Summary: ASETSDefense 2011, New Orleans

The briefings are now posted on the ASETSDefense website at:

http://www.asetsdefense.org/SustainableSurfaceEngineering2011.aspx

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1320	Ionic Liquid Aluminum Plating for Cd Replacement	Toshi Murai, Tarek Nahlawi Dipsol Chemical Co. Japan; Dipsol of America
1340	Qualification of Cold Spray for Repair of MIL-DTL-83488 Aluminum Coatings	Steve Gaydos Boeing Research & Technology
1400	Qualification of Brush Plating Repairs for Cd, Hard Chrome & Alternatives	Mary Gilman Boeing Research & Technology
1420	Environmentally Compliant Sealant Removers	James Tankersley Battelle Dayton
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1530	NASA TEERM Progress: Cr^{6+} Elimination for Electronics	Matt Rothgeb, Kurt Kessel NASA
1600	Cd and Cr^{6+} on Connectors: Alternatives & Issues	Gerald Tredan Radiall (France)
1620	Tin-Zinc for Cd Replacement on Connectors	Tarek Nahlawi Dipsol of America
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0900	Gun Barrel Coatings - PVD	Gennady Yumshtyk Paradigm Shift
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0950	New AMS Specs for Grinding and Superfinishing of HVOF Coatings	Jon Devereaux NASA
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1030	Corrosion Testing and the Non-Chrome Engineering Circular	Craig Matzdorf NAVAIR
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2. Computational & Database Methods for Design & Prediction – Side meeting

A summary of this side meeting is available on the ASETSDefense website.

3. Technical sessions

ASETSDefense is an initiative of SERDP/ESTCP (www.serdp-estcp.org) intended to develop information databases and organize workshops associated with technologies in the surface engineering field. Figure 1 shows the database of technical documentation that is available *http://db.asetsdefense.org*. Note that this a public database that contains unlimited distribution documents, including technical reports, authorizations, implementations, test plans, and information about ongoing projects. The username and password (available from ASETSDefense@rowantechnology.com) is only necessary for access to limited distribution documents.

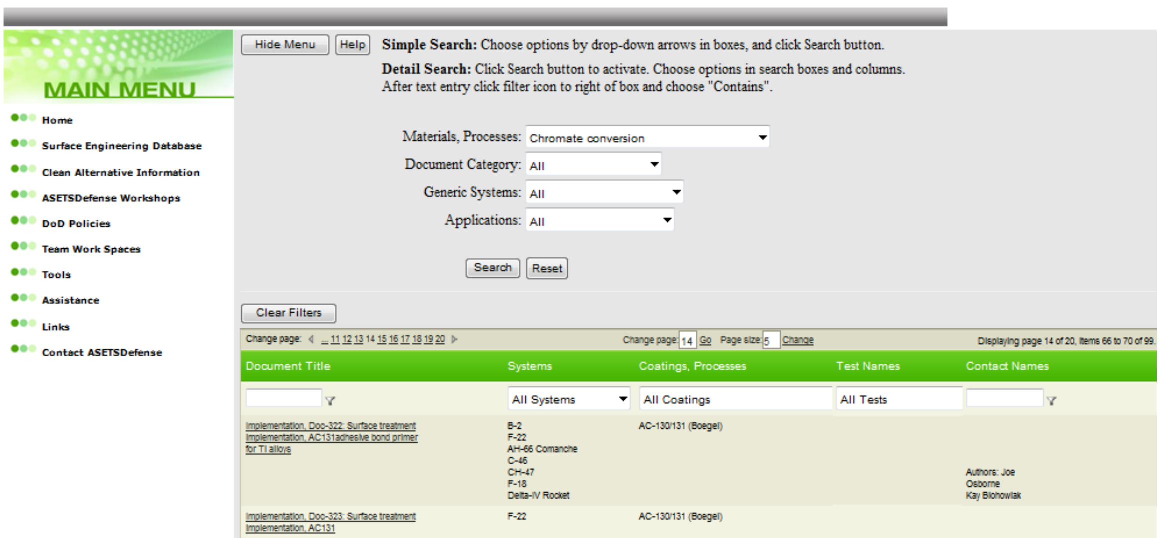

Figure 1. ASETSDefense public database (http://db.asetsdefense.org).

SERDP/ESTCP has invested $30 million in surface engineering technologies over the past four years, the bulk of which has been on projects related to reduction of hexavalent chromium.

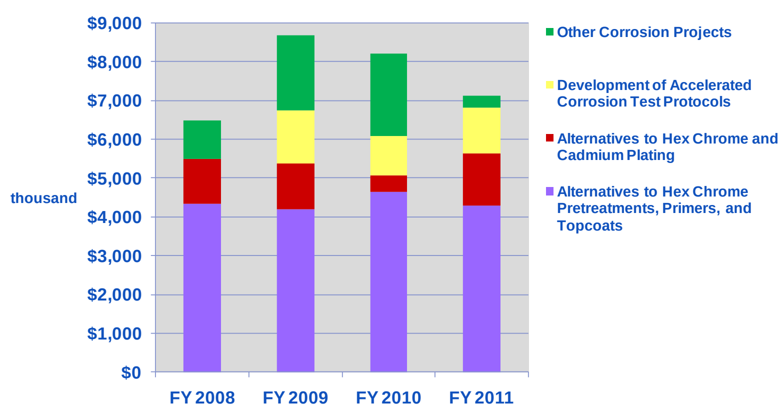

Figure 2. SERDP/ESTCP investments in surface engineering.

3.1. Drivers for material replacement

Replacement of materials, coatings and processes continues to be driven by Environmental Safety and Occupational Health (ESOH) requirements, including:

- The DoD memo of 2009 "Minimizing the Use of Hexavalent Chromium (Cr6+)"
- European REACH and RoHS regulations
- US EPA and OSHA regulations
- US state regulations

3.1.1. DoD policy and DFARS

The DoD 2009 memo was described by Paul Yaroschak as a policy designed to minimize the use of hexavalent chromium:

- The DoD policy does not ban the use of hexavalent chromium
- The policy does provide a strong forcing function to use substitutes…where they can meet performance requirements
- New systems: use requires executive level approval; must certify no acceptable substitute
- Legacy systems: evaluate substitutes during system modifications & maintenance, as practical

The intent of the memo will be enforced on OEMs and suppliers through the use of a Defense Federal Acquisition Regulation Supplement (DFARS), which will be included in future defense contracts. The wording of the DFARS has not yet been completely decided, but it will incorporate the following principles

- Purpose: Implement the DoD policy and prevent unwanted/unknown hex chrome products from entering the system
- DoD contracts/specs cannot result in deliverables with Cr^{6+} greater than 0.1% by weight
- Exceptions:
  - Legacy systems – those past Milestone A
    - But alternatives should be considered during system mods, overhauls, maintenance procedure updates
  - Sustainment contracts (parts, services) for systems where Cr^{6+} previously approved
  - Does not include Cr^{6+} produced as a by-product of a process

The DFARS is likely to be issued in the Federal Register in the first quarter of 2011.

To help DoD agencies adopt green products, DLA is involved in a number of HAZMIN projects, and has created an Environmental Attribute Code (ENAC). At the present time there are codes for non-asbestos, non-Cd, and non-Hg. However there is not yet a non-chromate ENAC, since that is still under discussion.

3.1.2. REACH

REACH continues to be a serious concern for both DoD and for the supply chain, not just because there are no Europe-wide Defense Exemptions, but also because of its effect on materials used in defense systems. DoD has developed a Strategic Plan for Managing Chemicals, Materials, and Impacts to Readiness from REACH. Paul Yaroschak summarized the effect of REACH on DoD, which will include supply chain disruptions and increased costs (see Table 1).

Table 1. Effect of REACH on DoD (Yaroschak).

EXPECTED OUTCOMES ON COMMERCE	POTENTIAL IMPACTS TO DoD
Limiting/eliminating some chemical availability	Negative effects on U.S. military operations and maintenance in the EU
Decreased material availability and increased costs for certain chemicals/articles	Disruption to defense supply chains outside the EU due to the global nature of supply
Undisclosed substitution of chemicals in Commercial, Off-the-Shelf items	Failure or marginal performance of weapon systems or components of weapon systems
Increased equipment costs passed on to foreign customers when substitute materials are available to satisfy individual country requirements	Increased equipment costs *eventually* passed on to DoD
Different interpretations of REACH by each of the EU / participating states (30)	Disruption of U.S. and NATO interoperability (e.g., FMS)
Accidental release of proprietary information	Accidental disclosure of classified or controlled unclassified information
Accelerating the need to test and evaluate substitute materials	Increased DoD research and development costs

Scott Fetter of Lockheed-Martin provided an OEM viewpoint on REACH. The aerospace and defense industry is very small compared to the chemical industry and general manufacturing. As a result, the markets for some chemicals that are critical to DoD and aerospace are too small to make it worthwhile for the chemical companies to register them under REACH for aerospace and defense use. At the same time producers of aerospace and defense products may not be able to afford registration, especially for products that represent only a small part of their business. The result is that the aerospace and defense industries are losing critical materials from the supply chain, such as Click Bond adhesive, which is used throughout the F-35.

Chemicals are often withdrawn from the worldwide market very quickly if they are put forward as candidates for authorization (REACH Annex XIV) or as Substances of Very High Concern (SVHCs) (Annex XVII). The result is that alternatives must be found, qualified and implemented far faster than system safety would dictate. Any problem or qualification failure is likely to leave OEMs without critical materials.

Under the REACH "Article Exemption" most products that contain SVHCs can still be imported into Europe, even though they cannot be manufactured in Europe. This situation is not expected to last a great deal longer.

European companies are using a similar approach to US companies in meeting the requirements of REACH. Figure 3 shows the approach being taken by SAFRAN to deal with short-term and long-term development of alternatives. Note also Objective 4 in this figure,

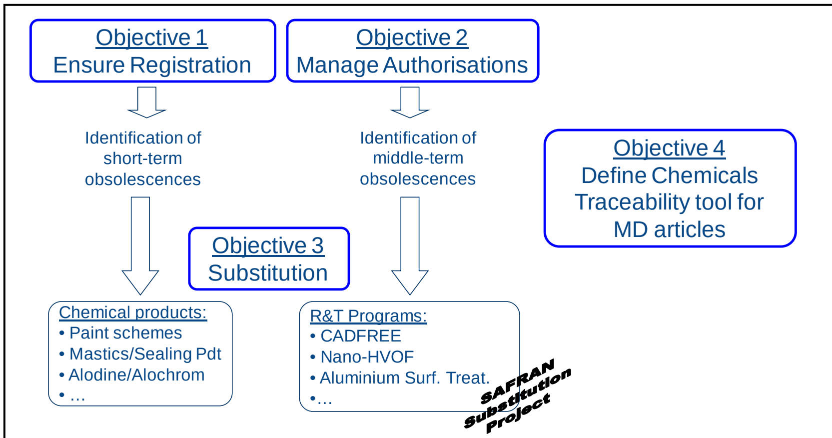

Figure 3. Short term and long term responses to REACH (Alain Viola, SAFRAN, France).

which is an issue also raised by US manufacturers. It is often very difficult to know what materials are actually in the product; one may know from the MSDS what the precursor chemicals were, but these are not necessarily to be found in the product itself.

A great many non-chromate and cadmium-free products are being used in Europe but not in the US, and vice versa. In general, however, European and North American companies are gravitating towards similar approaches in finding alternatives to restricted chemicals.

3.1.3. Nanomaterials

There is increasing concern over the potential ESOH risks of nanomaterials, and Europe is beginning to regulate them under REACH. However the definition of what constitutes a nanomaterial has not yet been decided, and some suggested definitions are so loosely formulated as to encompass not just those made with nanoparticles, but essentially all materials.

In an attempt to improve the corrosion performance of primers, paints, and other coatings, formulators are beginning to incorporate nanoparticles to carry inhibitors, limit the permeation of fluids, or improve the wear and abrasion resistance. Restrictions on use of nanomaterials may reduce the performance of chromate alternatives, inhibiting their adoption and forcing continued reliance on chromates.

3.2. Issues and solutions for vehicles and aircraft

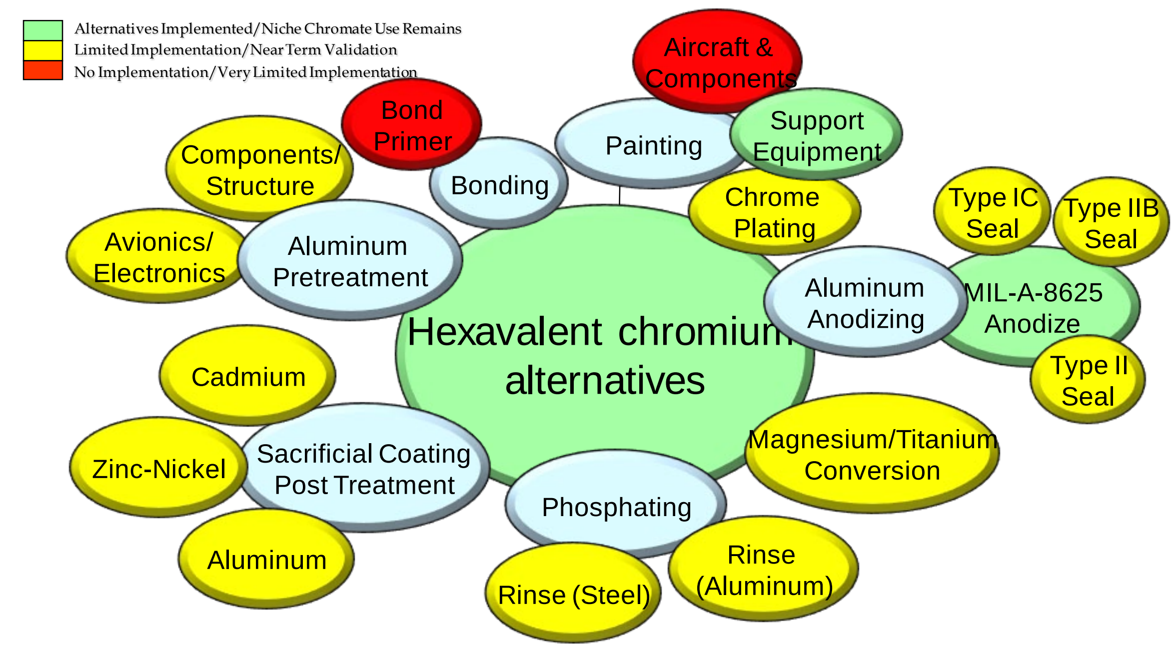

Figure 4. Current status of chromate replacement (Matzdorf, NAVAIR).

From NAVAIR's viewpoint the current status of Cr^{6+} replacement is summarized in Figure 4. The most important remaining issues are bond primers and painting of aircraft and components. NAVAIR has recently released a Non-chromate Coatings Engineering Circular to define how Cr^{6+} alternatives should be transitioned into Naval aircraft. This circular can be made available to DoD organizations.

3.2.1. Vehicles

Vehicles are moving towards e-coats and Zn-rich primers, while silica flattening agents have been eliminated from all MIL-DTL-53039 and MIL-DTL-64159 CARC topcoats. In the future the Army will be looking for more e-coating and powder coating, as well as low solar absorbing CARC.

For USMC vehicles a Corrosion Prevention and Control Plan (CPCP) is becoming an integral and essential part of the acquisition process. USMC Corrosion Prevention and Control (CPAC) Program is testing various potential solutions. Among the initiatives that have paid premiums is an Accelerated Corrosion Durability Road Test (ACDRT), in which an initial production vehicle is extensively road-tested so that design flaws that lead to corrosion can be corrected before large numbers of vehicles are produced.

3.2.2. Aircraft

Joe Osborne of Boeing provided an overview of lessons learned in material substitution. He pointed out that all technologies must meet "requirements". However the term may mean different things in different situations.

- For new technologies for which there are no established specifications, the performance requirements are defined by the new material capabilities (e.g. composites for B787), just as the requirements for most legacy materials are dictated by the capabilities of high-strength steel, Cd plate, hard chrome plate, etc.
- However, requirements may be met or exceeded by materials currently in use. When changes are driven by new regulations, it is important to understand what the technical requirements really are
    - Is the real requirement the spec limit, which is often the lowest acceptable performance?
    - Or is the requirement equal to or better than the performance of the existing material?
        - In this case, if the performance of the substitute falls below the current material, it may cause downstream failures, especially when the minimum spec requirements are barely adequate and designs rely on the performance level of existing materials.

If there is no problem with existing materials, there is no incentive for an engineer to accept changes. Joe's recommendations were to solve a problem on one component, and use the experience gained there to drive broader acceptance.

Craig Matzdorf noted that although non-chromate surface treatments and primers are improving, many applications still require Cr^{6+}, and NAVAIR has designed a waiver process to meet this need as required by the hexavalent chrome memo. The Metalast non-chromate anodizing process has been validated, and in fact FRC-SE and NAVAIR have been finding better SO_2 salt fog performance with trivalent sealers than with chromated sealers on anodized 2024 and 7075 aluminum. Non-chrome primers are improving, although their performance is not yet equal to chromated primers. Resin systems still need Improvement; MIL-DTL-23377 high-solids "solvent-borne" primers provide superior protection, but MIL-DTL-85582 "water-borne" primers have better application characteristics. Craig pointed out that many people have the misconception that water-borne primers contain fewer volatiles than solvent-borne, whereas in both cases the spec permits up to 340 g/L.

3.3. Light metal protection and coating repair

The Tagnite process has proved to be very effective for magnesium gearboxes. Until recently it has only been possible to apply it to new gearboxes since steel inserts are incompatible with the process. However Tagnite has now developed the capability to apply the process as an overhaul method for gearboxes that still contain steel inserts and threads. The method is being used to refurbish very old magnesium components from the B-52 and KC-135.

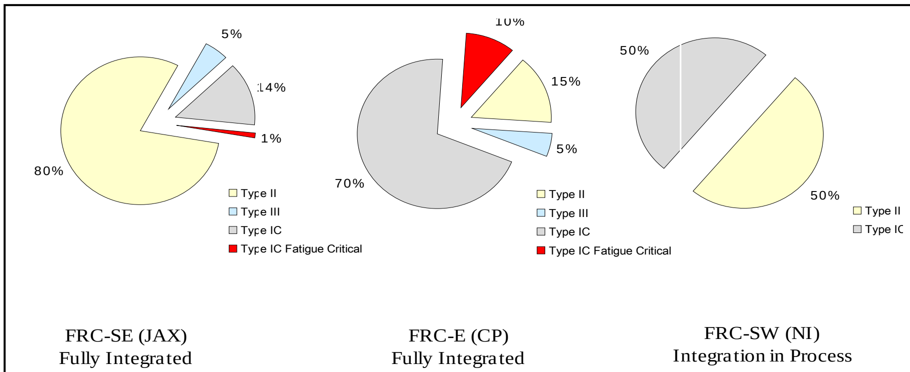

Figure 5. Adoption of advanced process control for anodization of Al (Matzdorf/Prado).

Both FRC-SE and FRC-E have adopted an advanced process for control of aluminum anodization (see Figure 5), while FRC-SW is in the process of doing so. The process eliminates Cr^{6+}, while improving process quality and reducing energy usage.

Cold spray aluminum and aluminum alloys are proving to be an excellent way of refurbishing magnesium gearboxes, as well as repairing IVD and AlumiPlate coatings. Boeing has qualified the use brush ZnNi for Cd plating repair, and a portable cold spray system for repair of MIL-DTL-83488 pure Al coatings, providing a way to repair coated components at the depot or operational level. ARL has validated high velocity cold spray for repair of magnesium gearboxes under an ESTCP program. With this method it appears to be possible to reclaim some of the structural strength of the material, a capability that other coating methods do not provide.

In Europe brush plated Ni followed by brush plated ZnNi (for galvanic compatibility) has proved to be an effective way of reclaiming wing slats whose leading edges have been damaged by pitting corrosion, creating a surface that is more resistant to damage than the original material (see Figure 6). The equipment used for this type of repair is compact and designed to prevent the leakage and run-down commonly associated with brush plating repairs.

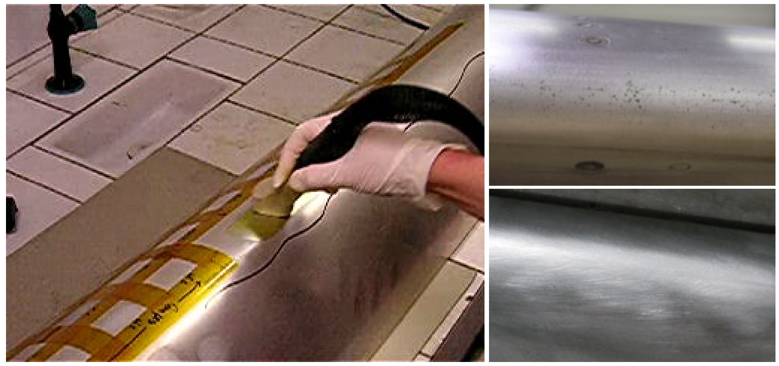

Figure 6. Brush plating repair of wing flap (left); pitted flap (top right); flap repaired with brush Ni+ZnNi (bottom right).

3.4. Cd Alternatives

3.4.1. Structural components

A major advance has been with the qualification of low hydrogen embrittlement (LHE) ZnNi as an alternative to cadmium for landing gear at Ogden ALC. The process qualified at Ogden uses the Dipsol IZ C-17+ ZnNi plating chemistry with a Cr^{3+} sealer in place of chromate conversion. The coating, deposited in a 325 gal prototype tank, successfully passed all of the qualification tests (Table 2), with performance equal to or better than Cd plating in all cases:

- Requirements for Non Destructive Inspection (NDI) were validated to work without removal of the plating: FPI, Ultrasonic, Eddy current, X-ray
- A trivalent sealer was optimized and used in place of a chromate conversion coating
- Brush plated ZnNi was developed for repair of damaged LHE Zn-Ni coatings (Touch Up)
- An accelerated hydrogen embrittlement test procedure was developed.

Table 2. Qualification tests passed by LHE ZnNi (Hill AFB).

Adhesion Primer adhesion	Embrittlement: ➢ Hydrogen Embrittlement ➢ Environmental embrittlement ➢ Liquid and Solid Metal Embrittlement
Fatigue Torque Tension	Corrosion: ➢ B117 salt fog ➢ G85 SO_2 salt fog

Ogden plans to install a production line and implement LHE ZnNi in place of Cd plating throughout the depot.

Interest has been growing over the last three years in the use of ionic liquids for electrodeposition. Dipsol of Japan is developing an ionic liquid electroplating chemistry for deposition of Al, Al-Zr and Al-Zr-Mn's coatings. Although this approach does obviate the requirement for a toluene bath, it nevertheless must be done in a nitrogen-purged atmosphere to eliminate oxygen and water.

3.4.2. Electronics

Electrical connectors remain an important use of Cd plating, while aluminum electrical equipment cabinets remain a particularly difficult problem for eliminating chromate conversion since they must remain unpainted.

The NASA TEERM project team held a side meeting at the workshop and reported on their progress in validating chromate conversion alternatives for aluminum electrical cabinets.

- Alodine 1600 (Baseline)
- Metalast HF
- Metalast HF/EPA

- SurTec 650
- SurTec 650 C
- Alodine T 5900 RTU
- Iridite NCP

The project began in 2010 and atmospheric corrosion testing is getting underway.

Radiall of France reported on their difficulties in finding Cd and chromate alternatives that would meet the requirements for the new finishes required in the MIL-DTL-38999 spec that was recently updated. They evaluated electrodeposited Al, ZnNi and electroless Ni-PTFE, not just for their corrosion resistance, but for meeting all the other requirements of the specification. They settled on high phosphorus electroless nickel-PTFE as the finish that best met the spec on their products. Note that other manufacturers have also adopted the same finish, but Radiall found that it was important to specify a high phosphorus electroless nickel, which generally has better as-deposited corrosion resistance.

3.5. Hard chrome plating replacement

FRC-SE is validating nanophase Co-P (nCo-P, known commercially as Nanovate CR), as a hard chrome replacement under an ESTCP project. The Joint Test Protocol has been developed and the test program is about to begin. nCo-P was developed under a SERDP project, and a significant amount of data has already been obtained. It is interesting to note that the hardness of this coating rises with bake time during hydrogen baking, while the hardness of hard chrome falls (Figure 7). The result is that, although nCo-P remains a little softer than EHC, the difference in hardness (and hence the expected difference in wear resistance) is quite small.

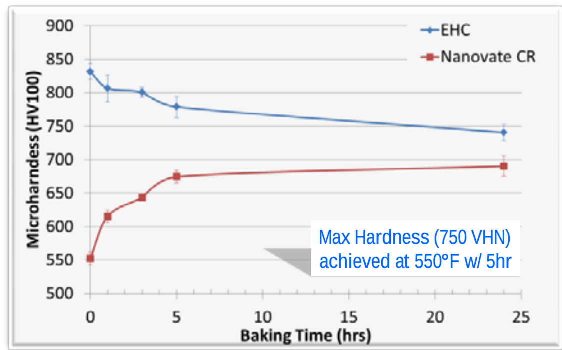

Figure 7. Hardness of nCo-P vs EHC as a function of time during a 375°F hydrogen bake.

Two very different technologies were reported that demonstrated performance superior to hard chrome for gun barrels, an application where previous coatings had always failed. One of these was an explosively clad Ta-W liner (shown in Figure 8), and the other an electromagnetically enhanced physical vapor deposited coating of a similar alloy. In both cases the new coatings showed very much lower wear than hard chrome in firing tests. (The explosive cladding project received the SERDP 2010 Project of the Year Award.)

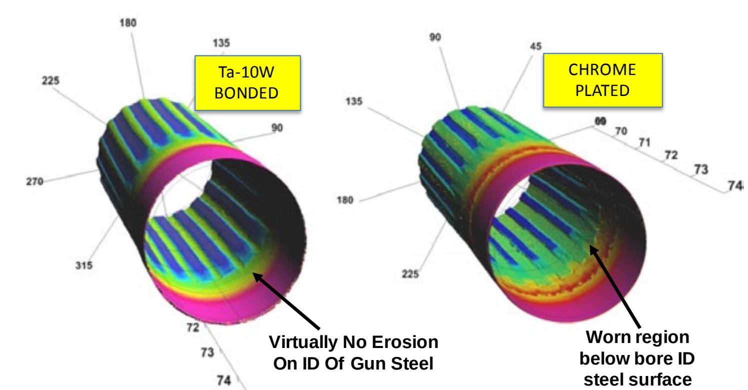

Figure 8. Laser measured wear of 25 mm Bushmaster barrel after firing tests of explosively clad gun barrel.

3.6. Paints and primers

Craig Matzdorf reported that, while non-chrome paint systems are still showing somewhat inferior corrosion results compared to chromated systems, non-chrome (Class N) systems are improving and becoming closer to chromated primer performance.

ESTCP has funded a number of non-chrome primer projects. Rather than continuing to fund individual projects, they have now funded an overarching initiative, to be run by Craig Matzdorf and Julia Russell of NAVAIR. This program is intended to evaluate non-chrome primers currently on the market and to develop test protocols for qualifying new coatings. Its purpose is to bring alternatives into production as soon as possible for low risk applications, while developing the data and experience needed for more difficult situations.

A great deal of progress is being made in electrocoat (e-coat) and powder coat alternatives to liquid painting.

Low-temperature cure powdercoats have been validated that cure at temperatures <300°F. Ultraviolet curable coatings have been demonstrated for use on aircraft, and a robotic curing system has been developed for use on large area UV cure coatings. E-coating, which is widely used for commercial vehicles and for some military vehicles, is now being evaluated for use on aircraft.

3.7. Accelerated test methods

The pace of change demanded by environmental regulations such as REACH requires the adoption of new coatings in a very short time compared to the typical 20 year adoption cycle for most aerospace and defense materials. The old accelerated test methods, such as ASTM B117, are known to be poor predictors of in-service corrosion performance, but are nevertheless used almost exclusively for that very purpose simply because there are no good alternatives for accelerated corrosion testing. NAVAIR has been developing an accelerated test protocol using a combination of beach exposure and cabinet testing. An ongoing SERDP project is evaluating the role of humidity in accelerated corrosion test methods.

Another critical test that has never been well defined is environmental embrittlement, for which the current test specification is ASTM F519. Under SERDP funding ARL and Boeing are using a Design of Experiment approach to improve and validate the test procedures in conjunction with the ASTM F07.04 committee.

3.8. Other issues

3.8.1. Coating removal

Both laser stripping and pulsed waterjet stripping are proving viable ways of removing coatings. While laser stripping has now been validated for paint removal, pulse waterjet is capable of removing HVOF coatings, which have previously been strippable only by the use of chemical solutions. A new technique that is showing some promise, and is being evaluated by FRC-E, is atmospheric plasma stripping. Like laser stripping this is a small area technique, whereas waterjet is a large area method.

3.8.2. Microbially influenced corrosion

Although we tend to think of corrosion as being purely due to water, salt, galvanic coupling, etc., there is evidence that corrosion can be accelerated by microbial and fungal attack. It is quite common (e.g. in F-16 cockpits) for water to accumulate on organic surfaces, including paints and insulation, leading to the growth of microbes and fungi. Their waste products can be acidic and lead to corrosion with the characteristic tunneling morphology. This type of attack is inhibited by chromated primers, and while some non-chromated primers also appear to inhibit this type of corrosion, not all do so. As more non-chrome finishing systems are being adopted, this is an issue that we should be considered.