



The William Blum Lectures

#46 – E. Jennings Taylor – 2007



The 46th William Blum Lecture
Delivered at NASF SUR/FIN 2008
in Indianapolis, Indiana
June 16, 2008

Adventures in Pulse/Pulse Reverse Electrolytic Processes: Explorations and Applications in Surface Finishing

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ABSTRACT

This paper focuses on the application of pulse/pulse reverse (P/PR) electrolytic processes to problems of relevance to the surface finishing industry. After a brief introduction to the concept and principles of P/PR electrolysis, I discuss the importance of electrolytic cell design in establishing a uniform hydrodynamic boundary layer. I then focus on past developments and current applications of P/PR electrolysis at Faraday Technology, including (1) cathodic processes for electrodeposition and electrophoretic deposition, (2) anodic processes for electrodeburring, electropolishing and electroetching and (3) processes for electro-assisted ion exchange.

Introduction

After it was announced at SUR/FIN 2007 that I was the recipient of the 2008 Scientific Achievement Award, named in honor of William Blum, I was extremely humbled and somewhat overwhelmed. As I perused the previous winners of this very prestigious award, many of whose works are familiar to me, I again felt humbled. I say this because I have had the benefit of working and learning from many colleagues and collaborators, both past and current, in the area of P/PR electrolysis. I hope to acknowledge all of my colleagues / collaborators at the conclusion of this manuscript.

My first introduction to P/PR electrolysis was as a graduate student while conducting my dissertation research at Brookhaven National Laboratory and I first begin working in the area while employed at Physical Sciences Inc. From these initial forays, I had the good fortune and family support to found Faraday Technology, Inc. in 1991, whose audacious vision was and still is:

"...to be known as the company that changed the focus of electrochemical technologies from the art of complex chemistries to the science of P/PR electric fields..."

Our approach has been coined "Electrochemical Magic of a Different Kind."¹

At Faraday, we have the freedom to explore our vision for P/PR electrolysis, as long as we can convince people to fund our ideas for their applications. The *quid pro quo* to commercial company funding is that in return, they have the exclusive rights associated with the P/PR developments. Consequently, intellectual property in the form of patents and know-how plays an important part in our strategy and I cite them below as appropriate. After seventeen years of commercial and government-sponsored research, twenty-one issued U.S and five foreign patents, numerous licensees and following the acquisition of Faraday by Physical Sciences, I am pleased to say the vision is indeed alive and well!

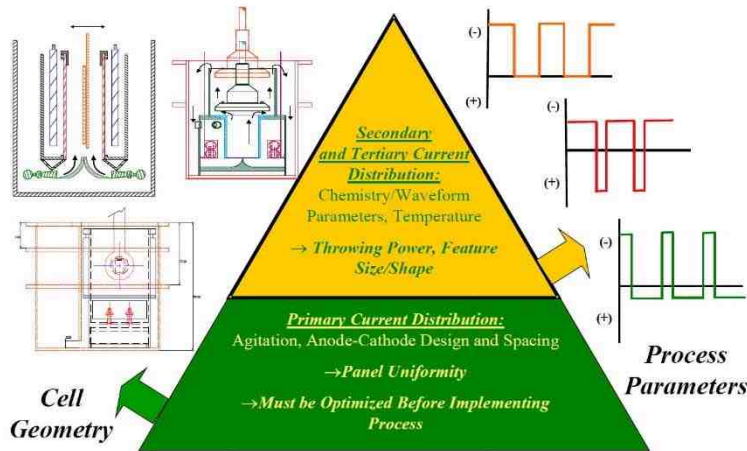


Figure 1 - Cell geometry as the foundation for electrolytic process development.

Although the focus of our activities is the development of P/PR electrolysis processes, an important and often forgotten consideration is the design of the electrolytic apparatus or cell. As depicted in Fig. 1, the cell geometry is the foundation of our process development. In fact, I submit that cell design considerations are often neglected when evaluating process parameters and a poor cell design often overshadows an otherwise superior process.

Critical to our development of P/PR electrolytic process parameters was the seminal work by Puipe and Leaman, published by the American Electroplaters and Surface Finishers Society, *The Theory and Practice of Pulse Plating*.² Although

out of print, several well-worn copies of this tome grace the Faraday laboratories.

Since the combination of P/PR electrolysis waveforms to achieve a given average current density is infinite, over the years we have developed some simplifying assumptions and guiding principles regarding the development of P/PR electrolysis applications.

P/PR electrolysis: guiding principles

As shown in Fig. 2, the generalized P/PR waveform consists of a forward modulation (shown as cathodic) with a cathodic current density, i_c , a cathodic on time, t_c , a reverse modulation (shown as anodic) with an anodic current density, i_a , an anodic on time, t_a , and an off-time, t_0 . Note, an additional off time between the forward and reverse modulation is not depicted for simplicity. The sum of the cathodic and anodic on-times and the off-time is the period, T , of the modulation and the inverse of the period is the frequency, f , of the modulation. The cathodic, γ_c , and anodic, γ_a , duty cycles are the ratios of the respective on-times to the charge modulated period. The average current density (i_{aver}) or net electrodeposition rate is given by:

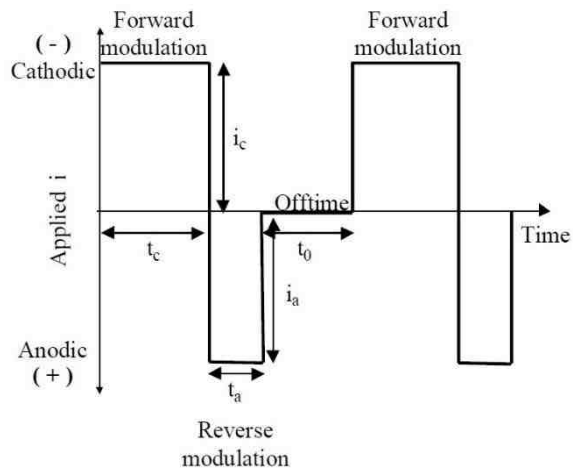


Figure 2 - Generic representation of P/PR electrolysis waveform.

$$i_{aver} = i_c \gamma_c - i_a \gamma_a \quad (1)$$

Just as there are infinite combinations of height, width, and length to obtain a given volume, in P/PR processes there are unlimited combinations of peak current densities, duty cycles and frequencies to obtain a given electrolysis rate. These additional parameters provide the potential for much greater process/product control versus DC plating.

Mass transport in P/PR electrolysis is a combination of steady state and non-steady state diffusion processes. Cheh and coworkers^{3,4} previously 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 hydrodynamic conditions. In P/PR electrolysis, however, δ varies from 0 at the beginning 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 P/PR 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 modulation. 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^{5,6,7} 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.⁸ Since the thickness of the pulsating diffusion layer is determined by the waveform parameters, we call this layer the “electrodynamic diffusion layer.”⁹ 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:⁶

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

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:

$$\tau = ((nF)^2 C_b^2 D) / 2i_c^2 \quad (3)$$

More recently, Yin,¹⁰ using a similar approach as Ibl, derived the same equation for the pulsating diffusion layer for “pulse-with-reverse” plating.

The key points used in our development of a P/PR electrolysis process are (1) the electrodynamic diffusion layer thickness is proportional to the pulse on-time, and (2) the transition time is inversely proportional to the current. Further, note that the on time is directly proportional to the duty cycle and inversely proportional to the frequency.

We consider two additional important aspects when designing a P/PR electrolysis process. The first is whether the current distribution is controlled by primary (geometrical), secondary (kinetic) or tertiary (mass transport) considerations. For example, if the waveform is designed so that the pulse on time is much longer than the transition time, the tertiary current distribution will be an important factor in metal distribution. The addition of kinetic or tertiary effects tends to make the current distribution more uniform, as compared to primary current distribution.

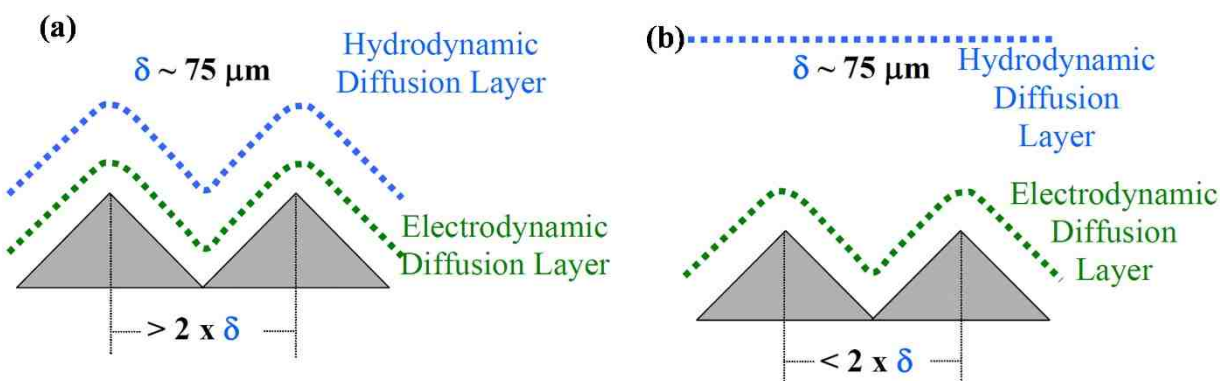


Figure 3 - Under pulse electrolysis: (a) Macroprofile converted to a smaller macroprofile and (b) Microprofile converted to a macroprofile.

When tertiary current distribution effects are important, additional criteria that influence current distribution are the concepts of macroprofile and microprofile. In a macroprofile, the roughness of the surface is large compared with the thickness of the

diffusion layer, and the diffusion layer tends to follow the surface contour. Under mass transport or diffusion control, a macroprofile results in a uniform current distribution or a conformal deposit during plating. In a microprofile, the roughness of the surface is small compared with the thickness of the diffusion layer. As shown in Figs. 3a and b, pulse electrolysis can convert a macroprofile to a smaller macroprofile and a microprofile to a macroprofile, respectively.

Our final “guiding principle” regarding the exploration of applications for P/PR electrolysis involves strict application of the scientific method by testing assumptions. Specifically, much of the prior art, P/PR electrolysis took process chemistries developed for direct current (voltage) processes and applied pulse waveforms. However, we asked two questions:

1. *Why should we expect P/PR electrolysis processes to use the same chemistries in terms of electrolytes and additives as those developed for DC processes?*
2. *In fact, do P/PR electrolysis processes require anything more than simple electrolyte and additive chemistries?*

Consequently, our approach to the exploration of P/PR electrolysis applications begins with simple electrolytes and easy to control additives.

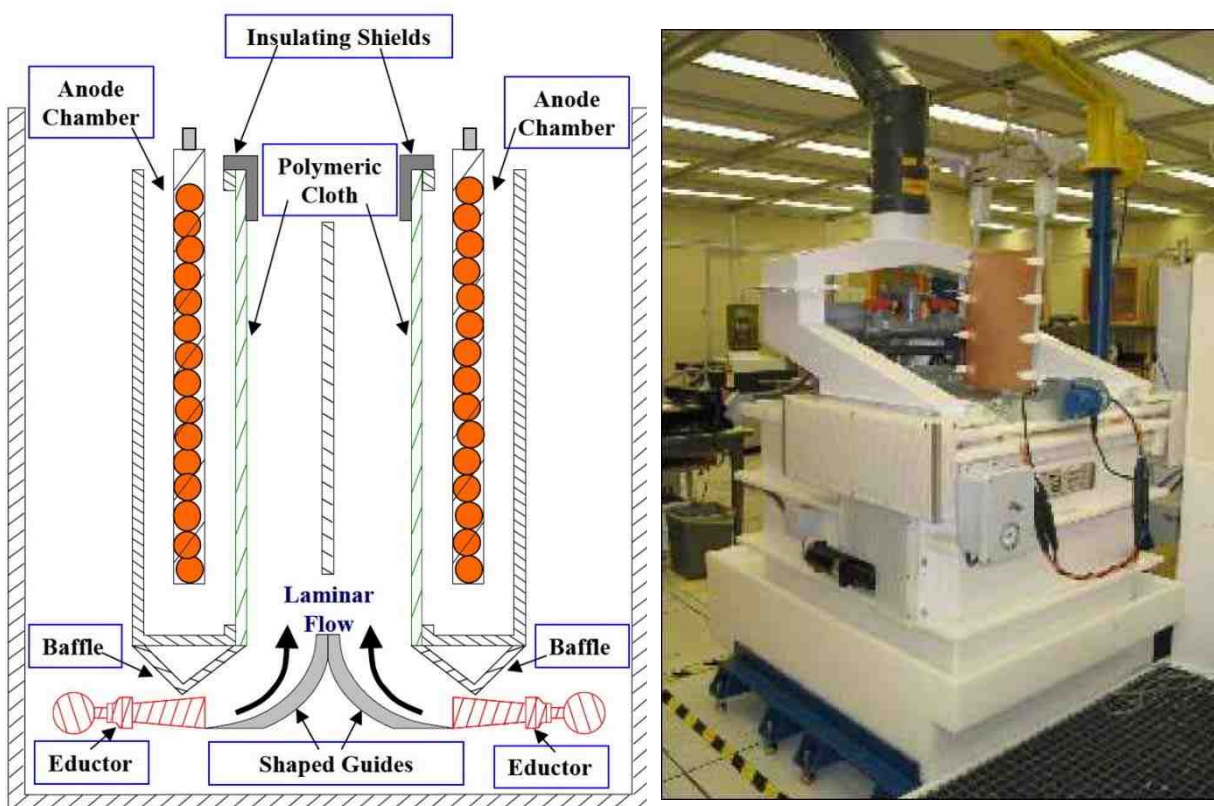


Figure 4 - (a) Schematic diagram of an electrolytic cell facilitating uniform laminar flow and (b) photo of the operating unit.

Electrolytic cell for uniform hydrodynamic boundary layer

As noted above, the design of the electrolytic apparatus is critical to the development of P/PR processes, as well as conventional process technology. Masking is an important geometric consideration for electrolytic apparatus design and we presented a simple model for guidance.¹¹ In addition, conventional agitation approaches typically use air/inert gas sparging or eductors

impinging on the workpiece with coefficients of variation of 14 and 18%, respectively, for a rectangular workpiece of 450 × 600 mm.¹²

A design developed at Faraday utilizes eductors to avoid the issues associated with non-uniform gas bubble size in sparging approaches. However, in our approach, the eductor flow does not directly impinge upon the workpiece as shown in Fig. 4.^{13,14} With this flow scheme, we have demonstrated coefficient of variation of 4 to 5% for 200 mm wafer package and 450 × 600 mm circuit board substrates, respectively. In addition, we have measured boundary layers approaching 10 μm in thickness using limiting current measurements.

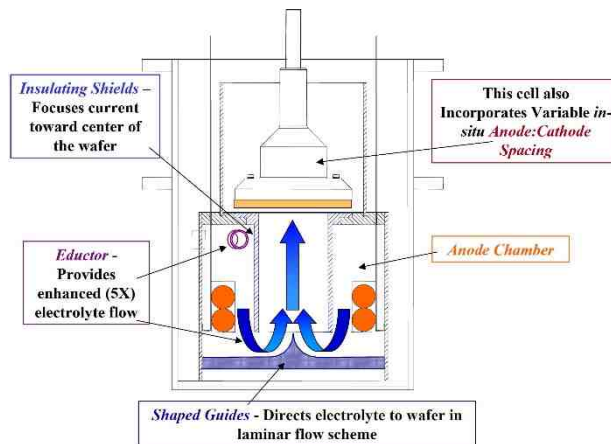


Figure 5 - Wafer plating tool with rotation and vertical movement.

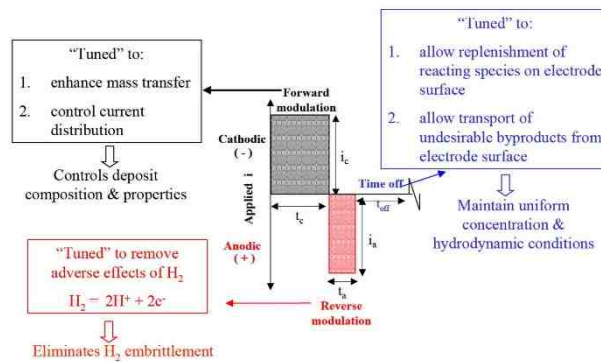


Figure 6 - Generalized pulse/pulse reverse waveform for cathodic processes – electrodeposition.

Another design for horizontal electrolytic processes uses a rotating wafer with vertical movement to compensate for terminal effects due to a minimally conductive seed layer.¹⁵ For this design, shown in Fig. 5, we demonstrated coefficient of variation of 4 to 6% and a boundary layer thickness of ~34 μm.¹⁶

P/PR cathodic electrodeposition processes

Figure 6 depicts a generalized P/PR electrolysis waveform for electrodeposition applications where the “forward” cycle is cathodic and the “reverse” cycle is anodic and the net process is cathodic. In addition to the current distribution issues discussed above in terms of macro and microprofiles and the electrodynamic boundary layer, we tune the P/PR waveform reverse (anodic) cycle to consume the nascent hydrogen generated in many cathodic processes. As discussed below, this is an important enabling function of P/PR electrolysis for certain applications.

An initial application we investigated for P/PR electrolysis involved metallization of interconnects for printed circuit boards and electronic packages. The main challenge for these applications is controlling the current distribution in order to prevent “dog-boning,” *i.e.*, to obtain good throwing power. While throwing power is generally addressed using plating baths

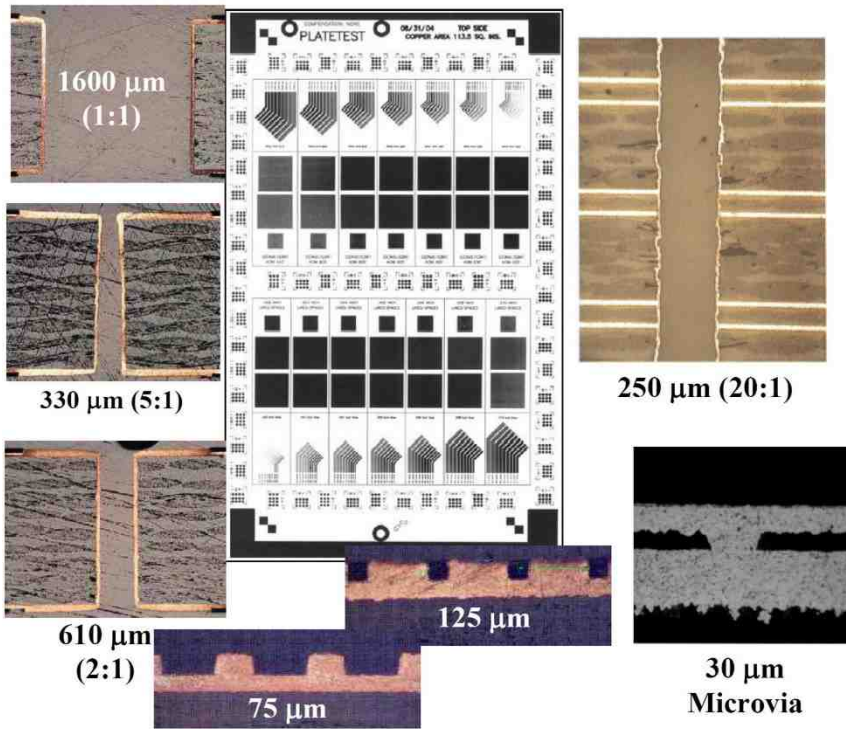


Figure 7 - P/PR electrolysis related to interconnects for electronic applications.

containing difficult-to-control additives such as levelers and brighteners, we used the P/PR waveform to control the throwing power. As shown in Fig. 7 for the test panel with various features, we obtained good throwing power for plated through holes and pattern plating over a range of feature sizes. Our approach generally used the concepts described above for microprofile and macroprofile boundary layers and tuning the forward (cathodic deposition) and reverse (anodic dissolution) waveform characteristics in order to yield the net result of conformal coating or filling of the subject interconnects.^{17,18} Another advantage of P/PR electrolysis is that for electronic packages having different feature sizes, a sequence or train of waveforms can be delivered to address the range of features.^{19,20,21}

Another important property of P/PR electrolysis related to electronic packages is the ductility of the resulting deposit. In Fig. 8, we present stress/strain data for copper foils prepared from a commonly used chemical additive bath using a conventional process at the Naval Surface Warfare Center - Crane (NSWC-Crane) Indiana and data from a simple chemical bath using a P/PR waveform sequence developed at Faraday. We observed equivalent yield strength and elongation with the P/PR prepared

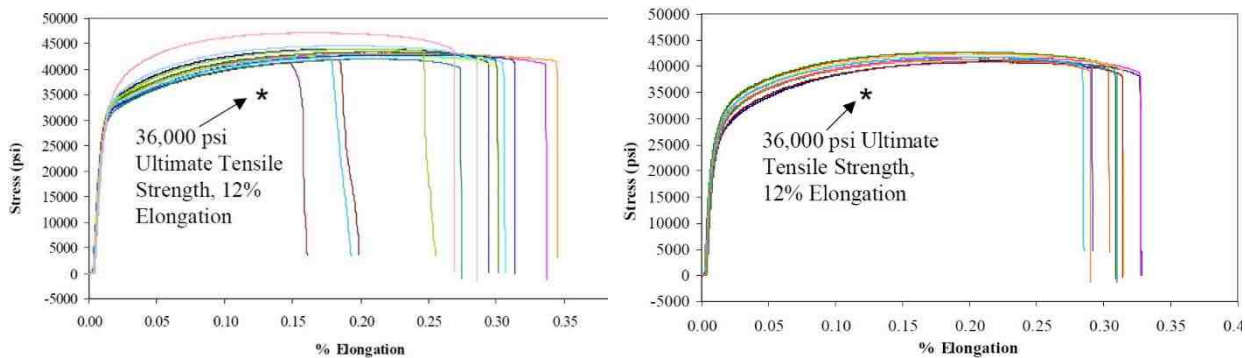


Figure 8 - Stress-strain curves for copper samples prepared using (a) a conventional additive chemistry process at NSWCCrane and (b) P/PR with a simple chemistry.

samples showing a standard deviation of ~2% for elongation compared to ~6% for the conventionally prepared samples.²² We demonstrated the versatility of P/PR electrolysis with semiconductor applications where the feature sizes are typically less than 0.25 μm and microprofile boundary layers are typically operative. A representative set of results is presented in Fig. 9.²³⁻²⁸

Another application for P/PR electrolysis is the electrodeposition of thick chromium coatings from an environmentally-friendly trivalent chromium plating bath. Current processes for trivalent chromium are limited to thin decorative applications. We speculated that this was due to the cathodic generation of hydrogen during trivalent chromium plating. Based on this assumption, we successfully demonstrated that we could plate thick chromium coatings from trivalent baths using P/PR electrolysis. We speculate that the reverse (anodic) cycle consumes the nascent hydrogen and allows the deposition to continue.²⁹⁻³² Although the chromium deposits prepared from P/PR electrolysis had a thickness equivalent to those from a standard hexavalent chromium bath, we observed poor properties in terms of wear and corrosion resistance. From cross-section data, we attributed these poor properties to continuous cracks through the coating to the substrate surface. Although chromium prepared from hexavalent processes also exhibits cracks, they are they discontinuous. Consequently, we used a looping P/PR waveform in order to interrupt the electrodeposition process with the idea of eliminating or reducing the contiguous cracks. In this manner, we demonstrated a significant reduction in contiguous cracks, as shown in Figure 10.^{33,34}

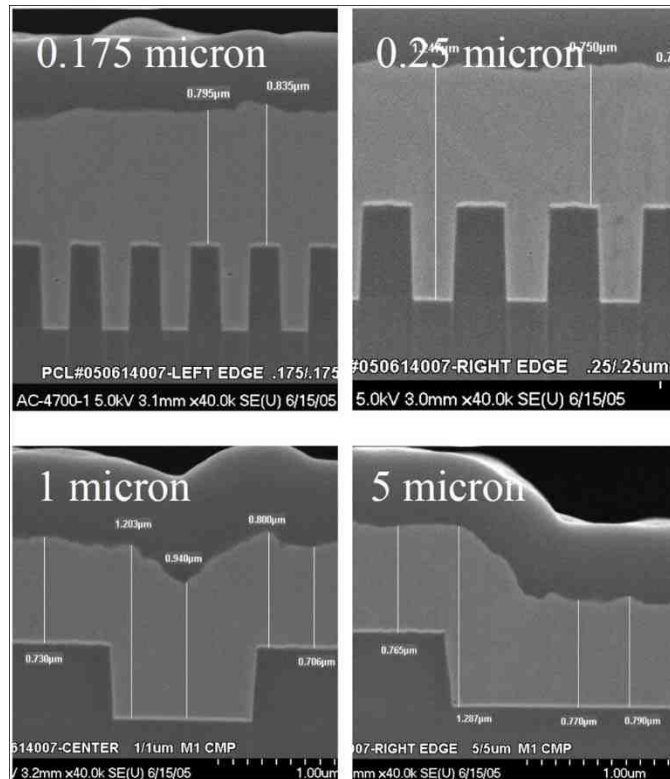


Figure 9 - P/PR electrolysis related to interconnects for semiconductor electronic applications.

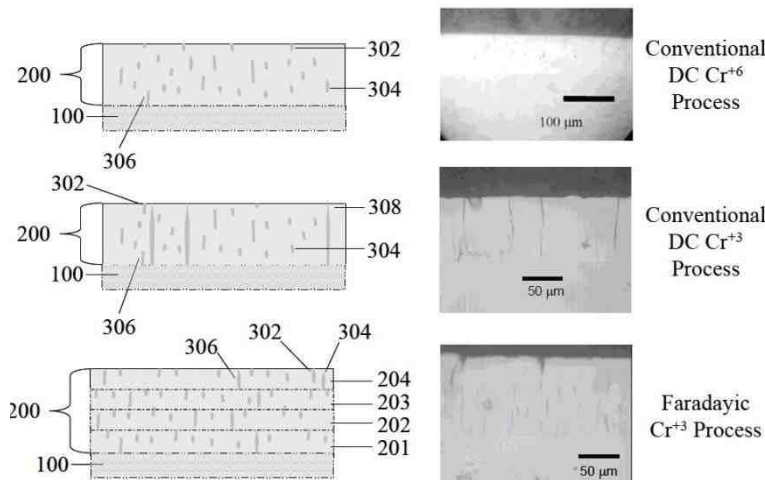


Figure 10 - Comparison of continuous and discontinuous cracks during DC chromium plating from a hexavalent chromium plating bath and from a trivalent chromium bath without and with P/PR looping.

Another application of P/PR waveform looping is related to tin electrodeposits as a lead-free solder. We previously demonstrated that various P/PR waveforms yield either tensile or compressive internal stress as well as different magnitudes of said stress.³⁵ Since internal stress is attributed to whisker growth in lead-free tin solders, we investigated several P/PR waveform sequences to generate layered tin deposits with different types and magnitudes of internal stress.³⁶ Preliminary data from thermal cycling indicate that depositing successive layers of different internal stress is a promising approach for mitigation of whisker growth in tin electrodeposits.

In a totally different application of P/PR electrodeposition, we demonstrated the feasibility of preparing platinum electrocatalysts for fuel cell gas diffusion electrodes.^{37,38}

Using the P/PR process we demonstrated the ability to form 3 to 4-nm catalyst particles with improved performance and the same loading of conventionally prepared electrocatalysts.³⁹ Another P/PR application we are exploring is the fabrication of three-dimensional structures for microelectromechanical systems (MEMS).⁴⁰

Thus far, I have described electrodeposition of metals from a plating bath containing ions of said metal. More recently, we are exploring the electrophoretic deposition of mixed oxides, specifically yttria-stabilized zirconia. We are investigating electrophoretic deposition of mixed oxides for applications such as thermal barrier coatings for turbine engines or varactor devices for RF filters, which are currently deposited using thermal spray or pulsed laser deposition, respectively. Electrophoretic deposition offers the benefits of speed and the ability to coat contoured surfaces. We are observing benefits of P/PR electrophoretic deposition and the ability to control coating morphology as shown in Fig. 11.^{41,42,43}

P/PR anodic (electropolishing, electrodeburring, electroetching) processes

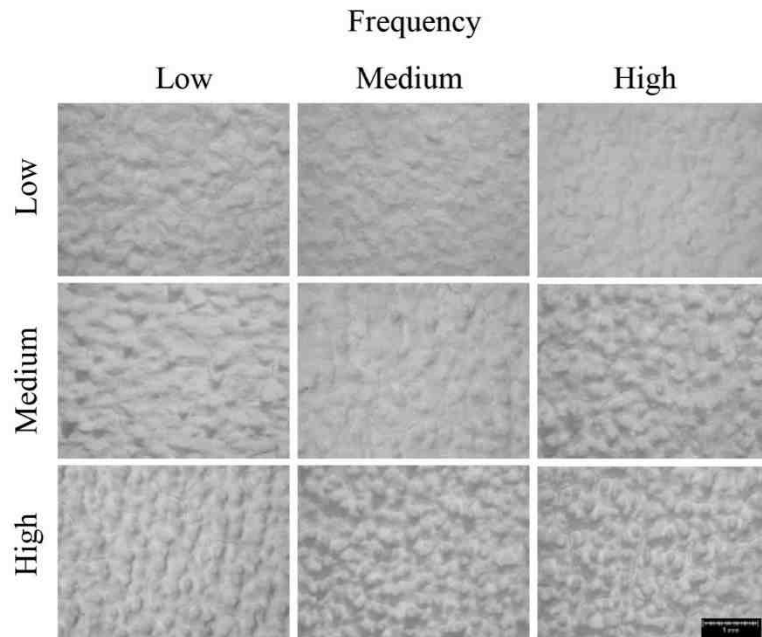


Figure 11 - Surface morphology of yttria-stabilized zirconia coatings prepared using P/PR electrophoretic deposition.

Figure 12 depicts a generalized P/PR electrolysis waveform for electropolishing, electrodeburring and electroetching applications where the “forward” cycle is shown as anodic, the “reverse” cycle is shown as cathodic and the net process is anodic. For P/PR electrolysis anodic applications, we tune the reverse cycle to remove surface oxides for certain materials and alloys in order to “activate” the surface for the anodic cycle.⁴⁴ In addition to activating the surface, we tune the waveform using the concepts of electrodynamic boundary layer and macro-and microprofiles to focus the current distribution for electropolishing, electrodeburring and electroetching applications.

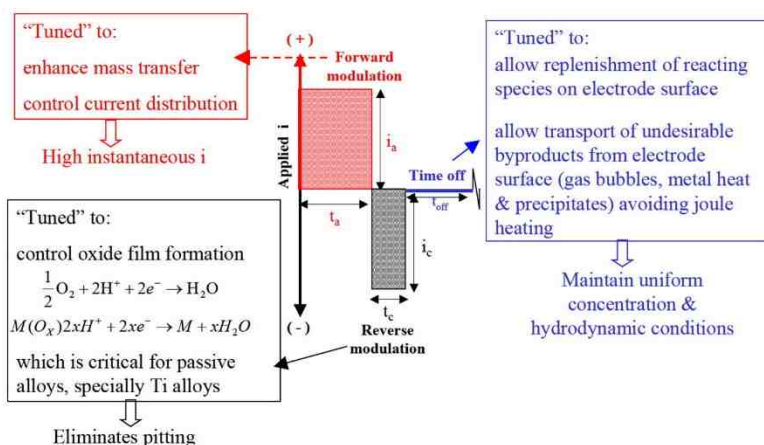


Figure 12 - Generalized pulse/pulse reverse waveform for anodic processes – electropolishing, electrodeburring and electroetching.

In Fig. 13, we present several exemplary electrodeburring applications using P/PR electrolysis. The planetary gear is made from tool steel and there was no need for a reverse cycle to remove the oxide and “activate” the surface. However, the blade was made from stainless steel and a reverse cycle was used.⁴⁵ In Fig. 14, we present a P/PR application that we are developing for removing molehills and radiusing leading/trailing edges of an aerospace integrally-bladed rotor. Of particular note is that by proper selection of the P/PR waveform parameters, we can achieve a radius, taper or nearly complete removal of artifacts resulting from prior

processing. The materials investigated to date include nickel and titanium based alloys.⁴⁶

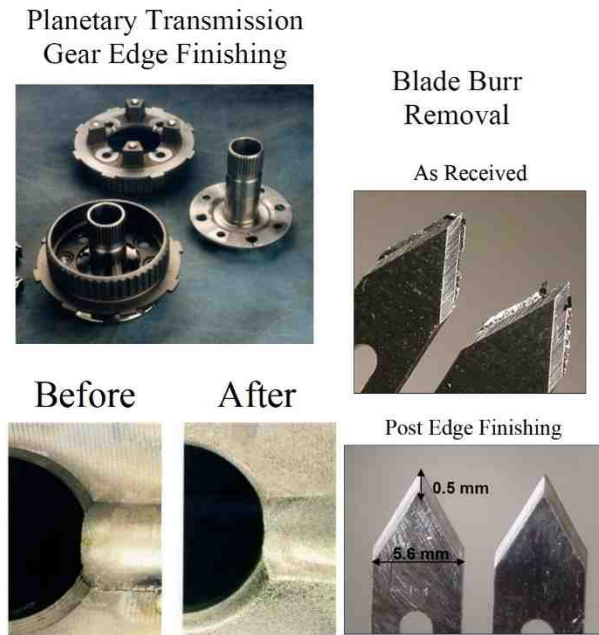


Figure 13 - Electrodeburning applications using forward pulse electrolysis.



Figure 14 - P/PR electrolysis applied to aerospace components.

In Fig. 15, we present several P/PR electropolishing applications related to various stainless steel parts.⁴⁷ An important consideration during P/PR electropolishing applications is that at the beginning of the process, the boundary layer may represent a macroprofile but as electropolishing continues a microprofile may evolve. To compensate for this change from macroprofile to microprofile, we use a sequence of P/PR waveforms to continue the electropolishing to the desired surface finish.⁴⁸ We are currently investigating additional P/PR electrolysis applications related to small parts and medical devices.^{49,50} Another application involves using P/PR electrolysis to prepare or “activate” a passive surface for subsequent electrodeposition.⁵¹

In Fig. 16, we present an example of P/PR electroetching through a mask on a substrate. P/PR electroetching applications include substrates consisting of a metal layer on a non-conductive layer (electronic packages)⁵² or substrates consisting solely of a metal layer (bipolar plates, microchannel reactors, microfluidic devices, cooling channels and the like). By tuning the P/PR waveform to focus the electric field, we minimize the mask undercut associated with isotropic nature of the chemical etching process.⁵³ We are exploring other applications of P/PR electrolysis including removal of sacrificial cores for internal channels^{54,55} and removal of excess metal plating for semiconductor applications.⁵⁶

Pulsed-assisted ion exchange

In Fig. 17, we present a schematic representation of pulsed assisted ion exchange for the treatment and regeneration of metal finishing process streams.^{57,58} Pulsed assisted ion exchange addresses several key limitations associated with traditional ion exchange. During the treatment cycle, the pulsed electric field enhances the ionic transport to the anion and cation exchange resin beds. During the regeneration cycle, the pulsed electric field enhances the regeneration of the anion and cation resin beds



Figure 15 - P/PR electropolishing of stainless steel components.

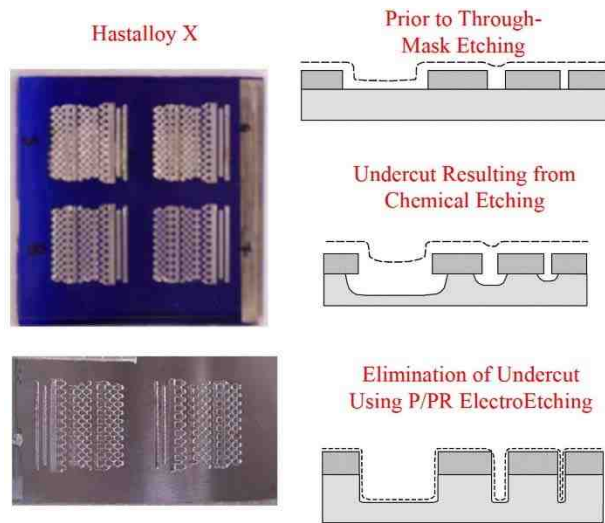


Figure 16 - P/PR through-mask electroetching.

without the need for strong acid or base process streams.⁵⁹ Although illustrated for copper rinse waters, we believe pulsed-assisted ion exchange is applicable to a wide range of process streams related to the surface finishing and nuclear industries.

Concluding remarks

My hope is that our collective body of work provides the basis for continued innovation related to the application of P/PR electrolysis to the electrochemical arts. I strongly believe that although our innovations to date are significant, we have only exposed the tip of the iceberg of possibilities related to P/PR electrolysis. In addition, I recommend that we as scientists, technologists and researchers continue to revisit and test our assumptions. Additionally, I submit that the patent literature is a valuable source of technical information and should be reviewed just as more traditional sources of technical information are consulted. Finally, I strongly encourage the use of collaborative teams where all viewpoints and ideas are considered in order to encourage creative thinking and innovation.

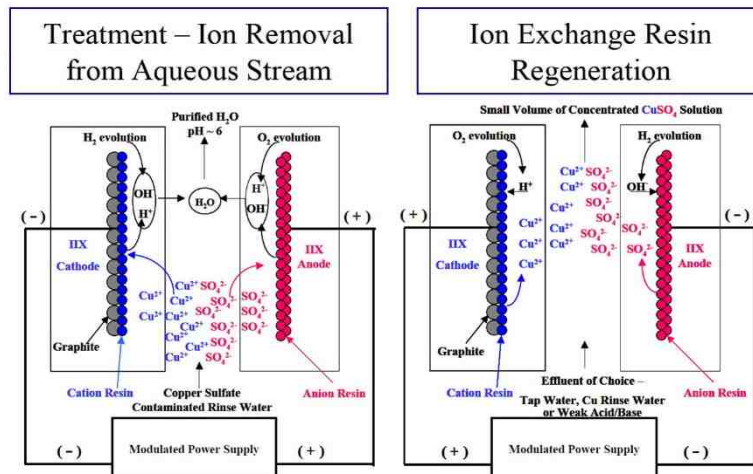


Figure 17 - P/PR-assisted ion exchange for treatment and regeneration of metal finishing process streams.

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The William Blum Lectures

#46 – E. Jennings Taylor – 2007



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About the author

This piece was written at the time Dr. Taylor was announced as the recipient of the 2007 Scientific Achievement Award.



The NASF Scientific Achievement Award is the Association's most prestigious award. Its purpose is to recognize those whose outstanding scientific contributions have advanced the theory and practice of electroplating, metal finishing and allied arts; have raised the quality of products and processes; or have advanced the dignity and status of the profession.

Dr. E. Jennings Taylor, Founder, Chief Technical Officer and Intellectual Property Director at Faraday Technology, Inc., Clayton, Ohio, was selected as the 2007 recipient of the award. The announcement was made at SUR/FIN 2007 in Milwaukee, Wisconsin. His selection was very well deserved, and he joins a list of esteemed technologists in the field of surface finishing.

He has been a leader in several facets of surface finishing research, including metal deposition and several related areas. His work has pioneered the application of non-traditional current waveforms to enhance existing processes and technologies, opening new possibilities for the science and the industry.



The William Blum Lectures

#46 – E. Jennings Taylor – 2007



Specifically, he has been instrumental in taking the concept of using non-direct current in electrochemical processes to fruition. Over the years, enthusiasm for pulse plating, the precursor of what Dr. Taylor has researched, has borne mixed results and had seen little use. A concept of more complex waveforms to enhance and expand electrochemical processes had been proposed but with little follow through. Dr. Taylor took the concept, studied and applied it to the point where it is viable tool that has a major impact of surface finishing technology. Using the concept of “electrically-mediated waveforms,” Dr. Taylor has been the driving force in applying these current forms to electroplating to enhance or otherwise control deposit structure, properties, uniformity and distribution into geometrically difficult areas. The concept has been successfully applied to hard trivalent chromium, electroforming, printed circuitry, fuel cells, nanomaterials and micro-electromechanical systems (MEMS). Beyond deposition, Dr. Taylor has expanded horizons to provide significant enhancements in areas of waste disposal, landfill recovery and ion exchange.

In addition, through founding his company he has successfully continued to provide the valuable need for industrial research laboratories devoted to surface finishing research and development. The highly regarded internal corporate research laboratories of years past have declined precipitously, and Dr. Taylor’s company has provided continuance to this necessary type of resource, in a new model, in the middle of the spectrum between academia and industry.

As Chairman of the NASF/AESF Foundation Research Board, he has revolutionized the operations of the NASF Research program, streamlining procedures and guiding important research. Looking to the future, he has placed equal emphasis on the students doing research as well as the research itself, assuring future R&D human resources for the science of surface finishing.