Functional Trivalent Chromium Electroplating

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This paper will discuss recent research work on the development of a functional trivalent chromium plating process from a single, simple-to-control trivalent-based electrolyte to replace hexavalent chromium plating. Hexavalent chromium plating has been used for many years to provide hard, durable coatings with excellent wear and corrosion resistance properties. However, hexavalent chromium baths have come under increasing scrutiny due to the toxic nature of the bath, effects on the environment and worker health. In this work, we are updating accomplishments to achieve properties comparable to existing hexavalent chromium plating for functional applications. Work on achieving desirable thickness, uniformity, adhesion, porosity and corrosion resistance, as well as other material properties, will be discussed.

Keywords: Functional chromium plating, hexavalent chromium alternatives, trivalent chromium plating, pulse plating, electrically-mediated current waveforms,

Introduction

The US EPA has identified chromium as one of 17 "high-priority" toxic chemicals. The US EPA selected the high-priority chemicals based on their known health and environmental effects, production volume and potential for exposure.[1] Under former US EPA administrator William K. Reilly's Industrial Toxic Program, the high-priority toxic chemicals were targeted for 50% reduction by 1995.[2] Additionally, worker exposure to Cr(VI) plating baths is regulated by OSHA, exhaust/scrubber systems must be installed for Cr(VI) plating operations and the exposure limit is 0.01 mg/m^3.[3] The Clean Air Act, as well as local constraints, regulates the emission of chromium to the air and water. Since Cr(VI) plating produces hazardous air emissions, all Cr(VI) platers must control and monitor the bath surface tension and report the results to the EPA. In contrast, less hazardous trivalent chromium [Cr(III)] platers are not required to monitor bath surface tension.[3]

The chemistry of chromium provides a basis for understanding the toxicology. Chromium can exist in oxidation states ranging from II to VI. However, only Cr(III) and Cr(VI) are stable enough to actually be used in electroplating. Cr(VI) is readily reduced to the more stable Cr(III) and in this process, substances in contact with the Cr(VI) are oxidized. Cr(VI) compounds are very soluble compared to Cr(III) compounds. Therefore, in the environment Cr(VI) is much more likely, upon release into a stream or an aquifer, to dissolve and move with the flow. In fact, one method that has been used to stabilize Cr(VI) (make it less mobile) in the environment is to reduce it to Cr(III).[4]

Cr(VI) is widely recognized as a human carcinogen. In a recently published study,[5] Cr(VI) was reported to cause increased incidences of lung cancer in a group of 2,357 workers at a chromate production plant. In the same study, the carcinogenic property of Cr(III) was also examined. Gibb, *et al*.[5] found, "Cumulative hexavalent chromium exposure was associated with an increased lung cancer risk; cumulative trivalent exposure was not." This study "offers the best quantitative evidence to date of the relationship between Cr(VI) and lung cancer." In previous studies, it was recognized that workers were exposed to a mixture of Cr(VI) and Cr(III). However, the relative contributions of each of the forms of chromium were not assessed. In the Gibb, *et al.* study, the relative exposure levels indicated that exposure of workers to Cr(III) did not result in an increase in lung cancer. When comparing the two forms of chromium, the chemistry and toxicology of each form is markedly different. Cr(III)

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is rather insoluble and does not oxidize organic material. Cr(III) is poorly absorbed from the gastrointestinal tract and is not considered to be a carcinogen. On the other hand, Cr(VI) is very soluble, penetrates cell walls, and is a human carcinogen. Cr(VI) is very reactive and causes ulceration of the nasal septum and other tissues upon exposure as well as reproductive, gastrointestinal and dermal effects. Even so, Cr(III) is not without toxic effects and should be handled appropriately. To put this last statement in perspective, a ubiquitous metal like aluminum, for example, is not without toxic effects. Although aluminum is formed into cooking utensils and soda cans, depending on the route of exposure and dose, aluminum can be potentially toxic (Occupational Safety and Health Guideline for Aluminum).

The use of Cr(III) in industrial and commercial processes is preferred over Cr(VI) on the basis of the comparison of the toxicities. Cr(III), like other metals (*e.g.*, aluminum) must be handled appropriately since each of these metals is at some dose and via at least one route of exposure, capable of causing harm to humans.[4]

From an environmental perspective, plating from additive-free Cr(III) solution has several advantages relative to Cr(VI):

1. Cr(III) is non-toxic, non-hazardous and is not an oxidizer. Therefore, meeting air quality regulations is easier and working conditions are greatly improved. The exposure limit for Cr(III) is an order of magnitude higher than Cr(VI).
2. Disposal costs are significantly reduced for Cr(III) plating. Hydroxide sludge generation is reduced ten to twenty times because Cr(III) generally operates at a Cr(III) content of about 4-20 g/L vs. 150-300 g/L for a Cr(VI) bath.
3. Since there are no proprietary additives in the Cr(III) bath, the rinse water may be recycled.

In addition, Cr(III) has the following technical advantages:

1. The Cr(III) plating bath is not sensitive to current interruptions.[6] Therefore, the innovative modulated reverse current approach used in this program is more suitable for Cr(III) plating than for Cr(VI) plating.
2. Drag-in of chloride and sulfate from any previous nickel plating operations into the Cr(III) process can be tolerated.[7] By contrast, chloride and sulfate drag-in upset the catalyst balance in a Cr(VI) process.
3. Throwing power for Cr(III) plating, which is poor in a Cr(VI) bath, is good and similar to other metals such as copper.[7]

Chromium coatings are widely used in a variety of industries. Plating operations are used to fabricate two types of chromium coatings, functional and decorative. Functional chromium coatings consist of a thick layer of chromium (typically 1.3 to 760 μm[8]) to provide a surface with functional properties such as hardness, corrosion resistance, wear resistance and low coefficient of friction. Applications of functional chromium coatings include strut and shock absorber rods,

hydraulic cylinders, crankshafts and industrial rolls. Carbon steel, cast iron, stainless steel, copper, aluminum and zinc are substrates commonly used with functional chromium. Decorative chromium coatings consist of a thin layer of chromium (typically 0.003 to 2.5 μm[8]) to provide a bright surface with wear and tarnish resistance when plated over a nickel layer. It is used for plating automotive trim/bumpers, bath fixtures and small appliances.

As described above, Cr(III) plating has numerous environmental, health and technical advantages relative to Cr(VI) plating. Considerable research has been done to study Cr(III) plating, including the effects of the plating bath chemistry on plating thickness, brightness, hardness and corrosion resistance[9,10,11] and the effect of current waveforms on chromium deposit structure, distribution, brightness and hardness.[12,13] Currently, functional chromium plating from a Cr(III) bath is not commercially available because of the challenge to plate thick chromium coatings with the appropriate properties. In addition, the low current efficiency and low plating rate of Cr(III) baths lead to unfavorable economics. Due to the rapid drop in current efficiency, the practical limit for existing conventional DC Cr(III) plating is 2.5 μm.[14] The plating thickness increases quickly at the beginning of the electroplating process. As plating continues, the deposition rate diminishes and becomes negligible. In this work we demonstrate a technique in which thick chromium coatings are formed.

Pulse/pulse reverse electroplating

During Cr(III) plating, chromium is deposited and hydrogen is evolved at the cathode, as described in the following reactions:

$$Cr^{+3} + 3e^- \rightarrow Cr \ (\phi^o = -0.74 \ V_{SHE}) \quad (1)$$

$$2H^+ + 2e^- \rightarrow H_2 \ (\phi^o = 0 \ V_{SHE}) \quad (2)$$

The current efficiency for chromium plating from a Cr(III) bath is usually below 20%. Therefore, about 80% of the current is used for the hydrogen evolution reaction. As a result, the pH near the cathode surface increases dramatically and chromic hydroxide ($K_{sp} = 5.4 \times 10^{-31}$) precipitates in the high pH layer at the cathode. The sedimentation of chromic hydroxide covers the cathode surface and its thickness increases as the plating time and pH increase. This promotes an increase of cathode polarization, a further decrease of chromium plating efficiency (*i.e.*, increase in the hydrogen evolution reaction) and the increase of impurities in the plating film. All of these factors retard the normal growth of crystals in the plating film, leading to the inhibition of chromium plating as the evolution of hydrogen continues. The precipitation of chromic hydroxide at the cathode also results in surface cracks and reduces the hardness and brightness of the chromium coating. Our approach deals with overcoming this hydrogen evolution problem by utilizing electrically mediated fields**.

The process utilizes pulse and pulse reverse waveforms. Figure 1 is an example of a pulse / pulse reverse waveform, consisting of a cathodic (forward) pulse followed by an anodic (reverse) pulse and a relaxation period (off-time). The cathodic peak current (i_c), cathodic on-time (t_c), anodic peak current (i_a), anodic on-time (t_a), and the relaxation-time (t_o) are individual variables for process control. The sum of the cathodic on-time, anodic on-time and relaxation-time is the period of the modulation and the inverse of the period is the frequency. The cathodic duty cycle (γ_c) is the ratio of the cathodic on-time to the period, and the ratio of the anodic on-time to the period is the anodic duty cycle (γ_a). The frequency and duty cycles are additional variables for process control. The average current density (i_{aver}) or electrodeposition rate is given by:

$$i_{aver} = i_c\gamma_c - i_a\gamma_a \qquad (3)$$

Just as there are infinite combinations of height, width and length to obtain a given volume, in pulse and pulse reverse processes there are unlimited combinations of peak current densities, duty cycles and frequencies to obtain a given electrodeposition rate. By controlling the cathodic on-time, anodic on-time, relaxation-time and the cathodic and anodic peak currents, precise control of the electrodeposition process is achieved and consequently the properties of the resulting deposit may be controlled or fine-tuned for a specific application. In conventional direct current (DC) electrodeposition, the current is turned on and held for the duration of the process. By interrupting this constant stream of current, as in the pulse / pulse reverse process, one may achieve results not possible with conventional DC electroplating, such as deposit property control and elimination of adverse side reactions, such as hydrogen evolution, which may become co-deposited with chromium, affecting the nature of the electrodeposit and resulting in samples with inadequate wear properties.

Elimination of hydrogen

Pulse / pulse reverse plating can be used to reduce the amount of H_2 inclusion in the coating. This section further explains how H_2 can be incorporated in the coating and discusses how the density of these inclusions can be mitigated. The evolution of hydrogen during a metal deposition process results in a low current efficiency and a high power consumption. In addition, hydrogen evolution during metal deposition leads to hydrogen embrittlement in the substrate or hydrogen bubble inclusion in the metal deposit. This adversely affects the electrodeposit in terms of:

1. Low corrosion resistance,
2. Poor adhesion to the substrate,
3. High internal stress
4. Excess porosity.

By properly adjusting the electrically-mediated parameters, one can alter the kinetics of both the metal deposition and hydrogen evolution reactions. While reaction kinetics can be modified using an electrically-mediated waveform with only
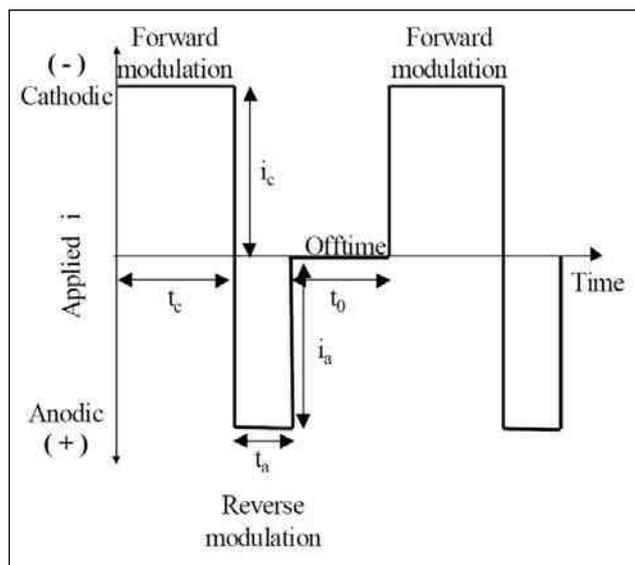

Figure 1 - Schematic of a generic pulse / pulse reverse waveform.

a forward modulation, hydrogen bubbles adsorbed on the electrode surface can be released during the off-time period. By using an electrically-mediated waveform with a reverse modulation, one can "tune" the anodic or reverse part of the waveform to consume the nascent hydrogen according to the following reaction:

$$H_2 \rightarrow 2H^+ + 2e^- \qquad (4)$$

In this manner, a low pH is maintained and chromic hydroxide precipitation is avoided. Furthermore, hydrogen is not permitted to diffuse into the substrate or be incorporated in the deposit. Finally, by compensating for the anodic current with an increased cathodic current, the overall electrodeposition rate is maintained and the net current efficiency is improved.

Mass transfer

The ability of pulse / pulse reverse waveforms to increase mass transfer to and from the surface during deposition is essential to producing a coating whose properties contain minimal side products, for instance H_2 bubble inclusion which for chromium reduces its wear resistance and adhesion. Mass transport in the pulse reverse process is a combination of steady state and non-steady state diffusion processes. The mass transfer limited current density (i_l) is related to the reactant concentration gradient (C_b - C_s) and to the diffusion layer thickness (δ) by the following equation:

$$i_\ell = -nFD \left(\frac{\partial c}{\partial x}\right)_{x=0} = -nFD \frac{(c_b - c_s)}{\delta} \qquad (5)$$

Cheh and coworkers[15,16] discussed the theory of mass transport with respect to pulse electrolysis. In steady state DC electrolysis, δ is a time-invariant quantity for a given electrode geometry and hydrodynamics. In pulse and pulse reverse electrolysis, however, δ varies from 0 at the begin-
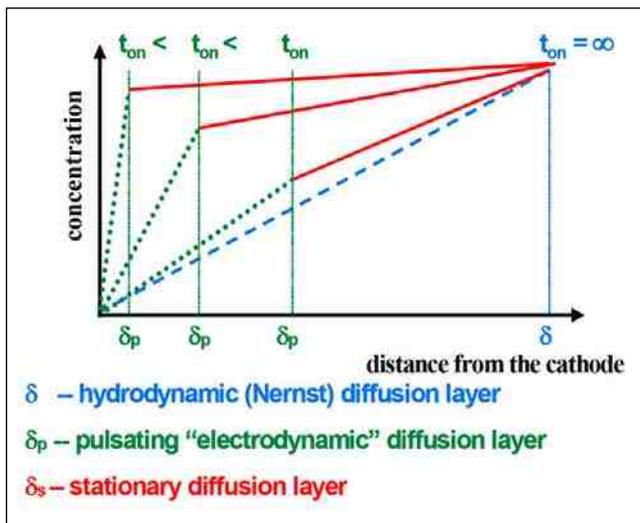
Figure 2 - Schematic representation of a diffusion layer.

ning of the pulse to its steady state value when the Nernst diffusion layer is fully established. The corresponding diffusion limiting current density would then be equal to an infinite value at $t = 0$ and decreases to a steady state value of the DC limiting current density. The advantage of pulse and pulse reverse electrolysis is that the current can be interrupted before δ has a chance to reach the steady-state value. This allows the reacting ions to diffuse back to the electrode surface and replenish the surface concentration to its original value before the next current interruption. Therefore, the concentration of reacting species in the vicinity of the electrode pulsates with the frequency of the modulation.

Under pulse electrolysis, Ibl and colleagues[17,18,19] proposed a "duplex diffusion layer" consisting of a pulsating layer, δ_p, and a stationary layer, δ_s. Modeling work by Landolt has also suggested the existence of a pulsating diffusion layer.[20]

Since the thickness of the pulsating diffusion layer is determined by the waveform parameters, we call this layer the "electrodynamic diffusion layer"(Fig. 2).[21] By assuming a linear concentration gradient across the pulsating diffusion layer and conducting a mass balance, Ibl derived the pulsating diffusion layer thickness (δ_p) as:[18]

$$\delta_p = (2Dt_{on})^{1/2} \tag{6}$$

and when the pulse on-time is equal to the transition time, τ, the concentration of reacting species at the interface drops to zero precisely at the end of the pulse:

$$\tau = \frac{[(nF)^2 c_b^2 D]}{2i_c^2} \tag{7}$$

More exact solutions are given by integrating Fick's diffusion equation:

$$\delta_p = 2\left(\frac{Dt_{on}}{\pi}\right) \tag{8}$$

$$\tau = \pi \frac{[(nF)^2 c_b^2 D]}{4i_c^2} \tag{9}$$

More recently, Yin,[22] using a similar approach as Ibl, derived the same equation for the pulsating diffusion layer for "pulse-with-reverse" plating.

The key points to consider for development of a pulse / pulse reverse manufacturing process for deposition of functional chromium include (1) the electrodynamic diffusion layer thickness is proportional to the pulse on-time, and (2) the transition time is inversely proportional to the current.
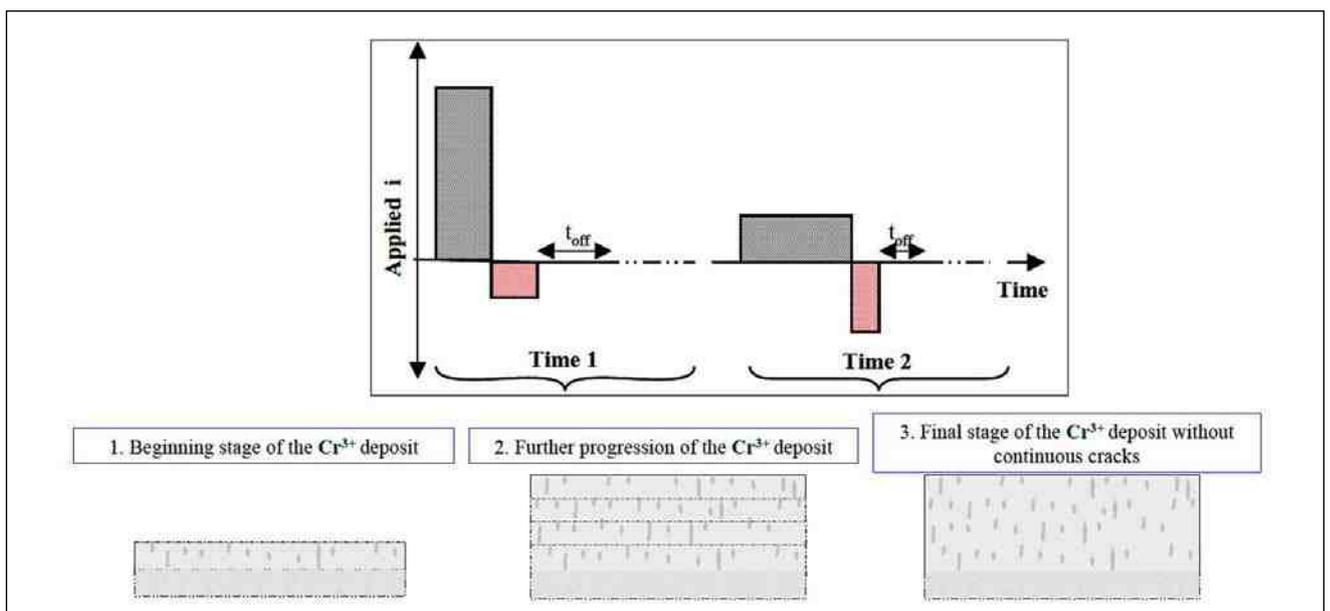

Figure 3 - Application of a sequenced pulse / pulse reverse process to produce a thick chromium coating with non-continuous through cracks.

Prior work on functional Cr(III) plating

In previously reported work we accomplished the following milestones:

- Plated from a trivalent chromium sulfate bath where the consumable was in the price range of chromic acid,[23]
- Simplified the bath chemistry using an electrically-mediated waveform,[23]
- Increased the chromium plating thickness to 250 μm,[23]
- Maintained a plating rate similar to that of hexavalent chromium,[23]
- Determined the maximum diffusion layer thickness for plating from a trivalent chromium sulfate bath,[24]
- Determined the acceptable range for achieving bright plating,[24]
- Replated chromium on chromium,[24]
- Displayed superior corrosion resistance compared to laser-coated surfaces,[25]
- Achieved a suitable chromium distribution for pump rotor wear tests at a manufacturer,[26]
- Achieved equal or better wear resistance compared to a Cr(VI) deposit against hardened steel,[26]
- Produced a similar micro-hardness to that of Cr(VI) deposits
- Developed a technique to deposit thick chromium up to 500 μm (20 mils) with minimal cracking by sequencing the electrical-mediation process parameters (Figure 3 demonstrates the looping behavior on the chromium deposit.).

Current work on functional Cr(III) plating

The current program is set to validate the functionality of the chromium deposits produced by the sequenced pulse / pulse reverse process and a Cr(III) bath. The evaluation criteria include:

- Thickness per AMS 2460, 3.4.1
- Porosity per AMS 2460, 3.4.4
- Adhesion per AMS 2460, 3.4.2
- Surface roughness and morphology
- H_2 embrittlement per ASTM F519
- Hardness AMS 2460, 3.4.3
- Corrosion resistance per ASTM B519 (Salt Spray)
- Wear
    - o Taber Abrasion per ASTM D4060
    - o Ball on Disc per ASTM G133
    - o Oscillation
- Amp-hr solution analysis for Cr(VI)
- Fatigue axial tension per ASTM E466 and ASTM E468
- Rotating beam tests per ISO 1143 and ASTM E468.

Results

Effect of surface pretreatments

The initial hurdle overcome by the programs was the development of a surface preparation process that was used to increase coating adhesion (AMS 2460 3.4.2, Bend to Break Test) while minimizing the porosity (AMS 2460, 3.4.4, Ferroxyl Test). The factors examined for this analysis included anodic electrocleaning, grit blast particle size (54, 120, and 220), H_2SO_4/HF anodic etch, post bake and reverse

etch in the Cr(III) plating bath composition without the chromium. The data indicated that a post bake at 190°C (375°F) for 24 hours was necessary in order to pass AMS 2460, 3.4.2 (Bend to Break Test). However, the baking process introduced non-uniformities and pores in the majority of the coatings. Figure 4 shows the effect of surface pretreatments on the porosity and adhesion of the coating in which all, except for the sample lacking additional pretreatments, passed the adhesion test due to the post-deposit bake. Additionally, the deposit with the least observed porosity was formed on a surface that was pretreated with both an anodic electroclean and a reverse etch in the non-Cr bath (pH 2.5).

Performance

Preliminary results have been obtained by plating 51-127 μm (2-5 mils) of chromium from a Cr(III) bath on 1 × 4-inch 4130 normalized steel strips and comparing these deposits performance head-to-head with conventional Cr(VI) deposits. We have successfully demonstrated comparable adhesion (bend break test), corrosion resistance (salt spray), thickness and surface morphology. This behavior is further demonstrated in Figure 5.
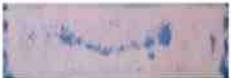

Figure 4 - The effect of pretreatment conditions on the porosity and adhesion of the chromium deposits after a 24-hr bake at 190°C (375°F).
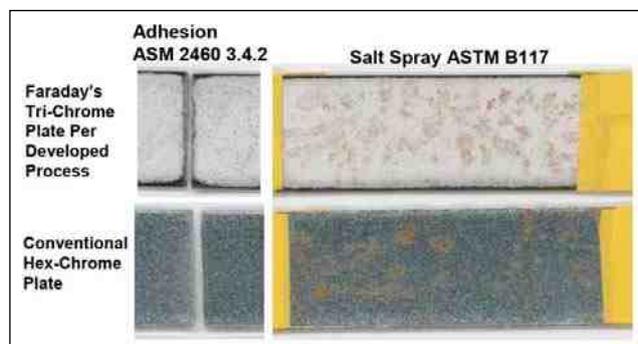

Figure 5 - Head-to-head comparison of the Cr(III) plating performance to that of a conventional Cr(VI) plate.

Discussion

Faraday has demonstrated the ability to produce chromium coatings, from a Cr(III) bath, that are functionally the same as the coatings produced from conventional Cr(VI) baths, in terms of plate adhesion and corrosion resistance. Future tests are planned to validate the micro-hardness through AMS 2460 3.4.3, the resistance to H_2 embrittlement, and the durability under wear and fatigue stresses.

Conclusions

In summary, we are continuing research and development to commercialize our trivalent chromium plating process for functional and decorative applications. We have established the following:

- Plated from a trivalent chromium sulfate bath where the consumable was in the price range of chromic acid,[23]
- Simplified the bath chemistry using an electrically-mediated waveform,[23]
- Increased the chromium plating thickness to 250 μm,[23]
- Maintained a plating rate similar to that of hexavalent chromium,[23]
- Determined the maximum diffusion layer thickness for plating from a trivalent chromium sulfate bath,[24]
- Determined the acceptable range for achieving bright plating,[24]
- Replated chromium on chromium,[24]
- Showed that the corrosion resistance of trivalent chromium Cr(III) deposits was superior to laser-treated coatings,[25]
- Constructed a pilot-line plating set-up to plate parts currently used in production to allow commercial evaluation,[25]
- Achieved a suitable chromium distribution for pump rotor wear tests at a manufacturer,[26]
- Achieved equal or better wear resistance compared to a Cr(VI) deposit against hardened steel,[26]
- Demonstrated the capability of the process to deposit chromium onto a bright nickel substrate for bath fixture applications,
- Produced a similar micro-hardness to that of Cr(VI) deposits,
- Developed a technique to deposit thick chromium up to 500 μm (20 mils) with minimal cracking by sequencing the electrical-mediation process parameters.
- Demonstrated a comparable corrosion resistance and plate adhesion to that of Cr(VI) plated samples and
- Developed a surface pretreatment that enhances the deposit quality.

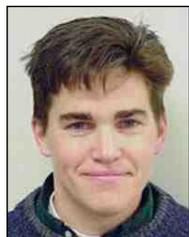## Acknowledgement

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