagnite 12 based systems with chrome and non-chrome primer were performing well after one year of exposure.
- In general, hex chrome free immersion and touch up applications of Alodine 5700 and T 5900 were total failures after one year of exposure.
- Outdoor exposure results similar to laboratory accelerated corrosion.



3B=Tagnite/HT Rockhard/  
23377N/53039  
12 Months



3E=Tagnite/LT Rockhard/  
23377N/53039  
12 Months

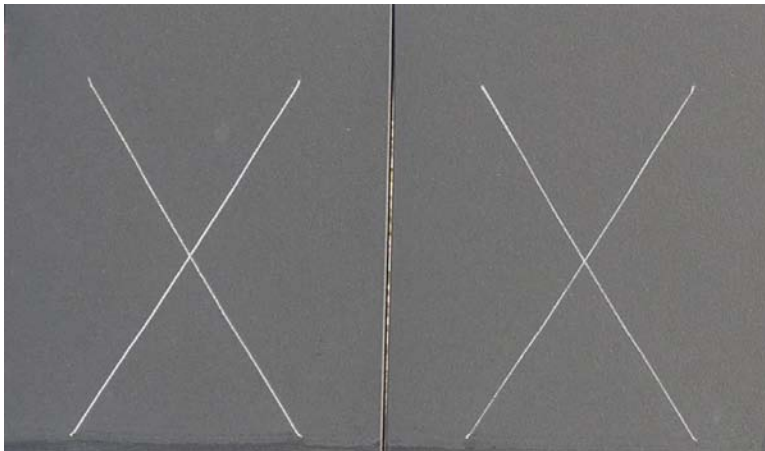


4G=BrushTagnite/  
Touch Up Rockhard/  
23377N/53039  
12 Months

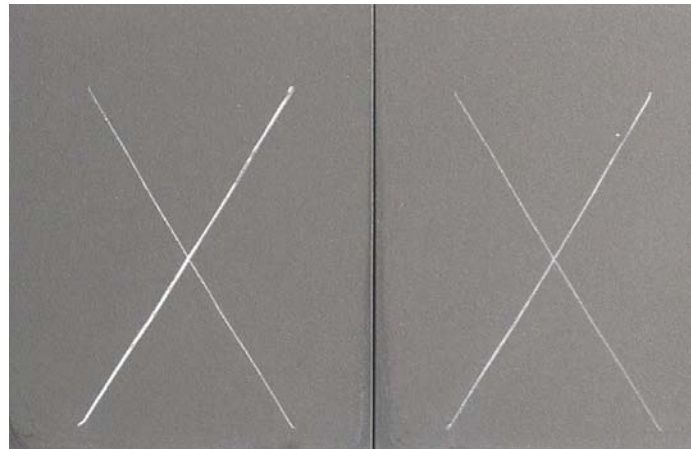


5D=Alodine5700Immersion/  
LT Rockhard/  
23377C/53039  
12 Months

- First mounted on the exposure racks on April 16, 2010 and last evaluated August 17, 2010 at Wittman, AZ.
- The specimens were assessed per ASTM D1654.
- After one year desert exposure, the majority of panels performed well except for some of the hexavalent chrome free conversion coatings.



3A=Tagnite/HT Rockhard/  
23377C/53039  
12 Months



3B=Tagnite/HT Rockhard/  
23377N/53039  
12 Months



## Overall Top Performing Coating Systems

- **Tagnite 8200 Type I/High Temperature Rockhard/MIL-PRF-23377 Type I, Class C2 or Class N/MIL-DTL-53039 Type II, polymeric**
  - High Temp Rockhard slightly better than Low Temp.
- **Dow 7/ High Temperature Rockhard/MIL-PRF-23377 Type I, Class C2/MIL-DTL-53039 Type II, polymeric**
  - Dow 7 with High Temp Rockhard slightly outperforms Low Temp with either primer.
  - Dow 7 with High or Low Temp Rockhard shows a slight decrease in performance with Class N primers.
  - Tagnite 8200 with High or Low Temp Rockhard coatings appears to be slightly superior to the equivalent Dow 7 coating system and less sensitive to primer type (Class C2 or Class N).
- **Brush Tagnite 12/Touch Up Rockhard/MIL-PRF-23377 Type I, Class N or Class C2/MIL-DTL-53039 Type II, polymeric for Touch Up Applications**



## ■ Path Forward

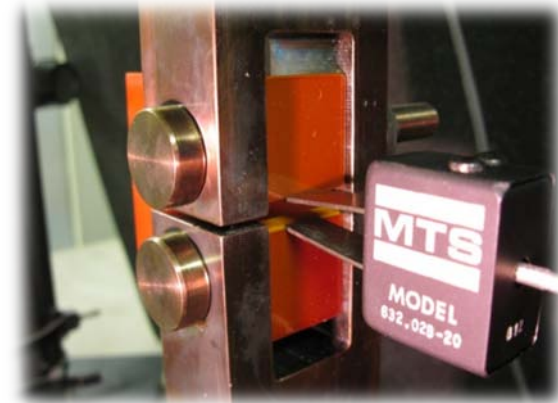
- All laboratory testing completed and final report distributed.
- Two additional years of outdoor exposure and galvanic testing recommended for promising magnesium coating systems identified under this program including Tagnite 8200 Type I, Brush Tagnite 12, and MIL-PRF-23377 Class N based systems.
- Dow 17 anodize controls should also be tested in outdoor environments alongside Tagnite based systems.
- Demonstration and validation on any promising coating system in a field or depot environment should be performed.
- SO2 testing at NAVAIR complete and results are being evaluated at AMRDEC.



# ***Tagnite Non-Destructive Inspection (NDI) Testing for Aviation Systems***

## Requirement/Impact Statement

- Eliminate the use of pretreatments containing hexavalent chromium on magnesium housings and obtain improved corrosion resistance over conventional processes.
- Identification of improved procedures for depot and organizational level maintenance.
- The impact will be DOD wide on all Tagnite/ Rockhard coated magnesium parts used in Aviation.



## Description

- This will leverage the work done under NDCEE Task 473-A2 Stripping of Tagnite Housings.
- The technical approach will show proof of magnesium crack detection through coated magnesium components.
- Specific weapon systems applications will be on any weapon system using Tagnite/ Rockhard coated magnesium parts.
- Funded provided by the SPOTA Program.

- **Schedule:**
  - End date – April 2011
- **Performance:**
  - All required project testing complete.
  - Final report complete and has been distributed.

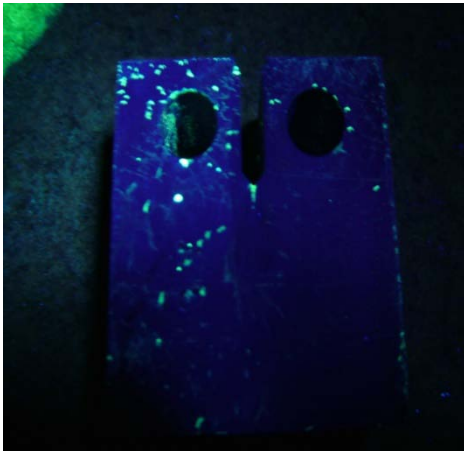


- **This program evaluated the capabilities of Fluorescent Penetrant Inspections (FPI) to determine if component flaws can be detected under coating systems**
  - ZE 41A T5 magnesium notched fracture toughness coupons purchased.
  - Cracking Methodology Developed and Verified (via Photo-Micrograph) at AMRDEC.
  - Coupons coated.
  - Coupons cracked to 0.03” and some to 0.05”.
  - Coupons NDI tested per ASTM E 1417, Type I, Level 3, Method D Swab Method followed by the Bleed Back Technique.
  - Project testing complete.
  - Final report distributed.

## Preliminary Results for 0.03” Cracks

### ▪ Bare Magnesium Coupons

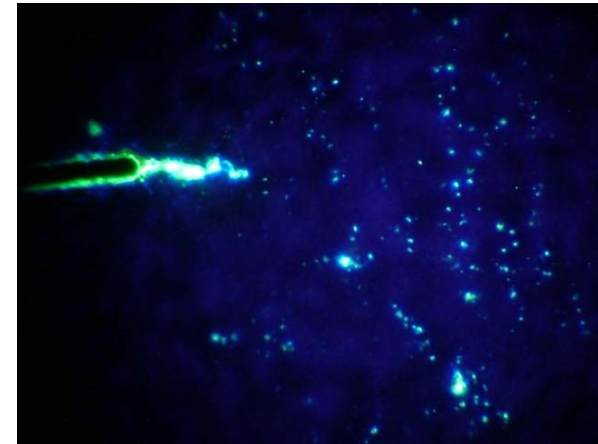
- Surface pitting occurred during the post inspection cleaning process.
- Residual penetrant remains on the bare surface after cleaning which could have detrimental effects.
- Penetrant bleed out is adequate with some bleed out from pre crack-notch at 0X,10X. Pre-crack notch is distinguishable from crack at 60X magnification.
- Background fluorescence is acceptable.



Surface Pitting



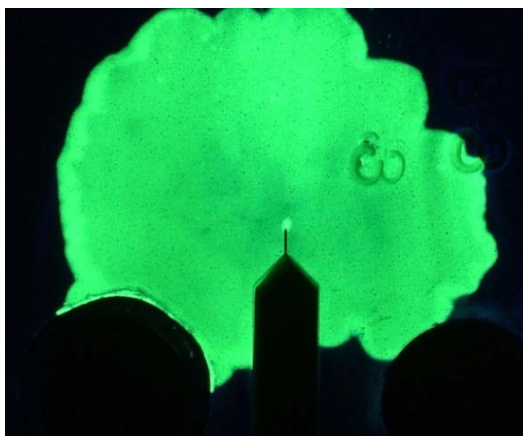
Residual Penetrant, 0X



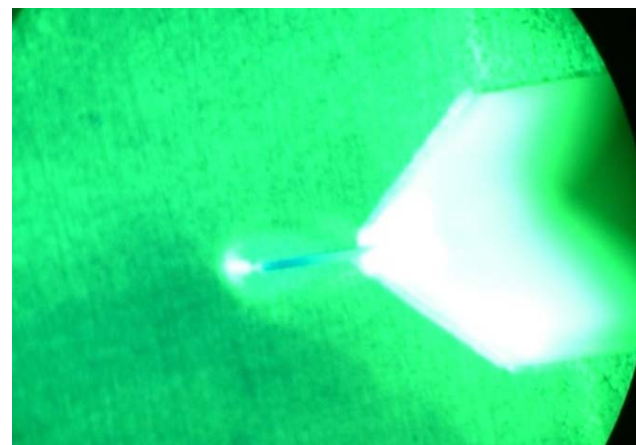
Notch and Crack, 60X

## ■ Tagnite 8200, Type I Anodize

- The coating porosity trapped the developer chemicals causing extreme, unacceptable background fluorescence.
- Penetrant bleed out nearly undetectable at 0X, 10X.
- The NDT CoE recommends that actual aircraft components with porous coatings comparable to Tagnite (only) should not be inspected with a Method D FPI process.



10X



Notch and Crack at 60X, Bleed Back

## ■ Tagnite 8200, Type I Anodize/High Temp Rockhard

- Rockhard coating over Tagnite eliminates the surface porosity and absorption of developer chemicals.
- Penetrant bleed out is adequate with some bleed out from pre-crack notch at 0X,10X. Pre-crack notch distinguishable from crack at 60X.
- No background fluorescence providing very good contrast.



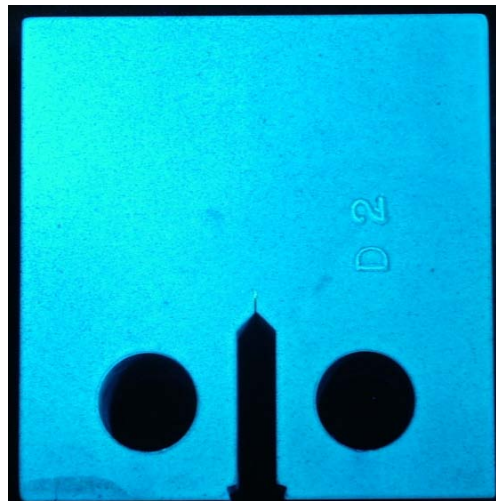
0X



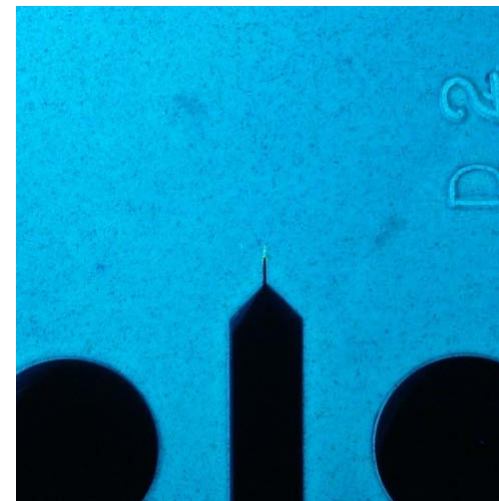
Notch and Crack at 60X

- **Brush Tagnite 12/Low Temp Rockhard 985**

- Penetrant bleed out is adequate with minimal bleed out from pre-crack notch at 0X, 10X. Pre-crack notch is easily distinguishable from crack at 10X.
- Background fluorescence is almost nonexistent.
- Coating surface is reflective, reducing contrast of penetrant indication.



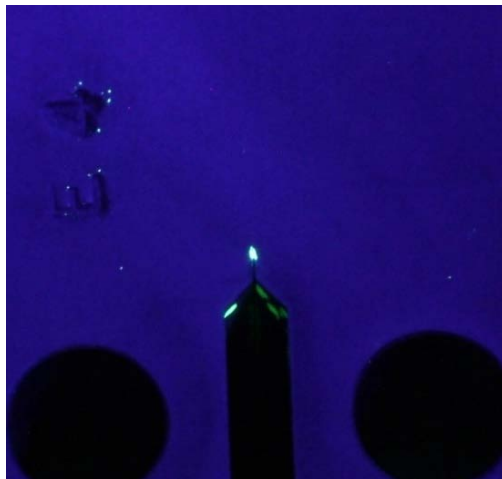
0X



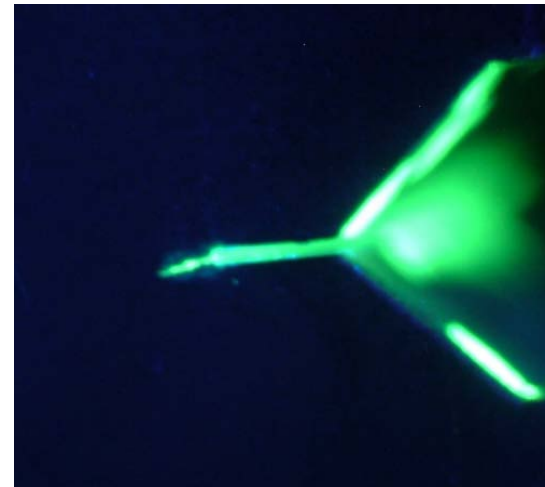
10X

## ▪ Acid Bi-fluoride Pickle/Alodine 5700

- Penetrant bleed out is adequate with some bleed out from pre-crack notch at 0X, 10X.
- Indication is blotchy and cannot distinguish notch from the crack.
- At 30X using bleed back techniques, the crack is more distinctive.
- Background fluorescence is minimal.



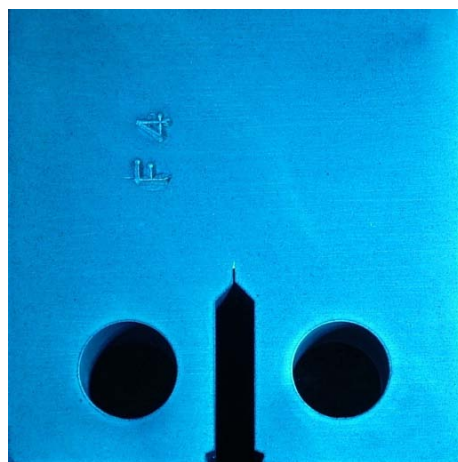
10X



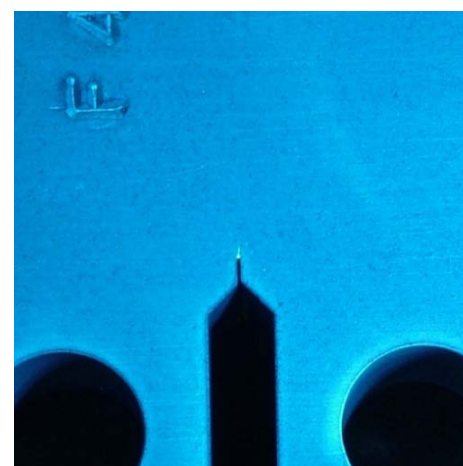
Bleed Back, 30X

▪ **Acid Bi-fluoride /Alodine T 5900/Low Temp Rockhard**

- Penetrant bleed out is adequate with minimal bleed out from pre-crack notch at 0X,10X. Penetrant bleed out from the crack is easily separated from pre-crack notch bleed out at 10X.
- Background fluorescence is almost nonexistent.
- Coating surface is reflective, reducing contrast of penetrant indication



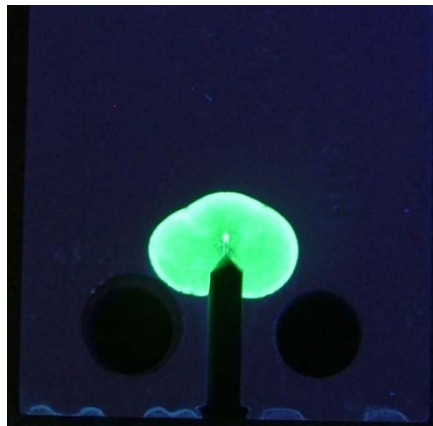
0X



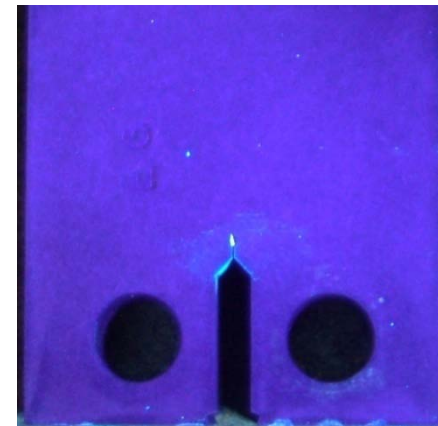
10X

## Preliminary Results for 0.05" Cracks

- **Only Tagnite and Alodine 5700 panels were tested.**
  - Background fluorescence, penetrant bleed out, and bleed back results were predictable based on 0.03" testing.
  - For Tagnite only, the background fluorescence is unacceptable. For Alodine 5700, background fluorescence is minimal.
  - The 0.05" cracks were relatively easy to detect. The cracks extended beyond the pre-crack notch bleed out.



Tagnite only



Alodine 5700



## Summary

- The NDT CoE recommends that if an actual aircraft component contains any geometric feature similar to the pre-crack notch, the dip method for penetrate application should not be utilized if cracks less than approximately 0.05" are expected.
- For Tagnite only coupons, the porosity of the coating trapped the developer chemicals and the entire part fluoresced. The Alodine 5700 coating did not trap the developer chemicals.
- For all Rockhard coated test coupons, detection of cracks was easier than for pretreated only coupons (Tagnite and Alodine 5700). The Rockhard surface appears to crack with the underlying magnesium substrate.
- The cleaning procedure after NDI caused surface corrosion on Bare and Alodine 5700 coupons. For Tagnite only with 0.03" cracks, NDT CoE recommends not using Method D FPI process on comparable coatings.

## Path Forward

- Recommend follow-on testing with components more similar in size and shape to aviation components due to a decrease in the probability of detection on larger, more complex geometries such as transmission housings.
  
- Evaluate the conclusions of the leveraged projects
  - NDCEE Task 473-A2 Stripping of Tagnite Housings
  - Hexavalent Chromium Free Coating System for Magnesium Housings on Aviation Systems projects.
  
- A demonstration and validation at an Army depot is recommended to evaluate realistic test sample conditions, inspection procedures, and leveraged projects (depot conditions, corroded coupons, etc).



# ***Demonstration of Hexavalent Chromium Free Coatings for Missile Weapon Systems***

## Requirement/Impact Statement

- This project is intended to address the AERTA requirements PP-1-02-04 (SPOTA) and PP-2-02-03 (TMR) Surface Finishing Processes
- Memorandum for Secretaries of the Military Departments, Subject : Minimizing the Use of Hexavalent Chromium (Cr<sup>6+</sup>)
- This project was intended to reduce the use of hexavalent chromium and VOC emissions associated with the depot maintenance of AMCOM missile weapon systems at LEAD.



## Description

- The products evaluated under this program included three alternatives to chromated DoD-P-15328 wash primer, TCP treatment for aluminum, non-chromated post treatment for zinc phosphate, and qualification for MIL-PRF-23377 Class N primer on steel.
- The technical approach involved performance, materials compatibility, and outdoor exposure testing.
- Funding provided by SPOTA and AMCOM Corrosion Program Office. Assistance provided by AMCOM G4 Environmental.

### ▪ Schedule:

- Anticipated end date: June 2012.

### ▪ Performance:

- Most accelerated corrosion and adhesion laboratory testing has been completed.
- Outdoor exposure complete.
- SO<sub>2</sub> and Pull Off adhesion evaluations and report in process.



## Objectives of program are to demonstrate the following for missile systems as part of a total hexavalent chromium free coating system:

- 1-Evaluate trivalent chromium pretreatment (TCP) for use on aluminum
- 2-Evaluate three hexavalent chrome free alternatives to DoD-P-15328 wash primer for mixed metal assemblies
- 3-Evaluate the performance of MIL-PRF-23377 Class N primers applied over steel substrates treated with zinc phosphate
- 4-Evaluate the use of a hexavalent chromium free sealers for post application sealing of zinc phosphate treated steel



# Demonstration of Hexavalent Chrome-Free Coatings for Missile Weapon Systems



SUBSTRATES	PRETREATMENTS	PRIMERS	TOPCOATS
AA-2024-T3	DoD-P-15328 (Wash Primer)	MIL-DTL-53030	MIL-DTL-53039, Type II, Silica Flattening Agents
AISI 4340	MIL-DTL-81706B, Type I, Class 1a (Alodine)	MIL-DTL-53030, Second Generation "Type II"	MIL-DTL-53039, Type II, Polymeric Flattening Agents
	MIL-DTL-81706B, Type II, Class 1a (TCP)	MIL-DTL-53022 Type I	MIL-DTL-64159, Type II, Polymeric Flattening Agents
	Spectrum Coatings EXGWP-508	MIL-PRF-23377 Type I, Class N (Non Chromate)	
	NAVAIR Chrome Free Process (CFP)	MIL-PRF-23377 Type I, Class C2 (Strontium Chromate)	
	TT -C-490, Type I, Zinc Phosphate with Cr <sup>6+</sup> Sealer		
	TT -C-490, Type I, Zinc Phosphate with TCP Sealer		

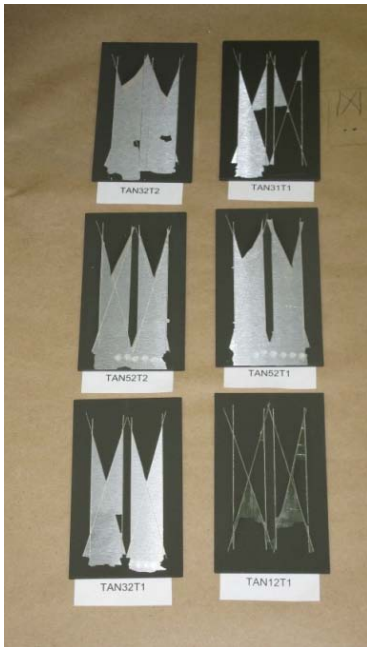


## Testing performed

- Wet Tape Test Adhesion per ASTM D3359, Method A
- Pull-Off Adhesion per ASTM D4541-95, Method E
- Neutral Salt Fog per ASTM B117
  - 2700 hours for steel or failure
  - 2000 hours for aluminum or failure
- Accelerated Corrosion per GMW14872
  - 80 cycles or until failure
- SO2 Salt Fog per ASTM G85 Annex 4
- Beach and Desert Exposure
- Exposure at Redstone Arsenal, Static Test Stand to rocket motor exhaust
- Hydrogen Embrittlement
- Fastener Hardware Assembly

# Wet Tape Test Adhesion

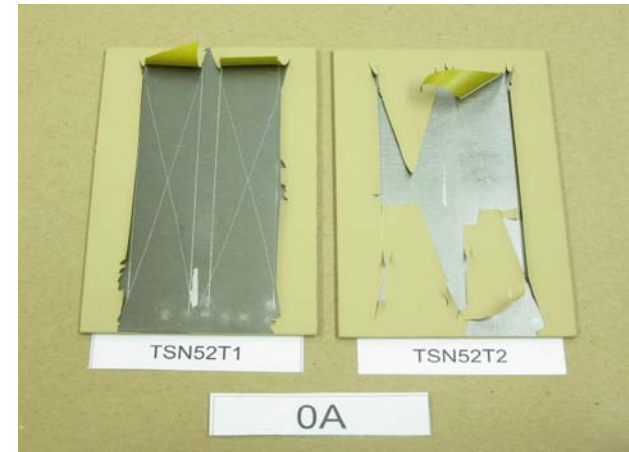
- Performed and evaluated per ASTM D3359, Standard Test Methods for Measuring Adhesion by Tape Test, Method A. Immersed panels in deionized water for 24 hours.
- Testing completed for all steel and aluminum panels.



**Aluminum, 0A  
 Predominantly Spectrum**



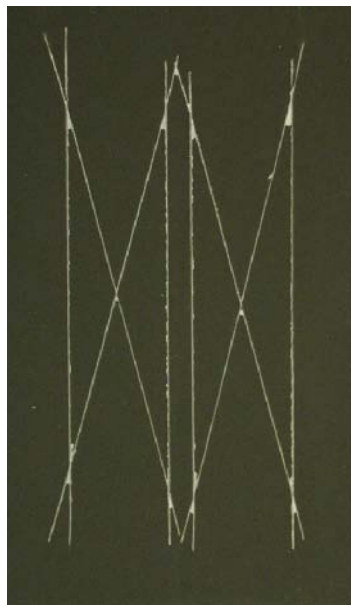
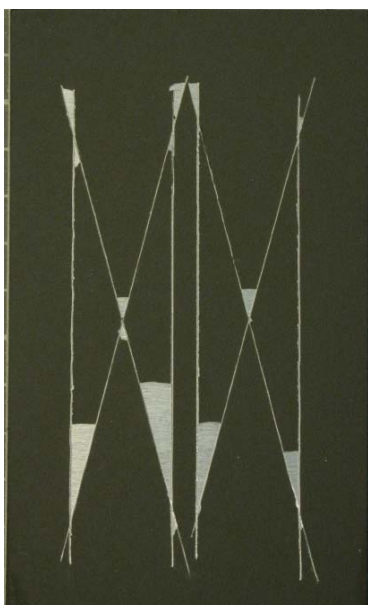
**Aluminum, 4A  
 One Spectrum**



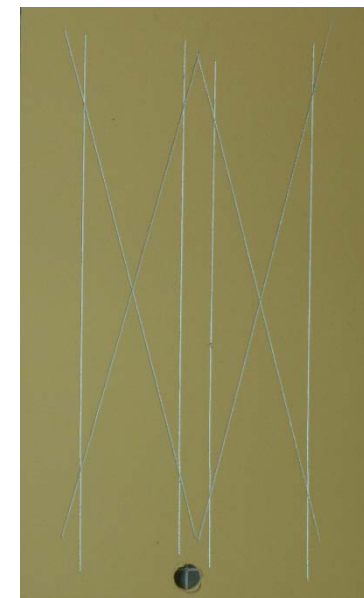
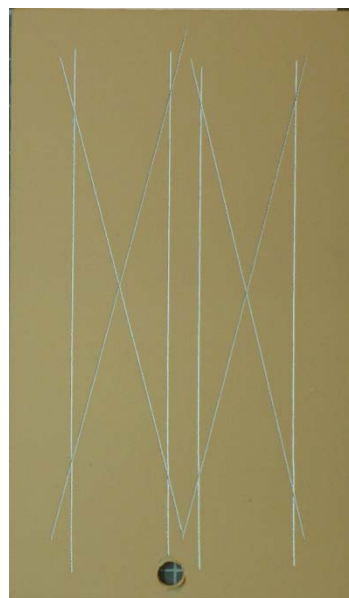
**Steel, 0A  
 All Spectrum**



## Wet Tape Adhesion Test Hexavalent Chrome Free Systems



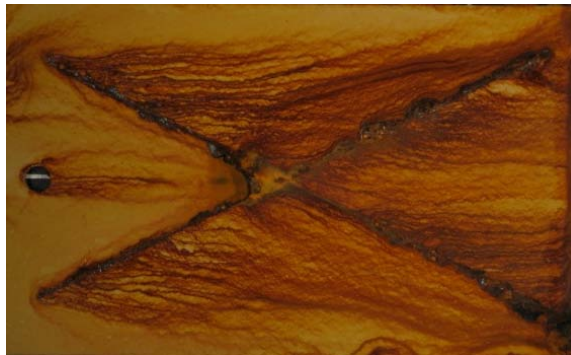
TCP based coating systems  
Objs. 1,2



Parcolene 99X hex chrome free sealer  
for zinc phosphate based coating systems  
Obj. 4

## Accelerated Corrosion Testing

- ASTM B117 for 2000-2700 hrs and GMW 14872 to 80 cycles.
  - Overall results show GMW 14872 was the more aggressive test for aluminum, steel.
- General results
  - 1- Alodine 600 slightly outperformed TCP for corrosion resistance. TCP exhibited less blistering.
  - 2- For mixed metal testing, DoD-P-15328 and TCP performed similarly on aluminum. On steel, DoD-P-15328 performed much better than any alternatives.
  - 3-MIL-PRF-23377 Class C2 outperformed all non-chrome primers. All non-chrome primers performed well except MIL-PRF-23377 Class N, which showed inconsistent performance for each corrosion test.
  - 4-One Parcolene 99X chrome free sealer systems outperformed the chromated control sealer for zinc phosphate.



Parcolene 99X system  
2700 hours



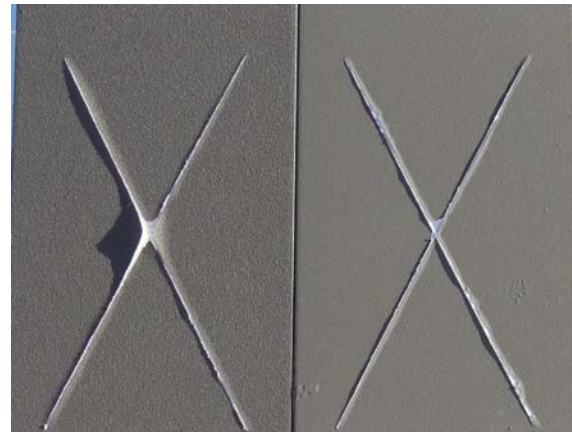
FH3 Chromated Sealer system  
2700 hours

## Desert Exposure Testing

- Exposure at Wittman, Arizona Test Site. The specimens were assessed per ASTM D1654 after 12 months.
- After one year, the majority of aluminum and steel specimens for all objectives were in good condition, with a few major failures and delaminations.
  - Spectrum and TCP showed coating delamination after a few months of exposure.
  - All primers tested and the chrome free alternative sealers for zinc phosphate performed well.
  - DoD-P-15328 and Alodine 600 chromated controls performed very well after one year.



Aluminum Spectrum EXGWP-508  
4 Months Desert Exposure



Aluminum TCP  
12 Months Desert Exposure

## Beach Exposure Testing

- Exposure at Kennedy Space Center Corrosion Test Site. The specimens were assessed per ASTM D1654 after 12 months.
- After one year, the majority of aluminum and steel specimens for all objectives were in good condition, with a some major failures.
  - The Alodine 600 and TCP performed well. Some delamination of TCP treated panels.
  - DoD-P-15328 was the best overall performer on aluminum and steel. Spectrum was the top performer on steel but performed very poorly on aluminum.
  - Parcolene 99X sealer for zinc phosphate based systems consistently outperformed the control.



## RSA TA5 Outdoor Testing

- Exposure at Redstone Arsenal, AL; Static Test Area 5, Stand E. The specimens were fully assessed per ASTM D1654 at the end of the 12 months.
- After one year, aluminum and steel specimens for all objectives were generally in good condition with no major failures.
  - TCP exhibited more blistering on aluminum and steel panels than alternatives tested under Objs 1,2.
  - MIL-DTL-53030 1<sup>st</sup> and 2<sup>nd</sup> gen primers did not perform as well as MIL-DTL-53022 or MIL-PRF-23377 Class C2 or N.
  - Several Parcolene 99X chrome free sealer based coating systems performed as well as the chromated sealer.





## ■ Current Status

- All laboratory testing has been completed. Evaluations and data analysis of SO<sub>2</sub> and Pull-Off adhesion panels are underway.
- A final report has been completed and distributed. An addendum will be submitted with SO<sub>2</sub> and Pull-Off data.

## ■ Conclusions

- **Objective 1:** TCP on aluminum exhibits adhesion and delamination failures in outdoor environments.
- **Objective 2:** Identification of an acceptable alternative to currently used chromated DoD-P-15328 wash primer for mixed metal assemblies was not successful.
- **Objective 3:** The use of MIL-PRF-23377 Class N primer over TT-C-490 E, Type I Zinc Phosphate (with a hex chrome free sealer) shows no performance gain over currently used hex chrome free missile primers.
  - No data collected for the use of MIL-PRF-23377 Class N primer with chromated DoD-P-15328.
- **Objective 4:** Hexavalent chromium free sealer for zinc phosphate treated steel, Parcolene 99X, shows potential.
  - Performed well in corrosion, adhesion, and outdoor environments.
  - Need mixed metal testing and dem val



# POCs

Mr. Chris Giesecking  
Digital Fusion/Aviation and Missile RDEC  
256-313-6768  
[Chris.giesecking@us.army.mil](mailto:Chris.giesecking@us.army.mil)

Mr. Dusty Holt  
Digital Fusion/Aviation and Missile RDEC  
256-842-9807  
[dustin.r.holt.ctr@us.army.mil](mailto:dustin.r.holt.ctr@us.army.mil)

Ms. Nancy Whitmire  
Aviation and Missile RDEC  
256-876-1667  
[Nancy.whitmire@us.army.mil](mailto:Nancy.whitmire@us.army.mil)