

# Corrosion resistant films from trivalent chrome based solutions applied to electrodeposited zinc and zinc alloys.

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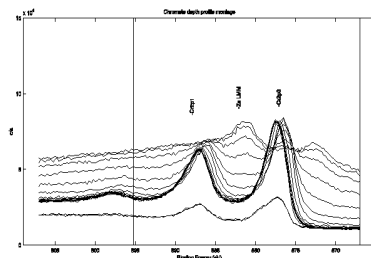
## INTRODUCTION

As early as 1951<sup>1</sup> inventors developed trivalent chrome based passivation treatments for zinc (C3P). These C3P solutions were an alternative to existing hexavalent chromium (CrVI) chromate treatments that were classified based upon characteristic film colors: clear or 'blue', yellow, olive drab, and black<sup>2</sup>. Although early C3P eliminated hexavalent chromium they did not gain commercial acceptance until the late 1970's as a niche process. The process produced a thin, ~40nm based upon XPS, clear film with modest corrosion resistance expectation<sup>3,4</sup> formed from solutions of inorganic CrIII compounds. Pragmatic as well as environmental concerns led to acceptance. C3P minimized blemishes due to reaction of CrVI and small amounts of codeposited iron common in, the then emerging, acid and non-cyanide alkaline zinc processes (CFZP).

With the acceptance of CrIII derived films as clear passivates a large variety of processes were patented, marketed, or otherwise described to improve corrosion protection. Heated and/or high [CrIII] solutions were described in 1982<sup>5</sup> that claimed thick (~200nm) films from solely inorganic solutions. The use of di and tricarboxylic acids as well as cobalt and rare earths was described beginning in 1983<sup>6,7,8</sup>. Iridescent C3P for zinc nickel<sup>9</sup> and black C3P on alloys of zinc iron with iron levels as low as 20 ppm, and zinc nickel (5-25%) range were developed in the early 1990's<sup>10</sup>.

Reviews<sup>11,12,13</sup> have been published that compare C3P passivation with chromating and indicated that a shared model for film formation, based upon early CrVI research<sup>14</sup> had achieved a consensus. This model describes film formation on the basis of: 1. Localized rise in pH near the surface as protons are reduced to hydrogen gas and zinc dissolves. 2. Precipitation of CrIII and other metal ions, if present, that would bridge or olate through oxygen and oxyanions in the higher pH region. Additional understanding of passivation based upon structural variation of the

Figure 1 XPS depth profile montage of C3P region illustrating the Zn Auger interference with Al X-ray excitation.



Zinc or zinc alloy has been suggested for ZnNi<sup>15</sup> as well as zinc<sup>16</sup>.

Post treatment for passivates and chromates are often recommended dependent upon final application. Described as seals, if the thickness is

less than one or two microns, as a topcoat if greater than 4 microns.

**Table 1 Summary of production scale C3P testing. Trials completed/failed (in progress). \*due to insufficient thickness (3-5 microns).**

	Organic Seal	Inorganic Seal	Top Coated	Totals
<b>Organic CrIII</b>	Zn	ZnFe 5/0	Zn 2/0	29/0(5)
	11/0(4)		ZnFe	
	ZnFe		2/0	
	4/0(1)		ZnNi	
	ZnNi 3/0		2/0	
<b>Inorganic CrIII wo Co</b>	ZnFe	ZnFe 3/0	ZnFe	21/1*(4)
	3/0	ZnNi	ZnNi	4/1*
	6/0(2)		ZnNi	5/0(2)
<b>Inorganic CrIII w Co</b>	Zn (3)			(3)
<b>Phosphate CrIII</b>	Zn	ZnFe		4(6)
	(trace Fe)	1/0(2)		
	ZnFe			
	3/0	ZnNi(2)		
<b>Total</b>				54/1(18)

For fasteners, layers may have specialized tribology characteristics to ensure uniform torque and clamp load for joints assembled with automatic machinery. The nomenclature's of these coatings are dry film lubricants (DFL), usually without additional corrosion protection, or integral lubricants (IL), if combined with a seal or topcoat. Seals, as well as topcoats, can greatly increase the reliability of CrIII passivates<sup>15</sup>.

Despite the strides of C3P, widespread replacement of CrVI did not occur until recently. Factors including costs of materials and qualification, final appearance, additional processing steps, alloy, and energy requirements did not compete well against chromate applied to non-deliberately alloyed zinc. Many

CrVI advantages could be met with CrIII, but not all<sup>16</sup>. In the recent past, this reluctance to replace CrVI has changed due to the European Union adopted End of Life

Vehicle (ELV). This initiative severely restricts the amount of CrVI, as well as eliminating other toxic materials, in motor vehicles. With enthusiasm came realization that a very long list of C3P as well as non-chrome methods needed to be evaluated. It has taken an immense ongoing international effort by OEM's, applicators and suppliers to sort through the offerings.

What C3P's have survived? At this time a universal CrIII or chrome free passivation chemistry that can be applied to all zinc and zinc alloys, regardless of performance requirements or anticipated post treatment, has not definitively emerged. Organic acid based C3P with cobalt and subsequent organic seal has met or exceeded nearly all requirements but has modest issues with operating costs, waste treatment, longevity, compatibility with dip spin and electrophoretic topcoats. Engineering solutions are evolving to eliminate these concerns but other C3P have achieved success in a variety of applications. Inorganic C3P, with and without cobalt, achieves many of the lower corrosion requirements on zinc and performs as well or better than organic C3P on some zinc alloys. Phosphate based C3P is effective for blackening ZnFe and ZnNi as well as zinc that is inadvertently alloyed with trace iron. At present, sealing of these coatings is necessary for reliable corrosion protection. Based upon application, desired final appearance, and performance different substrates employ require different passivation methods to achieve cost effectiveness.

In this paper we will summarize our morphological, compositional, and structural characterization studies of C3P

amps per liter of accumulated testing. Standard steel and brass (used to determine iron concentration) Hull cell panels were used as base metal substrate without modification, except for certain XRPD measurements where an

**Table 2 Summary of C3P characterization tests.**

	Morphology	Thickness (nm)	[CrVI] ug/m <sup>2</sup>	(dpc Application	Other
<b>Organic CrIII w Co</b>	Gen. smooth, occ. cracks	Zn 100-400	<0.1	Std Zn yellow alternative, generally with seal, 'clear' alloy.	ZnO in film., no spinel. Co reduces heat variation
<b>Inorganic CrIII wo Co</b>	ibid.	Alloy 70-200 function of alloy	<0.02 or nd	Alloy tie coat. With ZnFe and seal. With ZnNi as clear.	Ibid. Alloy sensitive ([Fe], [Ni]).
<b>Inorganic CrIII w Co</b>	ibid.	Alloy 70-200 function of [Co] and dense?		Lower corrosion req'ts (blue). Improved corrosion resistance with seal.	Low T app.
<b>Phosphate CrIII</b>	Rough, dense crack pattern	Zn trace Fe 40-80 nm. ZnFe 80-200 nm	<0.1	Black coatings.	Low T app.
<b>Organic Seal</b>	Low rms, inorganic nano particles	800-1500	0	Improve corrosion resistance. Can be integrated with lubricant	
<b>Inorganic Seal</b>			0	Interiors, when low cost needed, and with fluid compatibility issues.	

coatings and seals that have met or exceeded the expectations of recent production scale screening. We will not characterize the C3P used for modest corrosion protection. They have been widely accepted and previously reviewed.

**EXPERIMENTAL:** Testing was done at laboratory and production scale. At the laboratory scale, commercially available<sup>17</sup> electrodeposits of zinc, zinc nickel (12-15%), and zinc iron were used. With few exceptions, noted in the results section, these solutions were from pilot line process tanks that had over 10

'amorphous' electroless nickel coating<sup>18</sup> was applied prior to zinc or zinc alloy deposition due to concern about the rolling texture present with sheet steel. Plating was solely direct current and done in two liter beakers at uniform 20 ASF current density until thickness of 8 microns was achieved. The panels were treated with CrIII based passivates, described as organic CrIII with Co, inorganic CrIII, inorganic CrIII with Co, and phosphate based CrIII. In many cases CrIII derived film was subsequently treated with either an organic or inorganic seal. These CrIII processes and seals are also commercially available processes<sup>19</sup>. The

resulting panels were used for analytical measurements

For production scale testing, the zinc or zinc alloy, passivate and seal were applied in a continuous sequence by commercial applicators. For the dip spin and electrophoretic paint tests the resulting plated and passivated parts were then transported to either the vendor of the paint who performed or arranged the topcoat application. All paints used in the study are available from major suppliers and chrome free as verified by EDS.

Hexavalent yellow chromate controls were produced from solution as described in reference 11. Other chemicals were obtained from Fisher Scientific.

Instrumentation includes a Hitachi S4500 cold field emission (CFE) SEM with Noran EDS, PHI 5600 XPS (ESCA) System using monochromatic Al X-ray source and Argon ion beam for depth profiling and charge neutralization with an XPS beam diameter of ~1 mm, Scintag X1 XRPD equipped for grazing incidence X-ray analysis (GIXA), Bruker D8 4 circle XRPD, NEC 5SDH Tandem Pelletron Ion Beam Accelerator for RBS and PIXE, and a Digital Instruments Nanoscope III.

With XPS the strong Cr2p region is not particularly sensitive to oxidation state and is in the same region as a zinc Auger line with Al X-ray source (figure 1). Consequently, the interference free but weaker Cr3p and Cr3s regions were used to examine oxidation state and chrome concentration (figure 2). Aligning XPS spectra with the Zn2p or O1s did not effect the coincidental locations and peak symmetry of any chrome regions. The Cr3S region appears to have an undocumented peak, possibly another Zn auger, approximately 3eV from the CrIII 3s

which interferes with CrVI 3s. The combination of Cr3S and Cr3p appears to be a reliable indicator of CrVI presence based upon additional experiments with CrVI and CrIII prepared by drying aqueous mixtures of potassium dichromate and chrome chloride (figure 3). Complicating XPS quantification is the observation that CrVI signal intensity dropped after argon ion sputtering.

Diphenylcarbazine (DPC) testing<sup>13</sup> was used to determine trace presence or absence of CrVI for comparison to XPS.

Neutral salt spray (NSS) testing was conducted in accordance with ASTM B117 and all panels and parts were heated as noted, then stored at room temperature for 24 hours, prior to being placed in NSS.

ZnNi alloys produce an unusual corrosion product in NSS testing referred to as gray veil. This is not a voluminous corrosion and, unlike white corrosion products, it is not visible if a part is wetted with deionized water or removed if the surface is rubbed with a tool such as a q-tip. The appearance of gray veil in C3P on ZnNi does not correlate with corrosion failure such as white corrosion or red rust.

## RESULTS

### Corrosion studies:

Production scale corrosion studies have been performed at the request of umbrella organizations such as the German VDA, automotive OEM's and their tier one suppliers. Requirements or expectations

Table 3 XPS summary of C3P compositions (atom%).

SYSTEM	C1s	N1s	O1s	F1s	S2p	Cl2p	Cr3p	Co2p3	Ni2p3	Zn2p3	O:Cr	Cr:Zn
Zn Organic CrIII non annealed	27.76	-	52.38	-	-	-	11.60	0.70	-	7.57	4.52	1.53
Zn Organic CrIII annealed	27.13	1.77	50.25	-	-	1.69	11.34	0.57	-	7.24	4.43	1.57
Zn Inorganic CrIII + Co RT	2.50	1.33	58.00	3.42	2.13	-	21.67	0.02	-	10.84	2.68	2.00
ZnFe Organic CrIII	20.90	-	47.82	-	-	-	22.14	0.47	-	8.68	2.16	2.55
ZnFe Inorganic CrIII	1.10	-	49.00	4.21	-	0.75	28.00	-	-	16.00	1.75	1.75
ZnFe Inorganic CrIII RT	1.27	-	46.81	4.82	0.00	0.69	27.12	-	-	19.29	1.73	1.41
ZnNi Organic CrIII	16.23	-	50.00	-	-	-	19.00	1.66	0.12	14.00	2.63	1.36
ZnNi Inorganic CrIII	0.80	-	48.24	5.39	-	-	27.89	-	0.10	18.48	1.73	1.51
<b>Mean</b>			50.20				20.70			12.78	2.70	1.71
<b>Standard Deviation</b>			5.83				6.24			4.87	1.16	0.40

have varied. The results of the testing, in many cases, are considered confidential except for individual participants final results. In other cases detailed reports are being prepared by requesting organizations. In a few cases, the exact process variables and application (e.g. type of part) are to be kept confidential.

Within the scope of these requests we have attempted to summarize how recommended processes have fared in production scale tests by describing the corrosion results only (not tribology or other issues) in terms of met or exceeded expectation, and failed to meet expectations. These tabulated results are presented in figure 1. The majority of the completed tests are from European trials, many of the in progress tests are from North America.

We anticipate even more production trials. The results indicate that with well-defined systems C3P is successfully implemented.

#### Film morphologies:

Figures 4 and 5 contrasts the various morphologies present on

chromate and passivate film surfaces. With the exception of the phosphate containing trivalent used for blackening zinc with iron concentrations ranging from 20 ppm to greater than 1% (w/w), the C3P films did not have crack patterns similar to the yellow chromate control. In some cases the C3P had a broken glass appearance or often no cracks vs. the mud crack appearance of thick chromates. When organic seal is applied to the phosphate black the surface becomes devoid of cracks.

#### Film Thicknesses:

Figures 6 and 7 are typical photomicrographs of fractured coatings used to determine thickness. Figure 4 has AFM images and z-axis data used for thickness and rms. Figure 2 illustrates why and how XPS was used to determine thickness by ignoring Cr2p signal or by recording Cr3s or Cr3p. AFM (figure 4) of the control chromate cracks was able to measure the depth of the cracks when present<sup>20</sup>. C3P passivates such crack patterns are unusual. The AFM crack depth measurement as well as the fracture

study of CrVI yellow concur in thickness but the Ta<sub>2</sub>O<sub>5</sub> calibrated Argon ion beam indicate approximately half the thickness of those methods. The Ta<sub>2</sub>O<sub>5</sub> standard was checked for accuracy by fracture and by Rutherford back scattering. As a result the XPS ion beam etch rate is approximately double with chromate or passivate compared to Ta<sub>2</sub>O<sub>5</sub>. Table 2 compares the thicknesses of various combinations of zinc and zinc alloys, passivates, and seals with other characterization parameters.

The fracture method is easily applied to coupons but is very difficult to reliably apply to barrel plated fasteners. Preliminary data of C3P on Zn applied in rotating barrels provides data of 25nm by XPS (Ta<sub>2</sub>O<sub>3</sub> cal.) and 40 nm by fracture study. The organic seal thickness appeared to decrease from 1000nm in rack to 100-200nm in barrel. Inorganic C3P seems to be more tolerant of the barrel process and film thickness, in limited experiments, of 50 nm has been observed. There are variations in applying passivate films in batch mode. Some applicators remove plated parts and use baskets for applying passivate and seal. Others use the plating barrel, which can vary in diameter, velocity, load, and perforation size.

#### **Film and substrate compositions:**

In none of the examined samples were peaks consistent with CrVI found in both the Cr3S and Cr3p regions. Quantification by XPS is difficult due to interaction of the Argon ion beam with CrVI. DPC is likely a more reliable quantitative method.

The Cr:Zn ratios did not vary greatly regardless of type of passivate, substrate, or application temperature. O:Cr for organic C3P is greater than with inorganic C3P indicating the organic acids complexing CrIII have

Precipitated within the organic C3P film with CrIII. The organic CrIII on ZnNi film had greater concentrations of Ni than did the inorganic based films. Likely due to the organic acids being able to solubilize then precipitate Ni during film formation.

#### **Structure of films and substrates:**

Thickness nor composition appear to be the most significant factor for corrosion resistance the structure of the passivate film must be investigated.

Accordingly passivate films were analyzed by GIXA which failed to produce evidence for any crystalline species other than ZnO (figure 10). However, XRPD of the substrates reveals variation in crystallography as a function of alloy and additive.(figure 11). The expected difference with ZnNi as 'gamma' phase specific was evident but ZnNi alloys with nearly identical composition can vary their texture. The bright ZnNi with a preferred 600 peak, used for most tests, is distinct from the more bendable and matte appearing ZnNi.

XRPD reveals variations with zinc as a function of additive, and modest iron content. Suggesting that the structural variations of the substrate play the important role suggested by Ramanauskas<sup>16</sup>. We are continuing to explore the role of structure on passivation and corrosion resistance.

#### **Effect of codeposited iron:**

Deliberate ZnFe alloys are blackened by phosphate CrIII but low concentrations of iron can still be blackened<sup>10</sup>. To determine if inadvertent zinc iron alloy could be blackened by phosphate CrIII fresh zinc electrolytes were prepared and brass hull cell panels

plated then immersed into phosphate CrIII. The resulting appearance was gray. Fresh solutions were again prepared but ferrous sulfate was added to achieve concentrations of 100, 200, 400 ppm Fe in the plating solution. After each addition a panel was plated then passivated in phosphate CrIII. At 200 and 400 ppm Fe in solution blackening was observed.

PIXE analysis of the deposits revealed codeposited iron present at concentrations of 30-50 ppm.

Currently we are examining zinc solutions submitted for routine analysis to see if they blacken with phosphate CrIII. Many have blackened. The panels are being retained for future PIXE analysis.

With deliberate ZnFe alloy what is the effect of iron concentration in the alloy on the performance of different types of passivates? Fresh pilot line ZnFe solution was prepared and iron concentration sequentially increased to allow deposits with incremental iron content. Panels were then plated and passivated with organic C3P and inorganic C3P, and then subjected to NSS where 5% white rust was recorded. The organic C3P responded positively to iron presence at concentrations of 0.1%. The inorganic CrIII does not respond positively to iron until the concentration reaches 0.5% (figure 9).

#### **Effect of cobalt in C3P:**

Cobalt has been used for quite some time to help make C3P passivate films bluer. There were persistent claims about increased corrosion protection. With thin C3P film it is difficult to establish a corrosion resistance relationship. Cobalt

demonstrates a positive effect on corrosion resistance of thicker C3P films (figure 12).

**CONCLUSIONS:** Trivalent passivation, with and without seals and topcoats, has met or exceeded expectations in a large number of production trials.

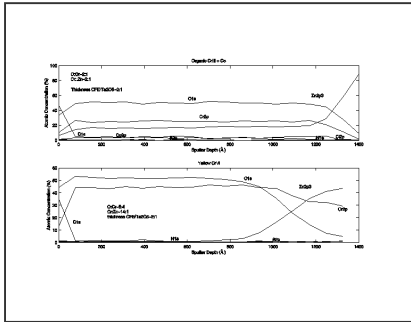
Passivate morphology of films that have achieved expectations tend to be more uniform, less colorful, and have less cracking than CrVI chromate films. Surface continuity likely plays a role in corrosion protection.

The easy prediction of performance based upon visual observation, possible with chromate, is no longer germane.

Thicknesses of trivalent passivates vary based upon type of passivate, the composition of the zinc or zinc alloy substrate, and process parameters. Passivate thickness does not always correlate with corrosion resistance or good coverage by dip spin or electrophoretic topcoat, particularly when applied to alloys. In the future we will attempt to study film thickness as a function of batch processing variables using focused ion beam (FIB) FE SEM.

Trivalent film compositions generally included significant amounts of zinc and, if present in the trivalent solution, cobalt. XPS did not confirm CrVI presence in C3P. DPC testing did not find frequent CrVI presence or concentrations exceeding ELV requirements. The poor sensitivity of XPS Cr3S and C3P regions, and the possible reduction of CrVI by argon ion beam make quantification of CrVI by XPS difficult. Examination of coupons

Figure 2. XPS depth profile of yellow CrVI (Cr2P) and organic CrIII (Cr3p). CFE thickness is ~ twice that of the Ta205 sputter rate.



with angle dependent XPS (ADXPS), incorporation of principle components analysis, use of alternative x-ray source, and other methods will be pursued in an effort to obtain spectrographic confirmation of CrVI presence.

GIXA XRPD of films found diffraction patterns that are consistent with zinc oxide within the films. No evidence for other crystalline compounds, such as spinels, was found. Continued work to attempt quantification of the amount of crystallinity is recommended.

The textures of the substrates varied greatly. Continued study of the role of texture in passivation is warranted.

Blackening of ‘zinc’ using phosphate based CrIII is likely due to inadvertently codeposited iron present at concentrations greater than 20ppm. However, corrosion benefits from ZnFe occur at higher concentration ranges that are dependent upon type of passivate film.

What is most important in achieving corrosion resistance with CrIII passivation: substrate, type of passivate, film morphology, thickness,

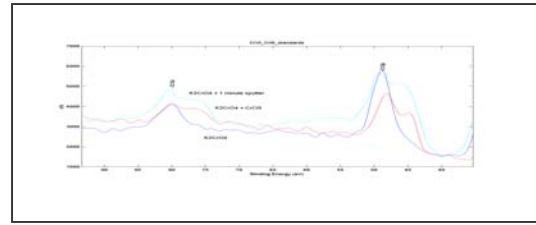


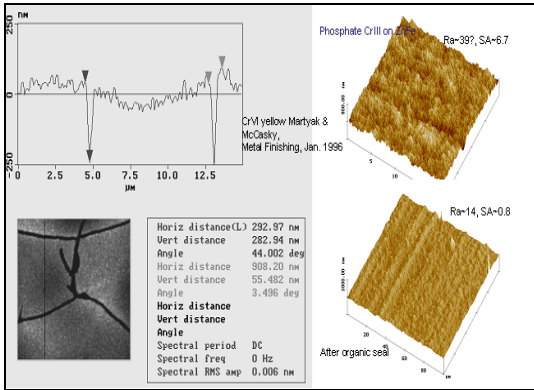
Figure 3. Cr3P, Cr3s XPS of K2Cr04 and CrC13 films.

composition, or structure? The relationships between these factors have not yielded a definitive relationship other than ZnNi (12-15%) has exceeded all corrosion expectations. For other systems, for the time being, CrIII passivation is a system where the performance relies upon the component layers and should be treated as such.

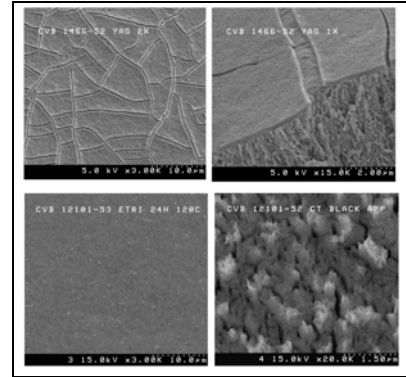
Assuming that a system component (passivate, seal, substrate, or process parameter) that has achieved good performance in one system can be substituted into another system, is unwarranted without extensive validation. Film thickness is a factor influencing corrosion protection. C3P on alloys is generally thinner than the C3P on Zn and the corrosion resistance of alloy-based systems, particularly ZnNi, is greater than for zinc.



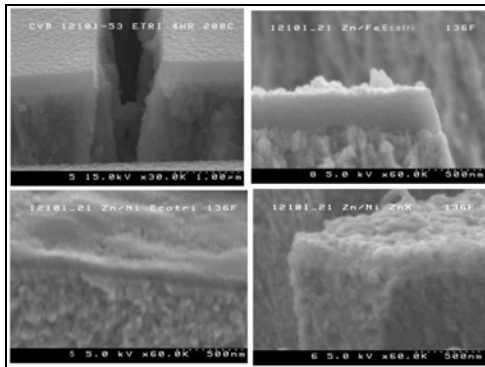
**Figure 4. Representative AFM data for CrVI yellow (left) and phosphate based CrIII on ZnFe (right).**



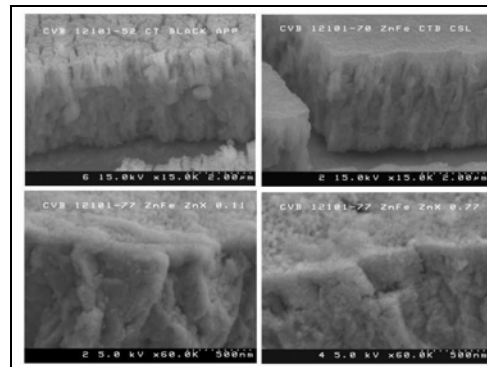
**Figure 5. Clockwise from upper left: chromate, chromate cracks, phosphate CrIII on ZnFe (0.5%), organic CrIII on zinc.**



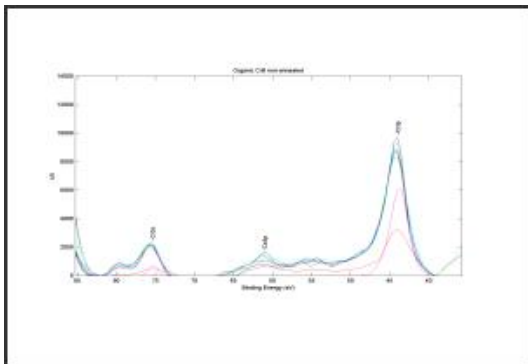
**Figure 6. Representative fracture studies clockwise from upper left: organic CrIII on zinc, organic CrIII on ZnFe, inorganic CrIII on ZnNi, inorganic CrIII on ZnNi. The application temperature is 137°F.**



**Figure 7. Clockwise from upper left, phosphate CrIII on ZnFe, treated with organic seal, inorganic CrIII on ZnFe 0.8% Fe, inorganic CrIII on ZnFe 0.1%.**



**Figure 8. XPS depth profile montage of Cr3P and Cr3s from organic C3P.**



**Figure 9. Typical GIXA XRPD of Cr-III passivated zinc or zinc alloy. Peaks are consistent with zinc oxide(s).**

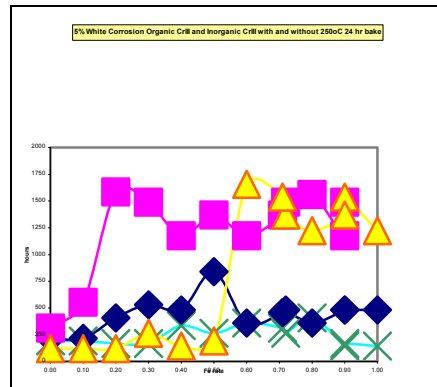


Figure 10. Typical GIXA XRPD of CrIII passivated zinc or zinc alloy. Peaks are consistently with zinc oxide (s).

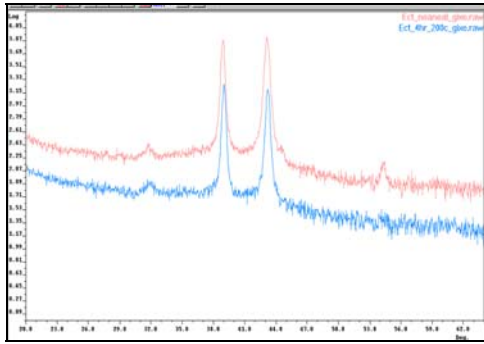


Figure 11. XRD scans of ED Zn (2), ZnFe, and ZnNi(2). Zinc hkl are vertical, ZnNi hkl at an angle.

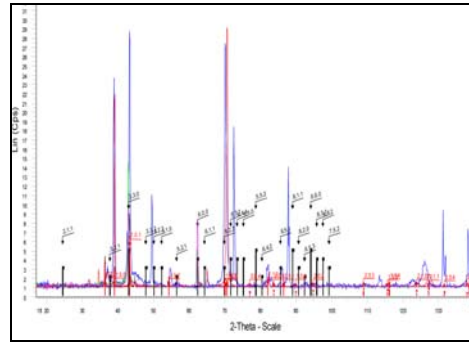
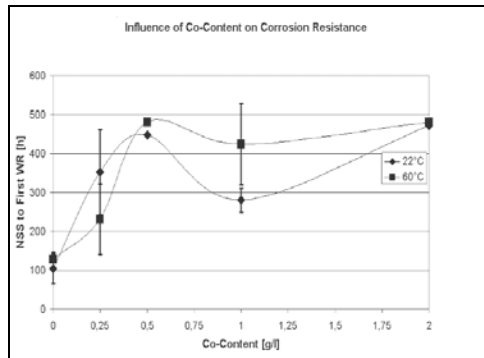


Figure 12. Effect of [Co] on C3P.



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- <sup>1</sup> D. Johnson, US Patent 2,559,878.
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- <sup>3</sup> C.V. Bishop, et al. US Patent 4,171,231
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- <sup>6</sup> R.J. Huvar. USP 4,349,392
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- <sup>10</sup> C.V. Bishop, et. al. USP 5,415,702
- <sup>11</sup> Bishop et al. "Eine mögliche Struktur für verschiedene Chromatierungsschichten auf elektroytisch abgeschiedenen Zinküberzügen" *Galvanotechnik D* 7968, **71** (1980)
- <sup>12</sup> D. Crotty, "Trivalent Chromium Conversion Coatings", AES Annual Technical Conference Proceedings, San Francisco, CA June, 1982.
- <sup>13</sup> C. Andrlé, et al. "Cr(VI)-Free Post-Treatment Processes for Zinc and Zinc Alloys", AESF Week 2001, Orlando, FL
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- <sup>15</sup> P. Upton "The Effect of Sealers on Increase of corrosion Resistance of Chromate-free Passivates on Zinc and Zinc Alloys"., *Plating and Surface Finishing*, Feb., 2001, 68-72.
- <sup>16</sup> C.V. Bishop & J.R. Kochilla "Non-Hexavalent Chrome Passivation Technologies for Zinc and Zinc Alloys"; AESF SUR/FIN 2000 June 2000.
- <sup>17</sup> Acid zinc = Zylite 1150, NCA zinc=Protolux 3000, NCA ZnFe=Protedur Plus, NCA ZnNi=Reflectalloy® ZnA(12-15), all from Atotech.
- <sup>18</sup> Nichem® 2000 from Atotech
- <sup>19</sup> Organic CrIII=EcoTri®, Inorganic CrIII=Rodip® ZnX, Inorganic CrIII with Co=CorroTriBlue Extreme, phosphate based CrIII=CorroTriBlack ZnFe, inorganic seal=Supreme Seal 500, organic seal=Corrosil CFSR (+501), all from Atotech.
- <sup>20</sup> Martyak, et al. "Surface Structures of Zinc Chromate Coatings", *Metal Finishing*, January 1996, 20-26.