

**The 17<sup>th</sup> William Blum Lecture**  
**Presented at the 63<sup>rd</sup> AES Annual Convention in Denver, Colorado**  
**June 28, 1976**

## **Electrodeposition and Government Laboratories**

by

**Fielding Ogburn**

**National Bureau of Standards**

*(today, the National Institute of Standards and Technology)*

**Gaithersburg, Maryland, USA**

**Recipient of the 1975 William Blum**

**AES Scientific Achievement Award**





# Surface Technology White Papers

103 (9), 11-21 (October 2016)



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#### ABSTRACT

*Government laboratories have made numerous contributions to the science and technology of electrodeposition in the form of commercial instruments and process as well as engineering data and scientific information. These laboratories and their more significant contributions are briefly reviewed as seen through the eyes of one Government scientist.*

#### Introduction and background

This is a novel situation for me, not only because of the honor bestowed upon me, but because I have a captive audience and I can talk about anything I wish for as long as I wish. At least nobody has told me otherwise. But this freedom given me actually made for a greater difficulty. With over a year to prepare a talk on any subject, I had too much opportunity for procrastination, but now the moment has come.

But first I want to say that this occasion means a very great deal to me. I consider the AES Scientific Achievement Award the highest one I could ever hope to receive, and I never expected it to come to me.

I had the good fortune to work for and with William Blum so there is an added satisfaction and pleasure in giving the William Blum Lecture. It was just 30 years ago that I walked in off the street looking for a job at the National Bureau of Standards. At that time I had not heard of Blum and had not seen a plating bath, but Blum quickly signed me up for what turned out to be a very satisfying career. Satisfying, in large part, because of the knowledge and interest he instilled, but also because of the great respect generally held for my boss.

When I joined Dr. Blum's staff he was already 65 years old. Five years later he reached the age for mandatory retirement of civil servants and he left his office and his old roll top desk but continued his career, as a consultant and by continuing his activities with the AES, ASTM and the Electrochemical Society. Twenty years later he enjoyed a 90th birthday party we gave for him at the Bureau. He was well and lively at that time, but before that winter was over, he had had a heart attack. About that time the ASTM made him an honorary member, but he was never able to receive the award at a formal meeting. When it became evident that he would not be leaving the nursing home where he was living, I took the ASTM plaque to him. He was pleased to receive the award and talked of the history of the Electrochemical Society that he was working on and of his grandchildren, but he was obviously tired after the short visit. He died December 7, 1972.

As I look back on my association with Dr. Blum, I do not recall ever having seen him angered or heard him say an unkind word about anybody or known him to complain about any of the unhappy aspects of his life. He was a generous man, always being helpful and desiring to be of service. When it came to receiving awards and recognitions, of which he received many, his

pleasure was evident; but he received them with great grace. These are a few of the impressions he left with me. And I take great pleasure in delivering, in his memory, another William Blum Lecture.

As I have mentioned, I have been working in the Government on electroplating for over 30 years and as this is a meeting of electroplaters it seems appropriate for me to talk about electroplating and Government. As a title, that is both too restrictive and not restrictive enough for my purposes, I have settled on Electrodeposition and Government Laboratories, even that modification is not completely satisfying. After all, how could I fail to mention electroless nickel? And then there is the question of what constitutes a Government Laboratory. So you may find that my remarks seem to digress a little bit from the stated subject.

It had been my impression that many of you do not realize how much Government Laboratories have contributed to the science and technology of electrodeposition. But as I worked up this talk, I discovered that I did not either. I began by starting to assemble some sort of bibliography of pertinent reports and papers, and soon found this approach to be getting out of hand because of the large number involved. Even the list of pertinent laboratories was longer than I had expected though I had known of each laboratory all along.

## Military laboratories

One obvious characteristic of this list (Table 1) is that most of the laboratories are military in nature. It is true that some of these may not normally be considered laboratories since they are production or manufacturing facilities, but nevertheless, they carried out investigations or development work of significance to the plating community.

One such facility is the Naval Air Station at Pensacola where Jimmy James was responsible for developing cadmium-tin alloy coatings shortly after World War II. Later, the Naval Air Materials Laboratory in Philadelphia evaluated the coating system as did Bennie Cohen at the Air Force Materials Laboratory.

The accomplishments of such "non-laboratory" installations are probably unknown to most of you and would be to me too if it were not for the extensive interaction of the NBS with other Government organizations and for the widespread connections of Dr. Blum. Such interaction introduced me to the Naval Air Station at Alameda. In this instance, the interaction was a matter of the National Park Service obtaining advice from the NBS and the Navy for the gilding by brush plating of four very large equestrian statues. It was then that I learned of Mildred Patterson's contributions to the technology of "selective plating" as it is called in some quarters. Unfortunately the contributions of such facilities usually do not get into the technical literature except as internal reports and specifications, and one wonders how many contributions have been made by these organizations that are directly engaged in the maintenance and manufacture of military equipment.

At another such facility, the Naval Torpedo Station at Newport, Mr. Groff developed fluoborate and fluosilicate baths for depositing a 50-50 lead-tin alloy and subsequently used the process on a commercial scale. Groff got his patent in 1920 and about that time, at the request of the Navy, the NBS carried out an investigation of Groff's process.

Table 1 - Government laboratories.

<ul style="list-style-type: none"> <li>• Frankford Arsenal</li> <li>• Rock Island Arsenal</li> <li>• Springfield Armory</li> <li>• Watertown Arsenal</li> <li>• Watervliet Arsenal</li> <li>• Naval Air Development Center Naval Air Station, Pensacola</li> <li>• Naval Air Station, Alameda</li> <li>• Naval Torpedo Station, Newport</li> <li>• Air Force Materials Laboratory</li> <li>• Sandia Laboratories, Livermore</li> <li>• Sandia Laboratories, Albuquerque</li> <li>• Nuclear Div., Union Carbide Corp.</li> <li>• Oak Ridge Gaseous Diffusion Plant</li> </ul>	<ul style="list-style-type: none"> <li>• Los Alamos Scientific Laboratory</li> <li>• Lawrence Livermore Laboratory</li> <li>• Kansas City Div., Bendix Corp.</li> <li>• Rocky Flats Div., Dow Chemical Corp. (Rockwell International since 1975)</li> <li>• College Park Metallurgy Research Center, Bureau of Mines</li> <li>• Reno Metallurgy Research Center, Bureau of Mines</li> <li>• Boulder City Metallurgy Research Lab, Bureau of Mines</li> <li>• Salt Lake City Metallurgy Research Center, Bureau of Mines</li> <li>• Albany Metallurgy Research Center, Bureau of Mines</li> <li>• Rolla Metallurgy Research Center, Bureau of Mines</li> <li>• Bureau of Engraving and Printing</li> <li>• National Bureau of Standards</li> </ul>
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The primary motivation of the military laboratories is the maintenance, manufacture and procurement of military equipment. These laboratories expend a considerable effort in evaluating coatings for various applications and developing plating techniques. The results can be of considerable significance to our industry, but usually they are not reported in the formal technical literature. An example of such work is the extensive investigations and evaluations, and actual in-house production, of chromium plated gun bores carried out at Springfield Armory, Picatinny Arsenal, Rock Island Arsenal, Watervliet Arsenal, Watertown Arsenal and the NBS during and after World War II. The investigative work of Lamb, Young, Zavarella, Kisner and others was very effective, leading to chromium plating of gun bores on a commercial scale and to gun barrels with a firing life some 30 times that of the unplated barrels. The effects of all the plating variables on the service performance of the chromium coating were evaluated: current density, temperature, solution composition, cell geometry, metal distribution, preplating treatments and so forth. Service performance was judged by actual firing tests with observations of mode of coating failure, velocity of projectiles, path of projectile, and tumbling and wobbling action of the projectile. During that investigation, there developed a much better understanding of chromium plating and coatings.

The Naval Air Development Center has been engaged in electroplating research, development and evaluation for some 30 years. In the electrodeposition literature, you can find about 20 papers by people such as Walter Beck, Ed Jankowsky and Sara Ketcham. Many of these pertain to hydrogen embrittlement resulting from plating processes. Others deal with effects of plated coatings and shot peening on the fatigue properties of high strength steels, chemical films for aluminum alloys and anodized aluminum. Current work is concerned with methods of pollution abatement for plating processes and with coating systems for aluminum alloy electrical connectors, beryllium aircraft brake components, and titanium alloy parts subject to fretting and wear.

At Springfield Armory, during the 1960s and 70s, such people as R. Bessey, W. Kisner, F.X. Hession and A. Zavarella did a lot of work on chromium plating. Their reports cover the plating of gun bores, adhesion, hydrogen embrittlement, analyses of chromium baths, ultrasonics in chromium plating, periodic reverse current and occlusion of hard particulate matter in chromium coatings.

Rock Island Arsenal got into the picture during the war years and is still active. I am sure many of you will recognize Lloyd Gilbert, Stanley Eisler, Russell Wolff and Walter Kisner. Under their leadership, the group has interested itself in many phases of metal finishing. These include phosphate coatings, supplementary chromate coatings, chromium plating, porosity, bath analysis, ion exchange for purification of chromic acid solutions, corrosion of cadmium and zinc, and black chromium. Several papers from this laboratory have appeared in *Plating* and *Metal Finishing*. It was in the 1952 *AES Technical Proceedings* that Gilbert, Morrison and Kahler described in detail their ion exchange installation for purifying chromic acid plating and anodizing baths and its development and evaluation. I understand that similar units are commercially available today.

Those of you who kept up with the technical literature of our field will recognize the names of F.K. Sautter, V.P. Greco, E. Chen and J. Sadak of Watervliet Arsenal. For many years, beginning in the middle forties, this Arsenal has been actively interested in the chromium plating of gun tubes and ordnance equipment as well as other finishing processes. In addition, their activities since the early sixties have included the development and evaluation of new materials for engineering coatings. Beginning in 1963, there has been a series of papers on the electrodeposited dispersion hardened alloys, fiber reinforced electrodeposits, rhenium alloys, and pulse plating. Their work on dispersion hardened alloys has been especially noteworthy and I think it is fair to say that they have been the pioneers in this area. Much of the information we have on this subject comes from Watervliet.

Also familiar to many of us are the publications from Frankford Arsenal. Under the successive leadership of Joe Mazia, Max Frager, George Nordbloom, Anthony Gallaccio and Fred Pearlstein, a small metal finishing group has been able to make significant contributions in the areas of supplementary coatings, electrophoretic deposition of metal powders, coatings on magnesium, electrochemical machining, and electroless plating. This laboratory has been very productive over the years. An early contribution was a variation of the magnetic type coating thickness gage, which was subsequently sold as the Lea Lectromag. I especially remember samples of a very hard coating on magnesium which Max Frager brought to the NBS for us to test and evaluate. There were samples of the HAE coating developed by Harry A. Evangelides and which subsequently became a commercial process. For the benefit of those of you who are not familiar with the process, it is essentially one of anodizing with AC in an alkaline solution at room temperature at about 1.8 A/dm<sup>2</sup>. The coating, mostly magnesium oxide, is brittle, very hard, and corrosion resistant.



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Fred Pearlstein and co-workers have given us substantial information on electroless plating including the boride reducing agent and nickel-tungsten deposition.

The Air Force Materials Laboratory had an active electrodeposition group for a number of years lead by Julius Terres and Bennie Cohen. Their publications cover evaluation of cadmium-tin and zinc-tin electrodeposits, cadmium embrittlement, and hydrogen embrittlement.

## AEC-ERDA Laboratories

In my list of government laboratories are eight (8) that were known as the weapons laboratories of the old AEC, now ERDA. In some circles, these may not be considered as Government Laboratories for they are operated and staffed by non-government people. My excuse for including them is that they have been completely financed by the AEC, but my real reason is that they have been very productive. These eight laboratories and especially Sandia, though oriented towards production of weapons, have been able to go beyond specific production problems, while maintaining an engineering orientation. The interests of this group have been in printed circuits, plating on uranium, joining properties of coatings, electroforming, adhesion, and testing of coatings. Its leaders, for the most part, are known to you: Jack Dini, Harold Wiesner, Rudy Johnson and Bill Harding. They and their co-workers have contributed almost 100 papers and reports to the electrodeposition literature. These have included useful data on properties of nickel-cobalt alloys and a process for plating a lead-antimony alloy. Their most significant contributions seem to be in the area of quantitative adhesion testing and the joining of metal parts by means of electroplating.

One aspect of the military and AEC laboratories is that a significant part of their activities in our field has been the financing of R&D in other Government agencies and outside the Government. I do not intend to say any more about this, but it needs to be mentioned.

## Bureau of Engraving and Printing

Leaving the military activities and going to civilian ones, we come to the Bureau of Engraving and Printing. The plating activity in this organization has been devoted almost entirely to the production of printing plates for currency and stamps ever since its initiation in 1920. Also it has the distinction, I believe, of having been continually engaged in chromium plating the same product line longer than any other establishment - since December, 1925. Such a narrow mission does not lend itself to research, but many of the techniques developed there have been transferred to similar operations elsewhere in the country. For example, it introduced the reproduction of intaglio printing plates by electrodeposition. There have been at least two technical publications from that group. Incidentally, Tom Slattery, who organized and established this activity, was at one time President of the AES.

## Bureau of Mines Laboratories

The interest of the Bureau of Mines in the field of electrodeposition began many years ago with the work of Oliver Ralston on the production of zinc powder and on the electrolytic recovery of lead from brine leaches, which was published in 1916. Since then, they have published some 200 papers on electrodeposition. That the plating community is not generally aware of this work is attributable to two things: 1) it was chiefly concerned with electrowinning and electrorefining, some of it from fused salt media and 2) most of the papers were published, not in conventional journals, but individually as one of the Bureau of Mines series, usually Reports of Investigations. Most of the Bureau of Mines research was done in three of its laboratories: College Park, Reno and Boulder City. Their laboratories at Salt Lake City; Albany, Oregon; and Rolla, Missouri also participated in this work.

The work of these laboratories is, of course, oriented to metal production processes, and it includes pilot plant operation. The investigations, however, include a wide variety of objectives: exploratory work, measurement of properties, test methods and the like. The two laboratories in Nevada have had the able leadership of Sullivan, Cattoir, Hennie, Baker, Couch and Leone. Much of the work at the College Park, Maryland laboratory, under the capable leadership of David Schlain, has been in an area that is more familiar to us. Some of their papers on the effects of ultrasonics on electrodeposition; on an adhesion test; and on the electrodeposition of the platinum-group metals, of titanium diboride and of molybdenum, have appeared in *Plating and Surface Finishing*, the *Journal of the Electrochemical Society*, or *Metal Finishing*. Other papers on porosity measurement of coatings, electrodeposition of tungsten and ultrasonics appeared as Bureau of Mines Reports of Investigations.



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A major contribution of the Bureau of Mines was a process for electrowinning manganese. Early research on this process was done under S.M. Shelton and J. Koster during the 1930s. During the early and middle 1940s, bench scale research was done by David Schlain and co-workers at the Salt Lake City Laboratory while pilot plant operations were in progress at Boulder City, Nevada under J.H. Jacobs. The process has been highly successful commercially.

Another commercially significant process is one for electrowinning chromium which was a development of Lloyd and co-workers at the Bureau of Mines (Boulder) in about 1947. This process deposits chromium from the trivalent ion. Though successful for electrowinning it is not useful for electroplating. More recently, in 1972, high purity chromium was produced electrolytically at the Bureau of Mines. They started with chromium produced commercially from an aqueous divalent bath, and deposited the high purity chromium from a molten salt divalent chromium solution.

Schlain and co-workers of the Bureau of Mines have published some papers on the effects of ultrasonic agitation on electrodeposition processes. Some of these appeared in AES publications and others as Reports of Investigations. They pertained to winning and refining of zinc and manganese and to plating of Zn, Cu, and copper alloys from cyanide baths.

The use of electrodeposition for the recovery of precious metals and other metals from scrap of the electronic industry has also been investigated by the Bureau of Mines. This, no doubt, is part of the government's efforts in the areas of recycling and waste disposal, as has been the work of Andrew Cochran and Lawrence C. George on the treatment of electroplating wastes.

## National Bureau of Standards

Before taking up my own agency, the National Bureau of Standards, I wish to inject just a bit of history. I was recently told that our Government Printing office started its electrotyping operations back in 1869. I suppose that dates the first use of electrodeposition on the part of our Government. I also suppose this activity had some effect on others in the same field, but electrotypers being a somewhat secretive group (so I am told) I doubt that they communicated their experience to others. As far as I am concerned, GPO's major contribution to us was the introduction of Dr. Blum to electrodeposition. In 1913, this electrotyping group had some trouble with their acid copper bath and asked the National Bureau of Standards for assistance. The Bureau did not have anyone who knew anything about electrotyping or electrodeposition, so it assigned a young analytical chemist to the job.

This was Dr. William Blum's introduction to electrodeposition and he was soon busy introducing science to electroplaters and contributing to their technology. One of his contributions was the organizing of an active Section in the NBS devoted to electrodeposition. That group has had a lasting influence on the electroplating industry. Many of the technical advances of the industry can be attributed to Blum's group and a number of the leaders of the plating community were or had been part of that group: Tom Slattery, Abner Brenner, George Hogaboom, Jerry Lux and others. Also Blum and the NBS played a significant role in the establishment of the AES research program and of ASTM Committee B8.

The charter of the NBS gives its activities a wide latitude and Blum's group took full advantage of that by investigating any aspect of the field that seemed of interest. One of the missions of the NBS has always been to assist other Government agencies and over the years the NBS "platers" assisted the Bureau of Engraving and Printing, the Army Map Service, the U.S. Mint, the AEC, the FTC, the Post Office, the National Park Service, the military agencies, the Department of Agriculture, the REA and so forth.

This provided food for a wide and extensive variety of investigations and provided material for over 300 papers. I cannot possibly tell you of each paper and contribution, so let me mention those of special significance and some that have interested me.

In 1933, Abner Brenner, at the NBS, wrote about the stripping method of measuring coating thickness. Subsequently the NBS published on the dropping test, the drop test, Mesle's chord method, a colorimetric technique for gold coatings, x-ray techniques and the microscopical method of measuring coating thickness. In 1937 the Magne-Gage was developed, the first nondestructive method of measuring coating thickness. Its significance must be evident to you for soon after that, coating thickness gages were in wide use. Subsequently Brenner and Garcia-Rivera, and Brodell developed an eddy current gage which also was soon manufactured in a modified form. That was in 1953 and now industry has a variety of non-destructive thickness gages to choose from.

The spiral contractometer for measurement of deposit stresses was introduced to platers at the Atlantic City AES Convention in 1948 where the paper by Abner Brenner and Si Senderhoff received one of the coveted AES awards. The internal stress of deposits from plating baths can be measured by other means and by instruments developed later, but my impression is that the contractometer is currently used more than any of the alternative methods. The newest device, which promises to replace the conventional contractometer, is simply a modification in which an electronic device replaces the old gear mechanism.

Turning to processes, the Bureau of Mines and the NBS both did considerable work on the electrodeposition of metals from organic and fused salt solvents. The metals deposited included titanium, vanadium, tungsten, beryllium, uranium, chromium, rhenium, platinum, molybdenum, aluminum and the rare earths. The most successful process, from the point of view of the AES, is the aluminum plating process announced by Dwight Couch and Abner Brenner in 1952 and, a number of years later, improved upon by Bill Harding at the Kansas City Division of Bendix Corporation, an AEC laboratory. Because the bath uses ethyl ether instead of water, it is regarded by the uninitiated as rather hazardous. Nevertheless, a modification of the process is used commercially at installations which will welcome your orders to plate fasteners and other small items.

Electroless (autocatalytic) plating was a discovery made in 1946 at the NBS by Abner Brenner and Grace Riddell. Of an experiment with a nickel bath to which they had added a reducing agent, they wrote: "In one of the experiments the surprising result of an apparent cathode current efficiency of 130 per cent was obtained, although during the plating there was considerable gassing at the cathode, which usually indicates a low current efficiency. Furthermore, the object being plated, which was a tube with an inside anode, was found to have been completely plated on the outside, although no external anode had been used." I do not think I need to tell you of the commercial significance of electroless plating. During the 30 years since the work of Brenner and Riddell, electroless plating has been the subject of investigation in many private laboratories. From Government laboratories we have contributions by Pearlstein and others at Frankford Arsenal; Dini, Petit, Roberts and others at the AEC weapons laboratories; and Johnson and Ogburn at the NBS. Though there was considerable interest in this process from the beginning, it did not really catch on until General American Transportation introduced its Kanigen process. Interestingly, no one seems to have anticipated its extensive use in the electronics industry or for plating on plastics.

The determination of properties of electrodeposits has been an activity of a number of laboratories: Sandia, Lawrence, Bendix, Frankford, Watervliet, Air Force Materials Laboratory and the Naval Air Development Center. The NBS, of course, has been active in that area also. Much of the activity has been with corrosion properties and in 1925, Dr. Blum began the first systematic exposure tests of plated coatings in the form of a joint project with the AES and the ASTM. These tests formed the basis for the first ASTM plating specifications and initiated a program which ASTM Committee B8 continues today.

In 1947, Brenner, Burkhead and Jennings published the results of their systematic measurement of mechanical and physical properties of electrodeposited chromium, then the only such collection of data. Chromium was deposited under various conditions of bath composition, temperature and current density; the deposits were analyzed for oxygen and hydrogen; and the hardness, tensile strength, density, Young's modulus, ductility, electrical resistivity and internal stress were measured.

Subsequently Brenner, Jennings and Zentner, as an AES Research Project, undertook a still more comprehensive investigation of the mechanical and physical properties of electrodeposited nickel. This was published in 1952 and continues to be a primary source for property data for nickel deposits. The AES Research Committee apparently was impressed by this work for it set up a similar project at the NBS on copper. Vernon Lamb and co-workers published their compilation, covering some 62 pages, in 1970. This was followed in 1974 by publication of property data on brass, obtained by Chris Johnson and myself in the course of a much less ambitious project, also supported by the AES. Then very recently, Chris Johnson and I have published some data on the hardness of electroless nickel-phosphorus after various heat treatments.

Sometimes I feel that Charles Kasper's work, in 1940 on current distribution, has been forgotten. It should not be, however, because it was a historical landmark. It gave us a firm foundation that was lacking and sorely needed, for understanding a very practical problem. What he did was to take existing theory of electricity and show how it could be used to calculate current distribution in the absence of polarization.

The technology of chromium plating was aided by the Government laboratories from the beginning when Dr. Blum's group did its part in making chromium plating a widely used commercial process. In 1924, his group was striving to find out what made the

process took. By the end of 1925, the Bureau of Engraving and Printing, with the assistance of the NBS, had begun to chromium plate its printing plates. That was probably the first such application of chromium plating. Then in 1925 and 1926, Haring and Barrows at the NBS published their papers which put the process on a firmer foundation. These were followed in the early 30s by papers on throwing power and on the conductivity and density of the chromic acid solution. During the same period, the Public Health Service published on the health hazards of chromium plating and the NBS conducted several investigations on the chromium deposits. The latter included studies on structure, wear resistance, porosity, and protective value of chromium-nickel coating systems. Also Kasper published his paper on the structure of the chromium bath and the mechanism of the deposition process and Blum and Olson worked out the spot test method of measuring decorative chromium thickness.

The interest in chromium plating continued after World War II. Brenner, Burkhead and Jennings published their data on the properties of chromium electrodeposits and Lamb, Young, Seegmiller and co-workers continued the cooperative program on gun bores. During this post war period, I became interested in the possibility of using radioactive tracers to find out more about the mechanism of chromium plating. Since I had never done any chromium plating at that time, I welcomed the assistance of Abner Brenner and between the two of us, we demonstrated that the trivalent chromium that forms during chromium plating is a by-product which is not further reduced to the metal.

Later, Lamb and Krasly designed a gage for measuring the thickness of chromium in tube bores and, more recently, Young showed how to deposit chromium with inclusion of hard particulate matter. Also Logan investigated the effects of chromium plating on the fatigue properties of the steel substrate.

Brenner's papers on the composition of the cathode film were very informative and were a significant addition to the plating literature. The experimental techniques he used initially was to plate onto a hollow cylinder. When the plating process had reached a steady state he poured a freezing mixture ( $-78^{\circ}\text{C}$ ) into the cylinder to freeze the electrolyte near the cathode and then machined off successive layers of the frozen cathode film. By analyzing each layer, he determined the composition of the cathode film as a function of the distance from the cathode.

Now the electrodeposition activities of the NBS did not include the writing of books. Nevertheless, Blum and Brenner each wrote a book on their own time. *The Principles of Electroplating and Electroforming* by William Blum and George Hogaboom has had three editions and has been very widely used. Abner Brenner's two volume tome on *Electrodeposition of Alloys* is a classic and is indispensable to anyone concerned with alloy plating.

Speaking of alloys, the NBS has been active in that area, too. In its list of publications we find titles on lead-tin; iron-, nickel- and cobalt-tungsten; cobalt molybdenum; nickel-aluminum; nickel- and cobalt-phosphorus; and brass.

## Personal reminiscence

Before concluding this William Blum Lecture, I will briefly describe two activities of my own. Neither are of especial significance to the scientific world, but they represent fun in the career of a Government chemist.

The first is fun because it is not burdened with the administrative aspects of a formal project. No deadlines, no project proposals, no progress reports, merely a struggle to find time to squeeze in the work.

It concerns the measurement of the thickness of a plated coating with a microscope. When I was introduced to electroplating, the microscopical method was the method. Most coating specifications cited it as the referee method and it was generally held in high esteem. Then we learned that the method was not so good. Reports came in that the method was not as reliable as other methods. As a referee method, it was dropped from specifications. An ASTM round-robin showed that five men with lots of experience with the method, when measuring the identical specimen, differed by as much as seven per cent with thick coatings and  $1\text{ }\mu\text{m}$  ( $40\text{ }\mu\text{-in.}$ ) with thin coatings.

This intrigued me because men regularly using the method swore by it and the repeatability of a measurement is demonstrably much better than seven per cent or  $1\text{ }\mu\text{m}$ . At that time, I had very little if any experience with the method, but I set out to satisfy my curiosity. And it did not take long to uncover the difficulty. That difficulty was that an evaluation of the method, with good discussion of the techniques of the method or of the causes of errors, was not available. People were using the method without

an understanding of the pitfalls and without ever checking their technique against an independent measurement. Under such circumstances, it was to be expected that the same errors would be made over and over again without any realization that an error was occurring.

Subsequently I did identify several possible sources of errors attributable to the technique of using the microscope, sources of which some experienced metallographers were completely unaware. As yet I have not had the opportunity to examine the errors arising from mounting and preparing the specimen. But I can report to you that accurate measurements can be made consistently if one takes a little care and makes an effort to avoid the known possible errors. To demonstrate this we measured the thickness of chromium-copper coatings of  $3 \times 3$  cm. squares of steel; first with a microscope, then by weighing the specimen before and after stripping the coating and calculating the thickness from the weight loss, density and area. The technique we used for the microscopical measurements were not those normally prescribed. Contrary to published recommendations we did not overplate the coatings; we did not etch after polishing; the mounting, polishing and measurements were carried out by a technician whose only metallographic experience was but one session with a practice specimen; the specimen was clamped and not conventionally mounted in a press or mold; and the optical systems had limits of resolution of 1.0, 3.8 and  $1.2 \mu\text{m}$  ( $0.6\lambda$  / N.A.). According to some people, we could not possibly have had an accuracy better than the limit of resolution.

Microscopical	Stripping	Difference
1.606 mm	1.604 mm	0.1%
1.053 mm	1.056 mm	0.3%
$267.2 \mu\text{m}$	$266.9 \mu\text{m}$	0.1%

In my opinion, the discrepancy within each pair of values may represent the uncertainty of density of the electrodeposited copper. I am not at all sure that it is due to the microscope and it remains to be seen how good the comparison can be for thinner coatings.

I believe we have demonstrated that microscopical measurements can be quite accurate, but I hope you will remember that this does not mean such measurements are generally that reliable.

At this point, I call your attention to the gilded equestrian statues I mentioned a few minutes ago. The restoration of these statues has been described in *Plating*, but this represents the subject of a fun project and I wish to tell you a little bit about it. It was fun because it was a radical departure from our routine and got us out of the laboratory.

These statues were badly deteriorated, both finish-wise and structurally. The National Park Service in Washington sought advice and guidance about their restoration from a number of individuals and organizations without much of any satisfaction. Then they came to the NBS. Restoring statues was foreign to us, but we thought we could offer some useful advice. We easily diagnosed the cause of the deterioration: the gold layer over the bronze castings was discontinuous and bronze corrosion products were forming and covering the gold; also the bronze castings were held together by steel bolts which were rapidly corroding away. The course of action to be taken was clear enough except for the method of refinishing the gilded surfaces. Originally the statues had been gilded by painting with a paste of gold amalgam and then evaporating the mercury with a blow torch. Since we felt this was a bit hazardous we did not give it much consideration. Cleaning and lacquering was suggested. And we talked of removing the old gold finish and letting an "attractive patina" form. I spent some time learning all about gold leaf and I had several interesting conversations with people from various states where the Capitol Buildings were surmounted with gold domes. I even got an estimate for building a tank around a statue and going to electroplating. Then somebody advised me to get in touch with Mildred Patterson at the Naval Air Station in Alameda. Her advice convinced the Park Service that brush plating was the answer and it was not long until a professional brush plater was given a contract to oversee the regilding of the statues. This application of brush plating is unique and has attracted considerable interest, but I was a bit shaken to read in one news account in an English trade journal that two tons of gold had been applied to these statues. I think someone must have mixed English and metric units. In reality, about 40 kg of gold were used to give an average thickness of about  $4 \mu\text{m}$ .

## Conclusion

During the preparation of this lecture, I became more and more impressed by the quality and quantity of the contributions of the various Government Laboratories. I could tell you much more than I have, but even then I would have conveyed to you only a small part of the whole story. I hope, however, that what I have said convinced you that these laboratories have been very

productive. They have contributed useful instruments and test methods, widely used plating processes, useful data, to the understanding of plating and plated coatings, and to the evolution of the industry and science over the years.

## About the author

*This piece was written at the time Mr. Ogburn was announced as the recipient of the 1975 Scientific Achievement Award:*



Fielding Ogburn is the choice of the AES Scientific Achievement Selection Committee to be the 1975 recipient. The announcement was made at the opening session of the 62<sup>nd</sup> Annual Technical Conference of the American Electroplaters' Society in Toronto, Canada, on June 23, 1975.

Mr. Ogburn was born in Philadelphia, August 20, 1919. During preschool years, he lived in New York and France. Most of his schooling was obtained in Chicago, culminating with a B.S. in chemistry from the University of Chicago in 1941.

He married Patricia Daly in 1942 while working in Midland, Michigan. After discharge from the Army in 1946, Mr. Ogburn joined his wife in Washington, D.C., where she had been working as an economist in the Office of Price Administration, and accepted a position at National Bureau of

Standards. They still live in the Washington area where they brought up two boys who are now grown and no longer live at home.

Mr. Ogburn continues to work in electrodeposition at the NBS. Until their respective retirements, he was associated with William Blum and Abner Brenner, first and fourth recipients of the AES Scientific Achievement Award. Researches at NBS have resulted in the publication of about 50 papers in the field of electrodeposition. These have covered such varied subjects as mechanism of chromium deposition, mechanical properties of brass, structure of dendrites, galvanic corrosion, porosity, measurement of coating thickness, acceptance sampling, surface roughness, metal distribution, electrolytic coatings on magnesium base alloys, hardening agents, etc. These include his publications as director of AES Research Project 13 on "The Nature, Cause and Effect of the Porosity in Electrodeposits," Project 19 on "Galvanic Effects Associated with Coating Failure," and Project 33 on the "Development of Property Data for Electrodeposits."

Mr. Ogburn is a member of American Electroplaters' Society, Institute of Metal Finishing, American Society for Testing and Materials, American Chemical Society, and The Electrochemical Society. He served two terms as chairman of the local section of The Electrochemical Society and served on various of its committees and the Electrodeposition Division. He was co-chairman of its recent symposium on the "Properties of Electrodeposits - Their Measurement and Significance" and is co-editor of the symposium volume.

He has been active on ASTM Committee B-8 on Electrodeposited Metallic Coatings and Related Finishes since the early fifties. At present, he is chairman of its subcommittee on Government Specifications, of the working section on coating thickness measurement, and of the Metrication Section. The ASTM duties include service as a delegate to the corresponding committee and subcommittees of the International Standards Organization and this has taken him to Italy, Poland and England.

Mr. Ogburn attended his first meeting of the AES Baltimore-Washington Branch a week before starting his career in electrodeposition at the National Bureau of Standards. With the encouragement of Thomas Slattery, William Blum, Kenneth Houston, and Arthur Pierdon, he served in the various offices of the Branch, and as its president, and as a delegate. He later served on the Books and Symposia Committee of the National AES when he coordinated the efforts of the American Electroplaters' Society and the Society of Manufacturing Engineers in the preparation of *Surface Preparation and Finishes for Metals*, a book edited by James A. Murphy of the SME. He subsequently served as technical chairman of the Fourth Plating in the Electronics Industry Symposium and organized a one-day symposium on the measurement of coating thickness. He also served on the AES Scientific Achievement Award Committee several years ago.

A recent but unusual assignment, which he enjoyed and which has led to several talks to non-technical groups and to the preparation of a 15-minute film, was as a consultant to the government agency responsible for the statuary in Washington. He



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provided technical advice on the restoration of four monumental statues facing the Lincoln Memorial which were suffering from severe internal and external corrosion problems. The information he provided lead to the regilding of the bronze statues by selective (brush) plating with gold.