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Electronics II
Session E

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Electronics II
Session E

Innovative Control Systems for the Electronic Plating Process—Today and the Future

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INNOVATIVE CONTROL SYSTEMS FOR THE ELECTRONIC PLATING PROCESS - TODAY AND THE FUTURE -

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1.0 General

The control system for the electronic plating process must be sophisticated enough to handle many complex processes simultaneously, while still having a simple and easy to use operator interface.
Among the tasks to be performed are the closed loop control of process parameters, chemical handling, data collection capabilities, the analysis of collected data, the control and tracking of parts through the process, as well as several other tasks.
All of these will be discussed in this paper.

1.1 Abstract

- As an important part of an automated plating facility, the control system for an automated plating line must have certain characteristics that allow it to be easily integrated into the overall facility control system.

- It is designed to automatically route parts to and from the plating line, automatically monitor and control the flow of chemicals to and from the plating line, and automatically monitor and control the process through which the parts pass.

- To accomplish this the plating line will need a closed loop control of process parameters, chemical handling (flow monitoring and control) capabilities, data collection capabilities, and the ability to communicate with a host computer or network.

- It is the intention of this document to describe a control system that accomplishes the above.

2.0 Control System Description

The plating line control system will have the following capabilities and utilities available.

2.1 **Dynamic Processing**

- The "Dynamic Scheduler" uses tank sequence, desired dwell times, and some ancillary data about the tanks, (i.e. tolerance for dwell times), to interactively create new hoist programs for any series of processes to be run.

- The system computer accepts requests for new processes from the line operator, and using the data files, design a new program for the hoist. This allows programming of new process recipes without interrupting the regular operation of the line.

2.2 **Closed Loop Control**

- The plating line will have closed loop control of important process parameters and variables. The control loops will be configurable to provide the necessary sensors and signal conditioning. The control parameters will be user alterable. Examples of control parameters include set points, alarm points, dead bands, etc.

- In addition, the control system will monitor variations or deviations of the controlled parameters from the desired values. Thus closed loop control also provides the capability for data collection.

3.0 Master Controller

The master controller controls programmable line parameters such as temperature, conductivity, pH, bath replenishment, rectifiers, and other ancillary equipment. It monitors the operation of the other controllers and coordinates their actions to operate rectifiers and bath replenishment systems. It also logs system activity by filing and/or printing collected data. Multiple hoist can be controlled by the system.

3.1 **Control**

- All line setpoints can be programmed 'offline' without changing setpoints in use.
- Process engineers can define new processes while the line is being used.
- All line setpoints can be changed through the master console.
- Inputs to the line computer may be provided as 0-5V, 0-10V, or 4-20 mA signals.

3.2 **Temperature Control**

- Values specified and displayed in deg C or deg F.
- ON/OFF control of heaters and coolers with dead band programmable by user, or PROPORTIONAL control using PID control algorithm.
- Warning levels programmable by user. Alarm inputs may be used to shut off outputs.

3.3 **Conductivity Control (Rinse Water)**

- Selective full scale range (50, 2000, 5000,...)
- ON/OFF solenoid control with dead band programmable by user.
- Proportional control with user alterable gains.
- Warning levels programmable by user. One discrete alarm input per conductivity input, may be used to disable controlled output.

3.4 **pH Control**

- ON/OFF solenoid control with dead band programmable by user.
- Proportional control with user alterable gains.
- Warning levels programmable by user. One discrete alarm input per conductivity input, may be used to disable controlled output.

3.5 **Bath Replenishment**

- Replenishment based on continuous analysis via interaction with instrumentation, i.e., Ion Chromatography Colorimeter, Polarograph, etc.
- Setpoints for all replenishment modes are programmable by the user.
- Replenishment 'on time' is programmable by the user in 1 second increments to 255 seconds.

Optional

- Replenishment on accumulated surface area which has passed through tank.
- Replenishment on accumulated amp-seconds in electrolytic tanks.
- Replenishment on accumulated time since last replenishment.
- Replenishment on accumulated immersion time since last replenishment.

3.6 **Ancillary Control**

- Programmed ON/OFF control for pumps, filtration systems, main water inlets, blowers, bubblers, mechanical and air agitators and the like.
- One discrete alarm input per ancillary, may disable controlled output.

3.7 **Flow**

- Flow rates are only monitored and displayed, (not controlled).
- Warning levels programmable by user. One discrete alarm input per flow input.

3.8 **Pressure**

- Pressures are only monitored and displayed, (not controlled).
- Pressures may be either single ended or differential.
- Warning levels programmable by user. One discrete alarm input per pressure input.

3.9 **Spray Rinse**

- Sprays can be actuated when work is entering a station, leaving a station, while in a station, or any combination of those options.
- Sprays for entry, exit and while in a station, may be from different solutions.
- One discrete alarm input per controlled spray, may be used to disable controlled output.

3.10 **Rectifier Control**

- Up to 72 unique power supplies may be controlled and monitored
- Up to 8 independently controlled rectifiers per station.
- Current range dependent on power supply used.
- Measured voltage displayed.
- Measured current may be displayed.
- User programmable delay-time and on-time for each rectifier.
- User programmable warning levels for measured voltage.
- One discrete alarm input per rectifier, may be used to disable controlled output.

4.0 Data Collection

The control system will collect data about events which pertain to the effective and efficient operation of the plating line. There are two kinds of events, process data events and process control events. One example of a data event of interest is the deviation of a controlled variable outside some defined limits.

4.1 An example of a control event would be the creation of a new bath.

- Another example of a control event would be the completed plating of a set of parts.

- The following is a list of the required events and status information to be available (perhaps as records in a file on the hard disk).

- a) Deviation of any controlled variable outside defined limits. This implies the capability to download these limits as well as the control parameters

- b) Current physical status of the plating line, hoist system, and automatic load and unload systems.

- c) Creation of new chemical baths or changing of tank chemicals with the chemical lot information.

- d) Location of parts within plating line.

5.0 Data Trending

Software includes the data trending feature which saves all sampled data for a period of approximately two weeks in a series of disc files. These files can be copied onto floppy discs, sent to a host computer, printed and/or displayed on the color graphics monitor.

The user may select sample points between 1 and 60 minute intervals. This sample record will contain the minimum, maximum and mean values for the previous sample periods. Should a load enter or leave a station during the sampling period, the sample record will be produced that covers from the start of the sample period until the time that the load entered or left the station.

5.1 **The file structure is:** TRymmddp.dat where

a) y is last digit of the year, 6 for 1986
b) mm is the month from 1-12
c) dd is the day of the month
d) p is the part of the day (A-Z)

Note: The files will be less than 300k bytes each so that they will fit onto a floppy disk.

5.2 **The general record format will be:**

<record type>;<data>....<date>;CR LF

Note: All the data will be in metric units

5.3 **Specific Record Formats**

a) Workbar lot information records. Recorded when a bar is loaded:

bar;<in|out>;<number>;<hours>;<minutes>;<seconds>;
<qty>;<lot>;<qty>;<lot>;<qty>; CR LF

i.e., bar;in;14;22;54;13;13542;
100;11432;10;13425;10;CR LF

b) Data record. Recorded at sample period or when a bar leaves a tank:

data;<station>;<parameter>;<hours>;<minutes>;<seconds>;
<min>;<mean>;<max>;<status>;<bar>;CR LF

i.e., data;N34;temp.,tank;12;10;15;
120.1;134.2;144.3;green;14;CR LF

5.4 **Hoist Interface** - optional

The control system can be connected to a host computer via an RS232 line. The host will send a message, "Send ymmdd Nongreen", this will start transmission of all nongreen parameters for the specified (ymmdd) date. The record will be sent and the system will wait for an acknowledgment of receipt prior to sending the next record.

5.5 **Reports** - optional

A report will be produced wit either all the data, all non-green data or all red (out of limit) data. This report can be directed to the local printer or to the host computer.

a) By lot number:
Data for lot 1234567:
Time in MM DD, YYY:
Workbar 34 first 32 boards
Workbar 45 second 54 boards
Workbar 15 third 20 boards

		GROUP 1 WORKBAR 34				GROUP 2 WORKBAR 45				GROUP 3 WORKBAR 15			
STA	PARAMETER	MIN	MEAN	MAX	STAT	MIN	MEAN	MAX	STAT	MIN	MEAN	MAX	STAT
13	Temp..tank	34	38	45	RED	39	40	41	GRN	39	40	41	GRN

b) All data:
From MM DD,YYYY HH:MM,YYYY HH:MM

STA	PARAMETER	TIME	MIN	MEAN	MAX	STAT	BAR
13	Temp..tank	23:15	34	38	45	RED	3

The actual format will be ##,###,## for all parameters. This report will be 132 columns.

5.6 **Color Graphics Display of Trended Data**

Up to three parameters may be plotted with the total period for the display from 1 to 14 days. For each parameter there will be three curves: the upper curve defining max values, center curve for the mean values and the bottom curve for the minimum values. The plating line will be displayed at the bottom of the screen while the trended curves are displayed.

Real time trending of parameters is optional. This option allows the user to see data points plotted as they are collected. A moving bar erase will be used to maintain current data on the screen.

6.0 The control system will accomplish the data collection, and provide overall facility control and incorporate the plating line controller into a communications network. An additional computer will be used to implement the line control.

7.0 PARAMETERS CONTROLLED

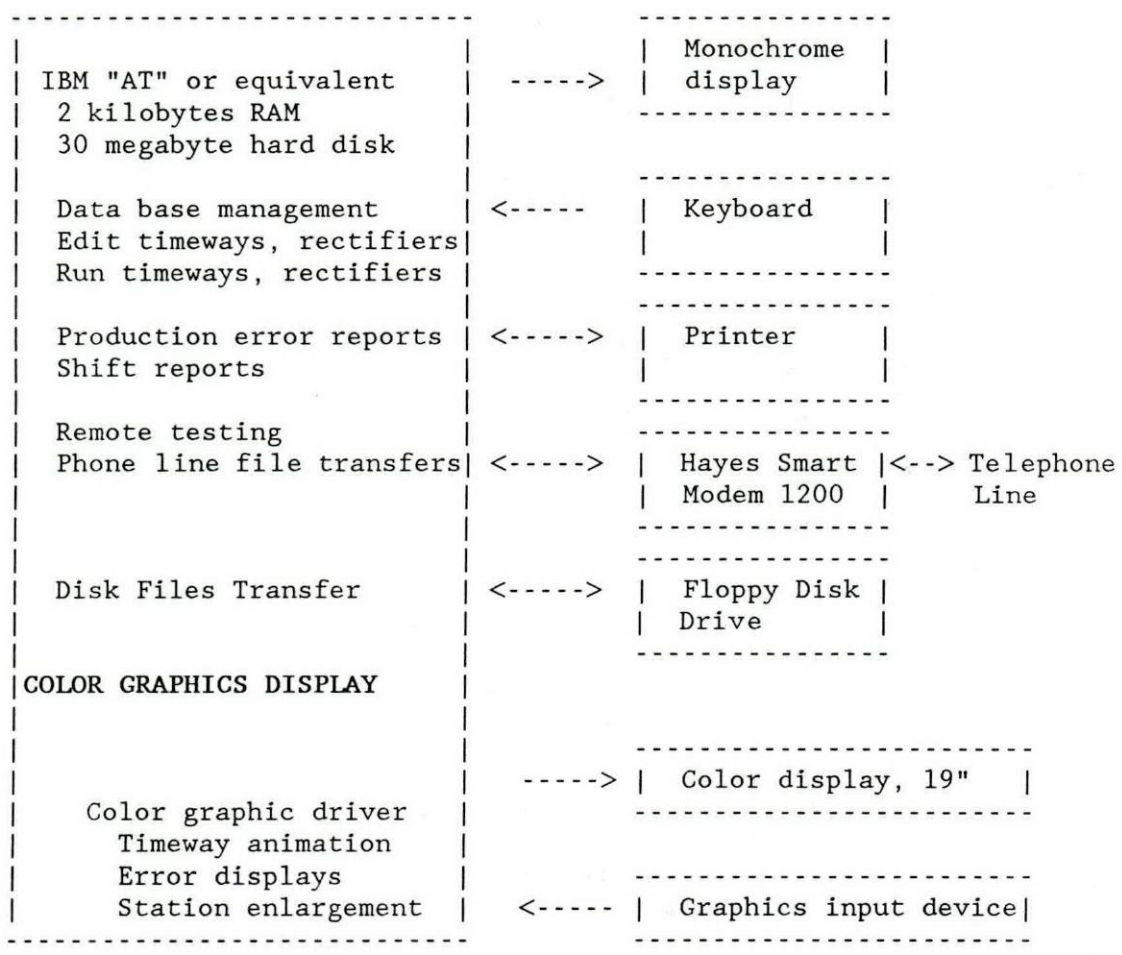
The following chart indicates which functions will be controlled by the control system and whether they will be controlled by value or on/off.

Parameter	On/off	Value
A. Temperature		^{o}F or ^{o}C
B. Conductivity or Resistivity		mhos
C. Flow	X	
1. GPM may be recorded		Optional
D. Level Controls	X	
1. Level status	X	Optional
E. Mechanical Agitation	X	
F. Rectifiers		
1. Current or voltage (both are recorded and limited)		
2. Ramp rate		
3. Amp-minutes		
G. pH		pH units
H. Ancillary Controls or Off-Line Devices (i.e., automatic analytical instrumentation, chemical feeds, automatic loading/unloading, etc.)	X	X

The values for your system will be input in a spreadsheet format and are alterable by the user with the correct password access.

CONTROL SYSTEM

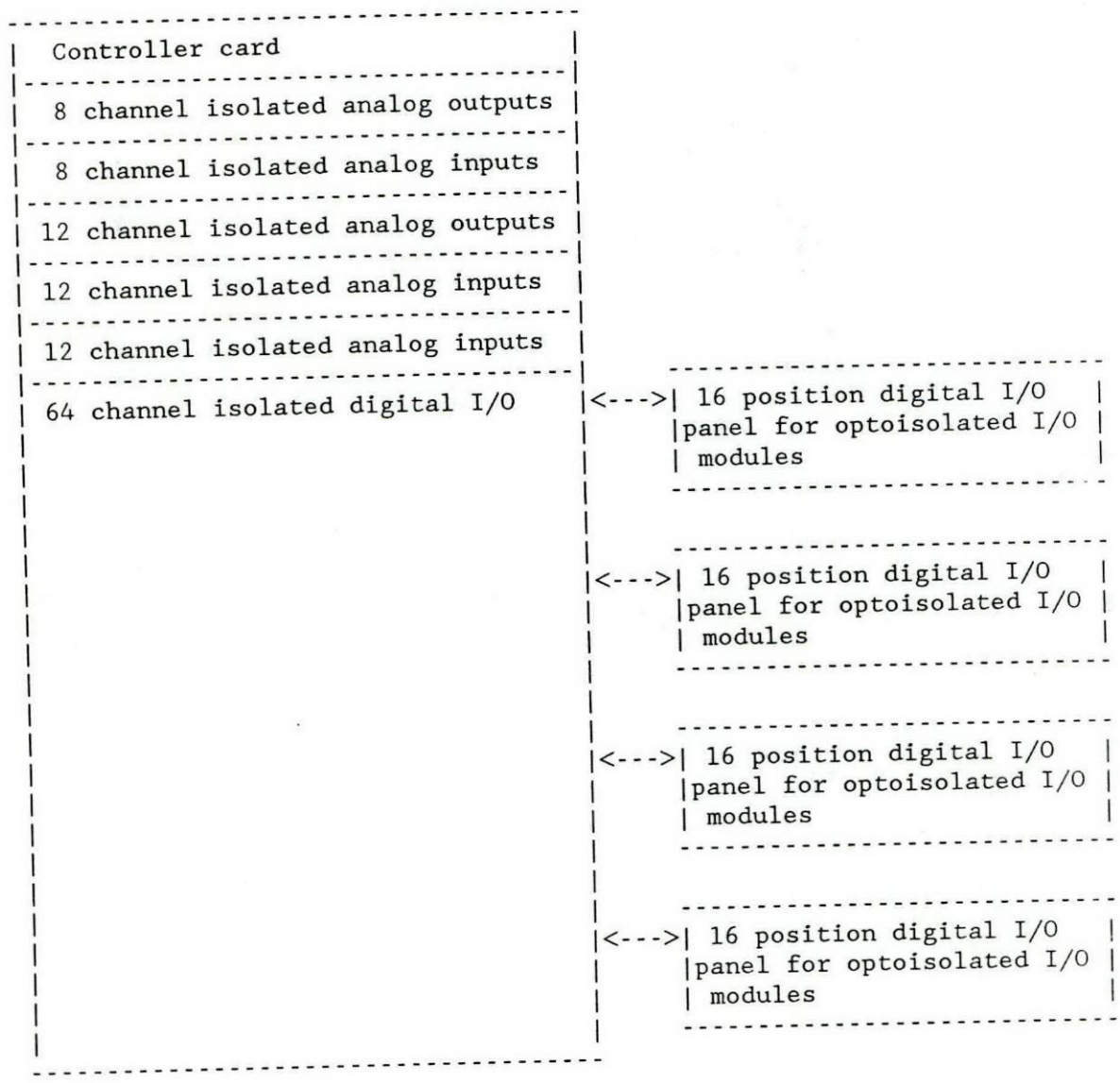
SYSTEM MANAGER



HARDWARE

Licensed Software Included
Battery Backed Clock
Also Provides for RAM Expansion
Modem
Monochrome Graphics
16 Channel Digital I/O
Require Clock Board

I/O SYSTEM



Control Card and Card Cage
8 channel isolated analog output (temperature control)
8 channel isolated analog input (flow counting)
12 channel isolated analog input (RTD temperature, pH, conductivity)
64 channel isolated digital input/output

CONTROL SYSTEM

Some features of the system are:

* All analog inputs are isolated to 1000 volts
  This is very important on the 60 mVolt current shunt input

* All analog inputs are filtered to 1 Hz
  This is very important in an industrial environment

* All analog outputs are isolated to 1000 volts
  This is very important on the control outputs to the rectifiers

* All the computer and analog I/O hardware is off the shelf.
  The computer hardware has local service in most cities.
  The analog I/O hardware has local service some cities

* The system is easily expandable to several computers for larger installations. The computers are all connected together with a local area network which is a high speed serial communications network using a coaxial cable

SPECIFICATIONS:

Analog Inputs ----------

Isolation 1,500 volts channel to channel and input to output
Maximum differential voltage 130 V rms without damage
CMRR with 10 ohm unbalance, 60 Hz is 120 db.
Inputs filtered to be down 3 db at 3 Hz and 160 db at 60 Hz
Accuracy better than 0.1% of full scale

Signal
+ - 50 mV for current sensing
+ - 10 V for voltage sensing. Larger signals may be divided down.

Analog Outputs ----------

Isolation 1000V channel to channel and input to output
Maximum differential voltage 130 V rms without damage
CMRR with 10 ohm unbalance, 60 Hz is 120 db.
Inputs filtered to be down 3db at 3 Hz and 160 db at 60 Hz

Signal
0 to 5 V with an output impedance of 250 ohms to control rectifier voltage

Digital Inputs ----------

Isolation 2500V channel to channel and input to output

Signal
90V to 140V AC
180V to 280V AC
10V to 32V DC

Digital Outputs ----------

Isolation 2500V channel to channel and input to output

Signal
12V to 140V AC
24V to 280V AC
0V to 60V DC

E-2

Electronics II
Session E

A Methodology for Laboratory Evaluation of Solder Electroplating Baths

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A METHODOLOGY FOR LABORATORY EVALUATION OF SOLDER ELECTROPLATING BATHS

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ABSTRACT

A low-cost, but effective methodology was designed to examine those aspects necessary to a comprehensive evaluation of solder electroplating baths for printed wiring boards. The evaluation included examining electrodeposits on test panels and selected circuit patterns by traditional and specifically developed techniques. Systematic examination of data, image analysis, and carefully chosen statistical analysis yielded a strong indication for a best first choice production plating bath. Moreover, this investigation precipitated the development of a comparative plating bath evaluation methodology which has capability for more general application.

I. INTRODUCTION

Selection of an electroplating process for high volume routine manufacturing use is always a risky and tenuous endeavor. The usual practice is to perform laboratory experiments as a preliminary step in the selection process. Unfortunately, a laboratory scale examination can often yield results that are not truly representative of what occurs in production size facilities. Hence, orderly comparative studies are typically necessary.

While engineers in the PWB industry periodically examine alternative or "improved" electroplating baths, there appeared to be a definite lack of a formalized general methodology which could be readily applied to facilitate the formation of a plan of comparative evaluation to address the specific needs at hand. When this investigation was initiated, it was approached with the idea that the experimental design should be of such a nature that a general investigative methodology might be abstracted from the design and formally documented.

The purpose of this paper is to present that methodology to help define the details of laboratory investigations which may enhance the probability of successful scale-up to manufacturing levels. Solder plating was the vehicle chosen to exemplify the methodology since the field of alloy plating is more complex than single metal deposition.

II. EXPERIMENTAL

A. Formation of List of Attributes

The first step was to compile all of the attributes of solder electroplating performance which must be included in order to make a sound decision on the relative performance of the baths under study. Collective engineering judgement, product requirements, and manufacturing experience were the prime tools employed in this phase. In general, the attributes which are selected will, of course, depend on the particular requirements on the product being manufactured, any manufacturing problem areas which need to be specifically addressed, and the manufacturing and environmental considerations prevalent at the plant location.

Some of the attributes chosen will most probably be those of typical concern such as throwing power and variation in alloy. Gathering of data in the established attribute areas can usually be accomplished by established methods, e.g., metallographic cross-sections, atomic absorption, etc. However, in

addressing the more sophisticated requirements of advanced product designs or eliminating weaknesses in a manufacturing sequence, it can become necessary for the investigators to develop specific additional attributes as well as associated methods of testing in order to obtain the proper data to meet the needs at hand. This was true of the evaluation discussed in this paper.

It must be emphasized that a list of attributes can become unnecessarily long if all possible items which might conceivably receive consideration are included. Therefore, any investigative team would find it profitable to reconsider selected items for deletion if their outcome is deemed predictable with an adequate degree of certainty or if their expected impact on the results is deemed minor. Usually available time, prior experience, or economic considerations will govern the extent of judicious experimental "trimming."

Using the techniques described above, the attributes specific to this comparative solder electroplating bath investigation were developed. The more traditional concerns included:

a. alloy composition
b. surface electrodeposit thickness
c. through-hole electrodeposit thickness
d. resultant throwing power
e. the distributions of the values of the above four items

The other concerns which were directed toward specific product requirements and which may have required specific development of analytical procedures as well as development of the attributes themselves included:

a. surface morphology
b. chemical nature of the surface
c. organic coating adhesion

B. Test Panels

Three different panel configurations were used during the plating experiments. The first was a three-inch wide strip of copper clad epoxy glass substrate. Once the designated solder plating runs were completed, these strips were used for three different tests. Firstly, they were subjected to an examination of the surface topography by scanning electron microscopy to determine the nature of the surface morphology and to observe the consistency of that morphology throughout the

plating tests. Secondly, portions of these panels were subjected to a surface analysis for a particular attribute associated with a specific requirement on a portion of the PWB product. This analysis was specifically developed for this investigation. The values achieved in this analysis allowed the test baths to be rank ordered according desirability of performance. Finally, the remaining portions of these panels were subjected to organic coating adhesion tests according to ASTM Test Designation D3359-83. This designation is essentially an assessment of adhesion by a tape test.

The second test vehicle was a quarter of a production panel of a circuit pack code which had gone through production copper plating but was removed from production processing immediately after the rinse following copper electrodeposition. The code that was chosen had a circuit configuration which was typical of a significant part of normal production. It had a relatively homogeneous circuitry distribution, but also an area of nonuniformity in order to create a skewed electrical field on a portion of the circuit pattern within the plating cell.

The third test vehicle was a quarter of a production panel of a thick backplane code where greater than 80 percent of the panel was exposed area (i.e., not covered by photoresist). This test vehicle was also intercepted immediately after the rinse following copper electrodeposition. This code was also chosen because it is typical of a significant portion of normal production.

Both sets of quarter panels were used for the same types of examinations:

1. alloy composition and the distribution thereof by differential thermal analysis, [1]

2. plating thickness on path and land surfaces and the distribution thereof by light section microscopy,

3. through-hole plating thickness and the distribution thereof by metallographic cross-sectioning,

4. throwing power by the ratios of the surface and through-hole plating thickness measurements.

A number of measurements (typically eight) were made for each of the above categories for each quarter-panel plated. Moreover, the sites where the samples were taken or measurements were made on the quarter-panels were carefully chosen to

include areas which represented extremes of current density for the specific circuit configurations chosen as the test vehicles.

C. Experimental Electroplating Cell

The experimental plating cell was designed and constructed specifically for this investigation. The cell was designed around the concept of plating the lower half of a quarter-panel on both sides with anode arrays symmetrical to a centerline lying within the plane of the cathode and perpendicular to the surface of the electrolyte. The tank itself was constructed of polypropylene. Teflon guides were fitted relatively snugly along both longer dimensional sides of the tank so that when the quarter-panel was inserted into the guides all the way to the floor of the tank, the plating cell was essentially divided into two separate mirror-image sub-cells. Agitation was provided for each sub-cell by medium-duty, variable-speed lab stirrers with polypropylene shafts and impellers. Short length tin/lead anodes, cut from regular shop anodes, reaching to approximately three-quarters of an inch from the tank floor were hung from anode bars which were parallel to and close to the short dimensional ends of the tank. Electrical contact from the rectifier was made with appropriate wires and alligator clamps which were kept meticulously clean throughout the entirety of the laboratory experimentation. The working volume of plating solution in the tank was 12 liters. The experimental cell is illustrated in Figure 1 and Figure 2.

Figure 1 shows the electroplating setup for the three-inch wide test strips. During this portion of the experimentation an unclad epoxy quarter-panel was inserted into the Teflon guides to divide the cell into the aforementioned two sub-cells. Two strips were clamped onto both sides of the insulating quarter-panel (one in each sub-cell) and plated simultaneously during a single experimental run. One anode was hung from each anode bar directly in line with the corresponding strip in its sub-cell.

Figure 2 shows the plating setup for plating the imaged and copper-plated quarter-panels. Here the quarter-panel itself divides the cell as mentioned before. In this case two anodes were hung from each anode bar, evenly spaced across the width of the tank.

As with all plating tests, various ancillaries were required in conjunction with the cell itself. In this case, one acidic cleaning tank, one deionized water pre-plate rinse tank, and

one city water post-plate rinse tank were employed. The polypropylene rinse tanks were placed on a large tray in which a corner drain had been fabricated so that the rinses could be operated in an overflow fashion. The acidic clean tank was placed in between the rinse tanks for convenience. When it was required, electric heaters were used for heating of the city water rinse and warming the plating bath. The entire setup is illustrated in Figure 3.

It can be readily observed from Figure 3 that the equipment was merely set up on the top of a standard laboratory bench. It required no "installation" as such. Most of the equipment employed was typical of small laboratory electroplating apparatus found in a testing or process control laboratory for general applications. The only specialized portion of the entire testing facility was the teflon (PVC or polypropylene would have been equally suitable) guides which held the panels in position during electrolysis. The construction of the guides was accomplished with the most basic of tools: hand drill, hacksaw, etc. The economies of the test apparatus employed should be obvious.

D. Electroplating Baths Studied

The data sheets of commercially available solder baths were examined for their suitability and advisability for this laboratory investigation.

The data sheets were examined for the recommended current density range, operating temperature range, chemical basis of the brightener system, and bath acid basis. Three were chosen on the basis of overall apparent suitability.

The vendors were then solicited to submit five-gallon samples of their baths for our evaluation. The intention was that the baths were to be formulated, mixed, and purified by the vendors prior to submission for our testing. The rationale for doing this was to allow the vendors to maximize their advantage by providing an optimum bath formulation.

The solder plating bath which is currently in use in the solder plating operation at the Richmond Works was used as the control bath in the comparative study. In the case of this bath, the sample for experimental plating was obtained by drawing an adequate amount from the shop production bath which was deemed to be functioning properly by the functional process engineer.

All baths, hereinafter, shall be referred to by their letter designation: A, B, C or D.

E. Experimental Electroplating and Rinsing Sequence

For each solder electroplating bath tested, a total of 24 test panels were plated. To start, four pairs of the three-inch wide unimaged panels were plated. The first pair and fourth pair plated received a high flow rate, approximately 80 to 85°, city water final rinse for two minutes. The second and third pairs of three-inch wide panels in this first segment of electroplating tests were rinsed in city water heated to approximately 150° Fahrenheit. [2]

The second segment of electroplating tests consisted of plating four circuit pack quarter-panels and four backplane quarter-panels. In order to offset any possible effects of bath aging as the test plating proceeded, plating of the circuit pack and backplane quarter-panels was alternated so that the circuit pack quarter-panels were plated in the first and consecutive odd numbered positions in the order of plating. During this second segment, the rinse water was maintained at approximately 90° Fahrenheit with a high flow rate.

The third segment of the plating test again involved four sets of three-inch wide unimaged panels. The third segment was essentially a repeat of the first segment. The rationale for running the three-inch wide unmasked panels in this fashion, bracketing the quarter-panel electroplating tests, was based on the fact that all surface characterization studies were to be run on the three-inch wide panels. Therefore, it was deemed necessary that these panels should be representative of the minimum and maximum aging of the test baths as the plating trials were performed.

The suppliers of all the baths claimed that the amount of proprietary additive contained in their respective baths would be adequate for the entire experimental sequence. Consequently, no brightener replenishments were made to the baths once the above discussed pattern of plating trials had commenced.

III. RESULTS

A. General Appearance

Upon completion of the electroplating tests and final rinsing, all of the three-inch wide strips possessed staining along the

edges and corners of the solder plated portion of said test strips when dry. This was attributed to the fact that they were all simply air dried in a plastic rack. The balance of the surfaces on the test strips displayed an even coloration and were not at all blotchy. Bath C samples presented the most desirable appearance, being light and even. The Bath B samples were still even but somewhat darker in coloration. The Bath A panels were lighter in appearance than the Bath B panels, but displayed some nodularity at the edges of these panels on those samples run near the end of the experiment. The Bath D panels were decidedly darker in general coloration than all the rest. However, they still were even in coloration and possessed no nodularity along the edges.

B. Latitude of Brightener Behavior

The observations discussed in this section concern the consistency of surface appearance resulting from an extended experimental run without brightener replenishment of the bath under test. The appearances of all samples from three of the baths were consistent throughout the entirety of their respective experimental runs. The exception was the Bath A which exhibited need for replenishment (the edge nodularity discussed above) during the last two runs of the total experiment.

C. Surface Character by Scanning Electron Microscopy (SEM)

This particular phase of the investigation yielded results which were relatively unambiguous in regard to interpretation for the characteristics under study. The baths are discussed in this section in the order of desirability of their surface character for the adhesion of an organic coating to the electrodeposited solder surface. The results are based exclusively on judgements made on the character of the topography as derived from experimental results on other projects which involved a study of an organic applied to a metallic surface. [3]

Within each group of scanning electron micrographs of the electrodeposit surfaces from the three-inch wide strips from a given electroplating bath, image analysis techniques were used to rank order all of the micrographs according to surface roughness. A simplified version for obtaining agglomerate and general particle size distribution by measurement of the intercept cord-length was employed. [4] These measurements were applied only where appropriate to the nature of the surface.

1. Bath D

Figure 4 shows the median micro-surface. The surface is composed mainly of ridges of sharper edged granules (as seen at the 5000X magnification) which form a harsh textured surface (as seen at the 1000X magnification). These sharper edged granules would be expected to form a good "tooth" to penetrate an organic coating, so as to maximize solder to organic coating contact while requiring a minimum amount of flow of said organic.

2. Bath B

Figure 5 shows the median micro-surface. Here the surface is composed of somewhat rounded granules which are 40 to 60 microinches in size (as seen at the 5000X magnification) with tendencies to become agglomerated along linear laterally oriented patterns which possibly correspond to the vendor generated brush marks in the copper substrate (as seen at the 1000X magnification). Such a surface would be expected to require a low amount of flow of an organic to maximize contact between the organic coating and the solder in order to achieve good adhesion. However, the rounder granules would limit the amount of penetration of solder into the organic coating.

3. Bath A

Figure 6 shows the median micro-surface as determined by image analysis. The surface is composed of granules, 20-40 microinches in size, which are intimately agglomerated to form generally rounded nodules (as seen at the 5000X magnification) which are, in turn, positioned in a random, but even close-packed array across the surface (as seen at the 1000X magnification). This type of surface morphology would require a greater flow of organic to maximize the organic coating and solder contact. However, the nature of the nodules provides ready sites for the organic coating to physically lock onto, hence, providing a surface whose organic coating adhesion properties would probably be comparable to the Bath B structure discussed above.

4. Bath C

Figure 7 shows the median micro-surface. This surface tends to exhibit a transition between nodular and rounded

granular surfaces which has the potential to bring out the detrimental characteristics of both. The larger irregular nodular agglomerates are generally rounded, being 60 to 160 microinches in size and composed of smaller intimately packed granules of such varying sizes to prevent a meaningful measurement (as seen at the 5000X magnification). These agglomerates, while generally positioned in a random close-packed array, are also seen to occasionally take on a somewhat linear alignment which may correspond to the vendor generated brush marks on the copper substrate (as seen at 1000X magnification).

D. Surface Analysis

Table I provides the results for the surface analysis performed on the solder plated surfaces of the three-inch wide copper strips.

The results achieved were totally unambiguous and the baths were readily rank ordered. This was first done qualitatively and then proportionally, based on the values of the data obtained. The nature of this portion of the investigation did not appear to require statistical evaluation to achieve a practical conclusion at the end of the study.

E. Organic Coating Adhesion Testing

The means of the results of the organic coating adhesion testing are also included in Table I. The results of the tests are expressed numerically on a scale of 1 to 5 (with a value of 5 indicating the highest adhesion). The results of the adhesion testing appear essentially to track one-to-one with the surface analysis ranking. However, it can just as easily be claimed that the organic coating adhesion testing results track one-to-one with the analysis of the results of the surface character regarding the desirability for adhesion as judged from the scanning electron microscope topography examinations.

Whatever the case, the more darkly colored deposits from Bath D produced the highest level of adhesion. The Baths A and B results were approximately comparable when all the values from which the means were calculated were examined. Bath C unquestionably produced the electrodeposit which was the poorest performer, having not only the lowest composite average adhesion values, but also generating the lowest single values for any individual tests (i.e., a value of 1).

F. Alloy Variation

When the tin/lead composition means and standard deviations for each of the two different types of quarter-panels (circuit pack and backplane) were calculated for the four baths under examination, the relative capabilities for consistency of performance of the individual baths were not readily evident. These values were then subjected to further examination by appropriate statistical analysis. Initially, a form of a box plot of the distribution of the data was made. This is illustrated in Figure 8. This type of plot shows the range of values for each of the two types of quarter-panels plated in each of the four baths under study. The asterisks within each of the thin bar plots is not indicative of the number of measurements made. The broken line connects the arithmetic means of the individual groups of data.

Upon initial examination of this plot, it would appear that Bath C and Bath D performed more consistently since the length of the bars covering their data points are obviously the shortest. One would also be tempted to award the Bath D a slight edge in performance. However, it was deemed most advisable to apply appropriate statistical analysis to the numerical values of the data to provide a means for a firmer judgment on the experimental results.

The object of good bath performance is consistency across the workpiece. In other words the less variation in the values of the attribute under observation (be it alloy composition, plating thicknesses, etc.) the better. Hence, a statistical test for differences in variance was applied. The specific method chosen was the Siegel-Tukey non-parametric test. [5] In this test, statistical significance was acknowledged when the confidence level exceeded 90 percent in rejecting a two-sided equivalency hypothesis.

The results indicated that for both backplane and circuit pack quarter-panels the variation of the Bath D was indeed less than the Bath A or Bath B. However, no statistical difference in variation could be demonstrated between the performance of Bath C and Bath D.

G. Surface Thickness Variation

The distribution plot of the data for each of the two types of quarter-panels plated in each of the four baths is illustrated in Figure 9. While a definite difference is seen in the data distribution between the backplane and circuit pack quarter-

panels, it is not really possible to make a judgment on the performance of the individual baths by simple observation. The Siegel-Tukey non-parametric test was applied for the same reasons as discussed in the above section. No statistical difference between baths was indicated for the experimental runs using the circuit pack quarter-panels. However, for the backplane quarter-panels, the variation in Bath D was again lower than either Bath A or Bath B.

H. Hole Thickness Variation

The distribution plot of the data for each of the two types of quarter-panels plated in each of the four baths is illustrated in Figure 10. There is a temptation to make a judgment in favor of Bath D based upon a simple observation of the distributions. However, the Siegel-Tukey non-parametric test still provided the best means for making a judgment on the results. Once again, no statistical differences in variation were obtained among the four baths for the circuit pack quarter-panels. However, the data for the backplane quarter-panels indeed indicated in favor of Bath D as having less variation than Baths A, B, or C.

I. Throwing Power

Figure 11 illustrates the composite deposit distribution plots of the data for both surface and hole thicknesses for each of the two types of panels plated in each of the four baths. On this plot the upper and lower broken lines connect the surface thickness and hole thickness means, respectively. The ratio of the value of the upper broken line to the value of the lower broken line would give an "average" throwing power. However, the variation for the data as well as the values of the means preclude any observational judgment on the graphically presented results. Moreover, because of the observed differences in variance, the means do not provide adequate information for a meaningful statistical evaluation.

IV. DISCUSSION

While the general appearance of the plated specimens varied in hue, all had approximately the same degree of evenness in surface appearance. However, this cannot be viewed as strongly indicative of relative shop performance since typically the evenness (or lack of blotchiness) upon drying is closely related to the drying system for any given production plater. Judgments made on the operational latitude of any of the baths, especially regarding the nodularity along the edges of the latter Bath A samples must also be viewed

with reservation since routine operating replenishment could offset the observed nodularity. This cautionary commentary appears justified since the SEM study of the plated surfaces generally indicated an acceptable latitude in operation for all of the baths. This can be said for two reasons. Firstly, the surface character observed for all experimental groups was reasonably constant from the first three-inch wide panel plated at the beginning of the experiment to the last three-inch wide panel plated at the end of the total experiment for any given bath. Moreover, those scanning electron micrographs illustrated in this report as the median samples were generally not associated with their position in the sequence of electroplating trials. This was also true of samples judged roughest and smoothest from each of the four bath groups.

Initially, the generally dark coloration observed on the samples plated in Bath D was suspected of being due to a lead rich electrodeposit. The data used to plot the Bath D distribution in Figure 8, however, shows that indeed this is not the case. In fact, the Bath D yielded the second highest average tin content of all the alloys examined. Thus, it would be logical to say that the surface configuration observed in the scanning electron microscope studies (Figure 4) is apparently the main contributor to the darker coloration. The relatively jagged surface topography would be expected to be the least reflective of all of the surfaces produced during the experimentation.

The statistical analyses for alloy and deposit thickness variations fortunately followed a pattern which was generally noncontradictory to the surface characterization results. In this particular case, it proved fortunate that the results indicated the same bath as the most likely candidate for continued production use or as an alternative for production trials (whichever the case may be for any given investigation). This is so since surface characteristics and minimal deposit characteristic variations are both important attributes to be considered in the choice of a solder plating bath. Had the results been otherwise; that is, different baths exhibited prime surface characteristics and minimal deposit characteristic variations, the decision methodology for suggesting a course of action would have become far more complex. It would then become the task of appropriate engineers to perform a typical weighted rank ordering of all of the attributes studied. In order to perform this properly, the engineers would first give "best judgement" weightings to each attribute. These weightings would then be subjected to an established scaling routine in order to give the weightings more meaningful relative values in the decision making process. Then each of the baths under study would be ranked ordered within each attribute category (according to the outcome of the experimental results and analysis of data). Finally, multiplying each rank for a

given bath in each category by the weighting of that category and summing the products of said multiplications would generate a figure of merit for that bath. The resultant figures of merit for the individual baths examined could then be used in making the decision regarding shop testing of an alternative or continuation of the current bath.

Prior to the initiation of this investigation an accepted formal methodology for comparative testing for evaluation of electroplating baths for printed wiring board applications did not truly exist. More often than not, electroplating engineers have had a tendency to resort to a short series of beaker-scale trials with a procedure derived for the needs at hand each time such needs arose. The beaker trials would then be followed by shop trials, ofttimes initiated with inadequate preliminary characterization of the bath "selected" for testing. Too often a program to test a group of plating baths smacked more of improvisation than investigation.

Beside performing a periodic reassessment of an electroplating bath vital to printed wiring board production, the basic methodology of testing, setup of equipment, and the scope of attributes examined, described herein, also constituted a simultaneous attempt to fill the chronic void described above. The authors submit that the design of this investigative technique has accomplished that end. It would appear from the nature of the results achieved, that such a submission is reasonable and proper.

The apparent lack of ambiguity of the results, while not a necessary outcome of even the most well-designed experimental procedure, are possibly attributable to the careful screening and choice of the baths tested. Technical judgment and prior experience which involved an assessment of the published technical vendor data played a major role in the selection of the baths to be tested. The balance of the screening was done according to the suitability of their advertised operating conditions to fit into the Richmond Works production shop.

While, all in all, it would appear that the methodology of this investigation forms a thorough and valid screening procedure with accompanying applicable statistical tests, it must still be emphasized that the ultimate value of such an undertaking to the overall engineering effort can only be proven out by the performance of the indicated "best choice" bath during actual shop operation. The scaling up of an electrolytic process from laboratory to production has always been one more of experienced engineering judgment as opposed to a mathematical transformation. It does, however, seem totally reasonable to expect that if a given bath from a group of baths performed the best when subjected to a carefully

followed experimental procedure performed in a properly designed test vessel, it would retain its relative performance in a larger system designed for overall balance.

V. CONCLUSIONS

A comprehensive comparative solder plating testing program has been developed which appears to be capable of providing enough information to select the most likely candidate(s) for production trials. By conducting a series of carefully coordinated experiments involving both small panel and selected product electrodeposition followed by examination of results using SEM topography, surface analyses, chemical analyses of the baths, organic coating adhesion testing, surface and hole plating thickness variation by cross-section and light-section microscopy, alloy variation by differential thermal analysis, and finally subjecting all appropriate data to statistical analysis by Siegel-Tukey non-parametric testing, the original objective was accomplished. From a carefully screened group of baths, one bath emerged as the most likely candidate.

VI. ACKNOWLEDGEMENTS

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TABLE I

Surface Analyses and Organic Coating Adhesion Tests

Bath Designation	Surface Analysis Rating		Average Organic Coating Adhesion Values
	Qualitative	Weighted	
A	3	3.13	4.0-
B	2	2.05	4.0
C	4	4.0	3.5-
D	1	1.0	5.0

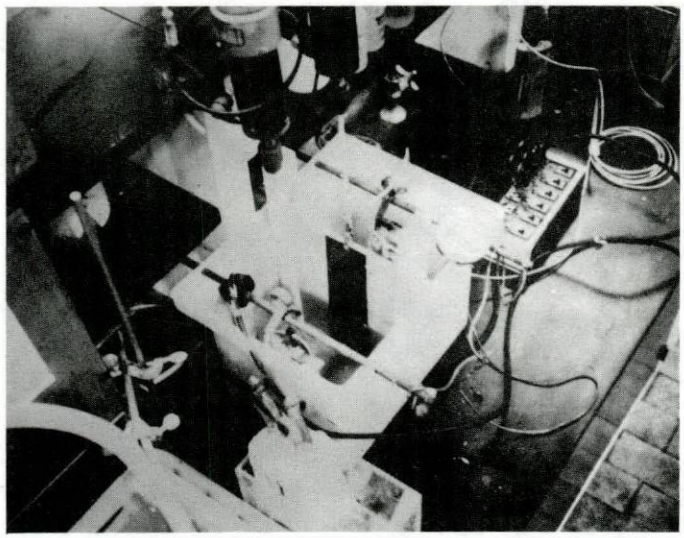

FIGURE 1

Electroplating Test Cell Set Up For 3-Inch Wide Panel

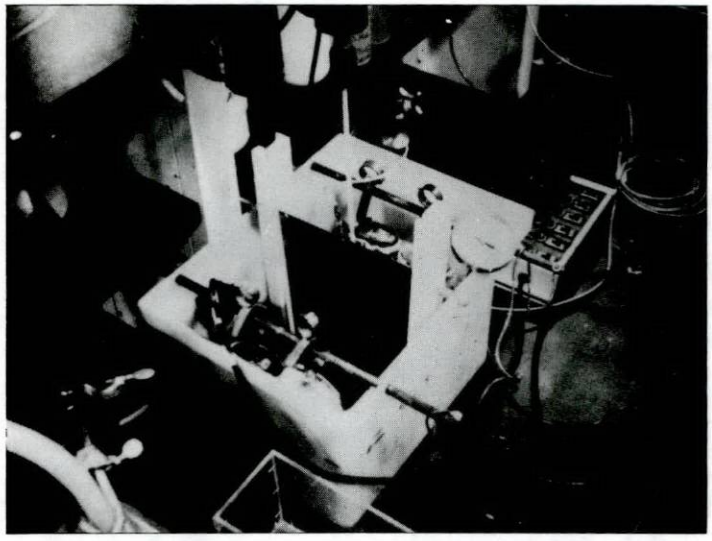

FIGURE 2

Electroplating Test Cell Set Up for Q-Panels

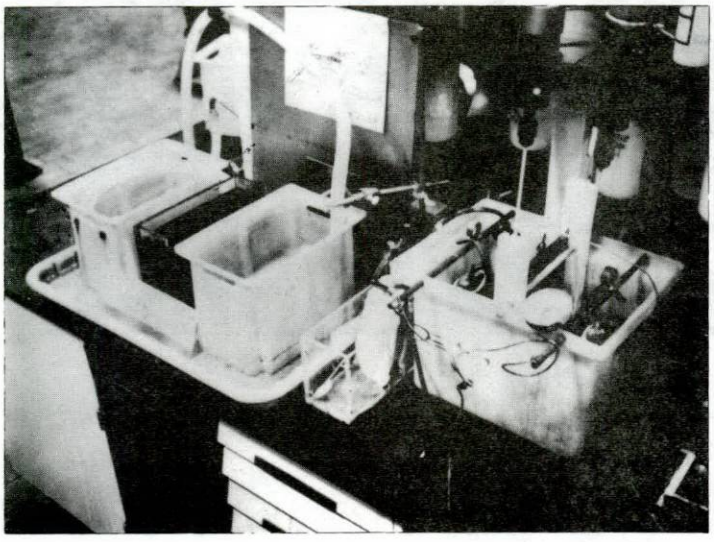

FIGURE 3

Entire Experimental Electroplating Apparatus
(Test Cell and Ancillaries)

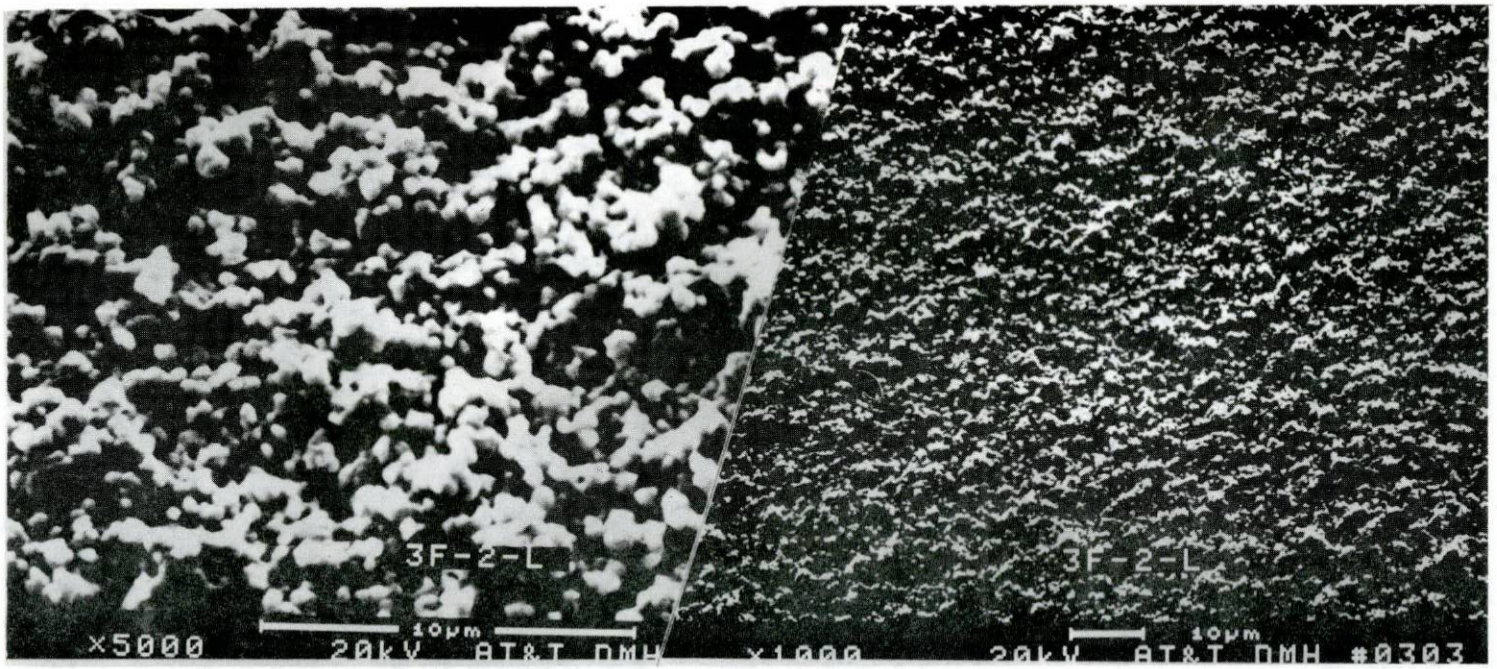

FIGURE 4

SEM Topograph of Bath D Electrodeposit Surface
Ranked Median (5000X/1000X)

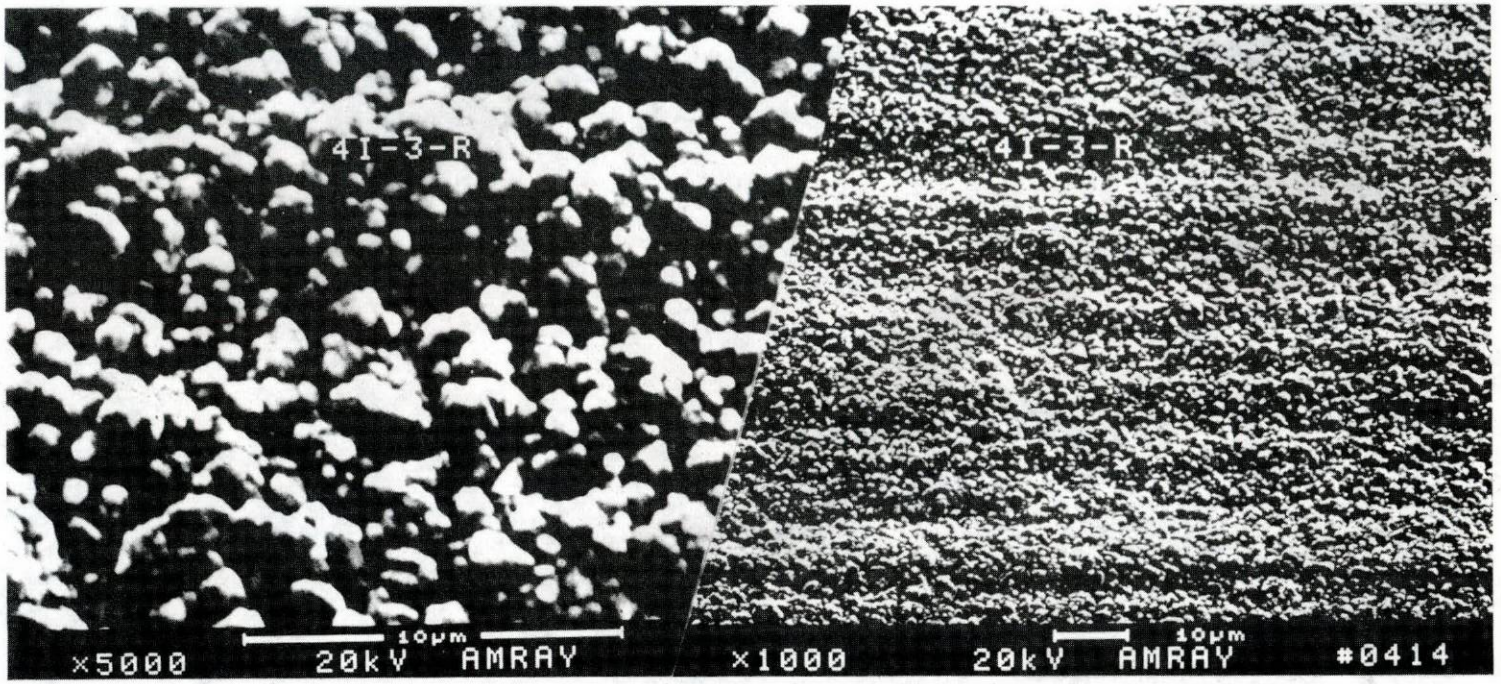

FIGURE 5

SEM Topograph of Bath B Electrodeposit Surface
Ranked Median (5000X/1000X)

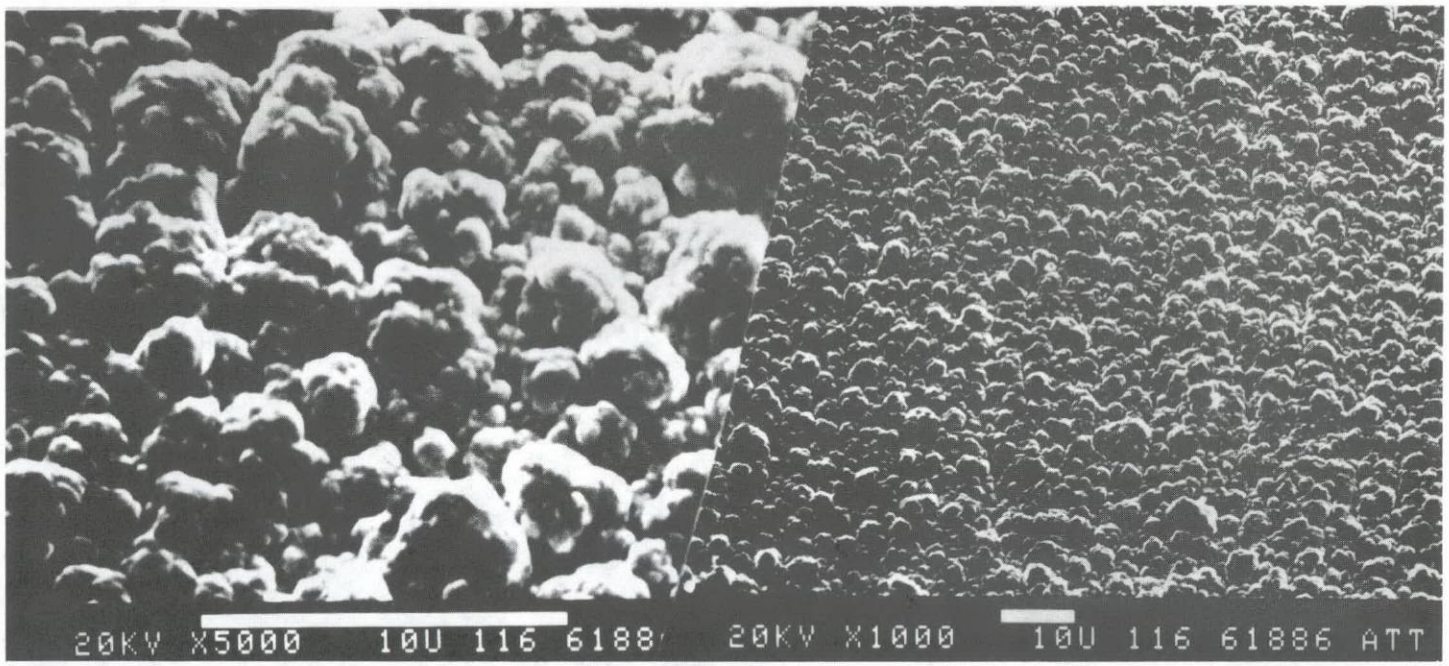

FIGURE 6

SEM Topograph of Bath A Electrodeposit Surface
Ranked Median (5000X/1000X)

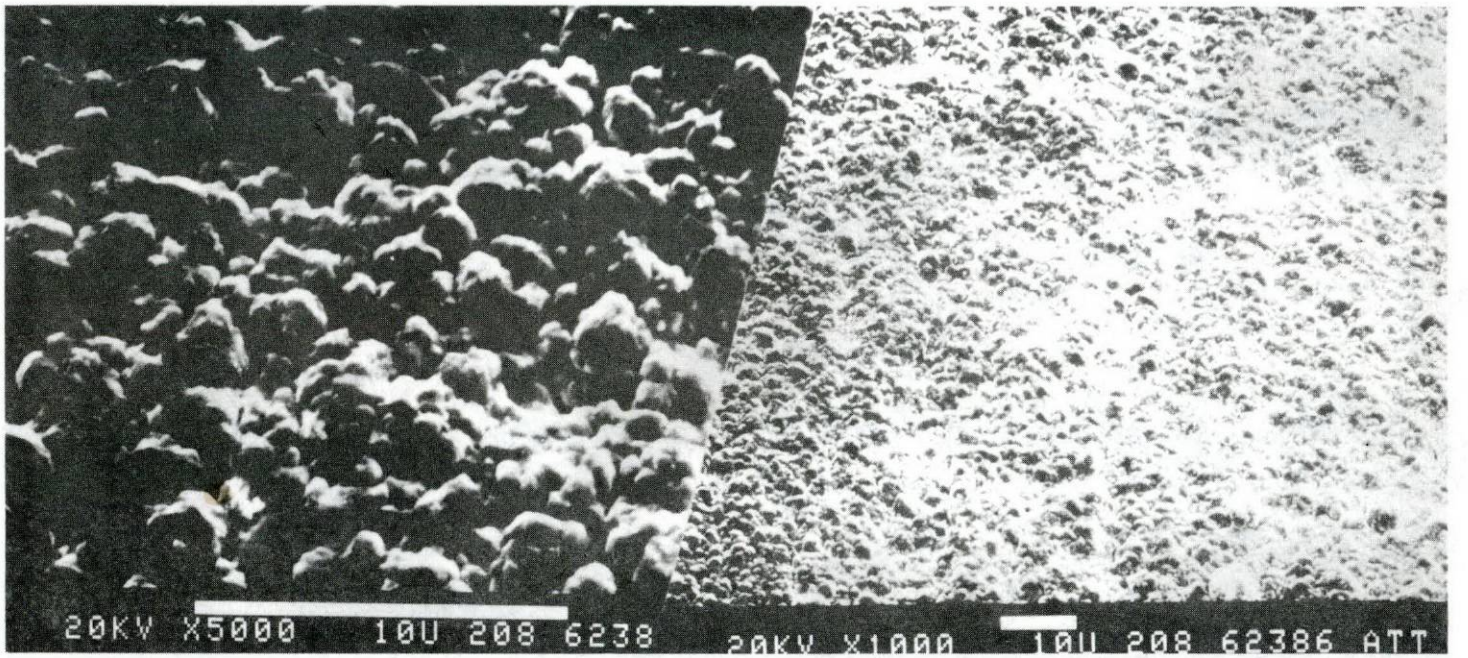

FIGURE 7

SEM Topograph of Bath C Electrodeposit Surface
Ranked Median (5000X/1000X)

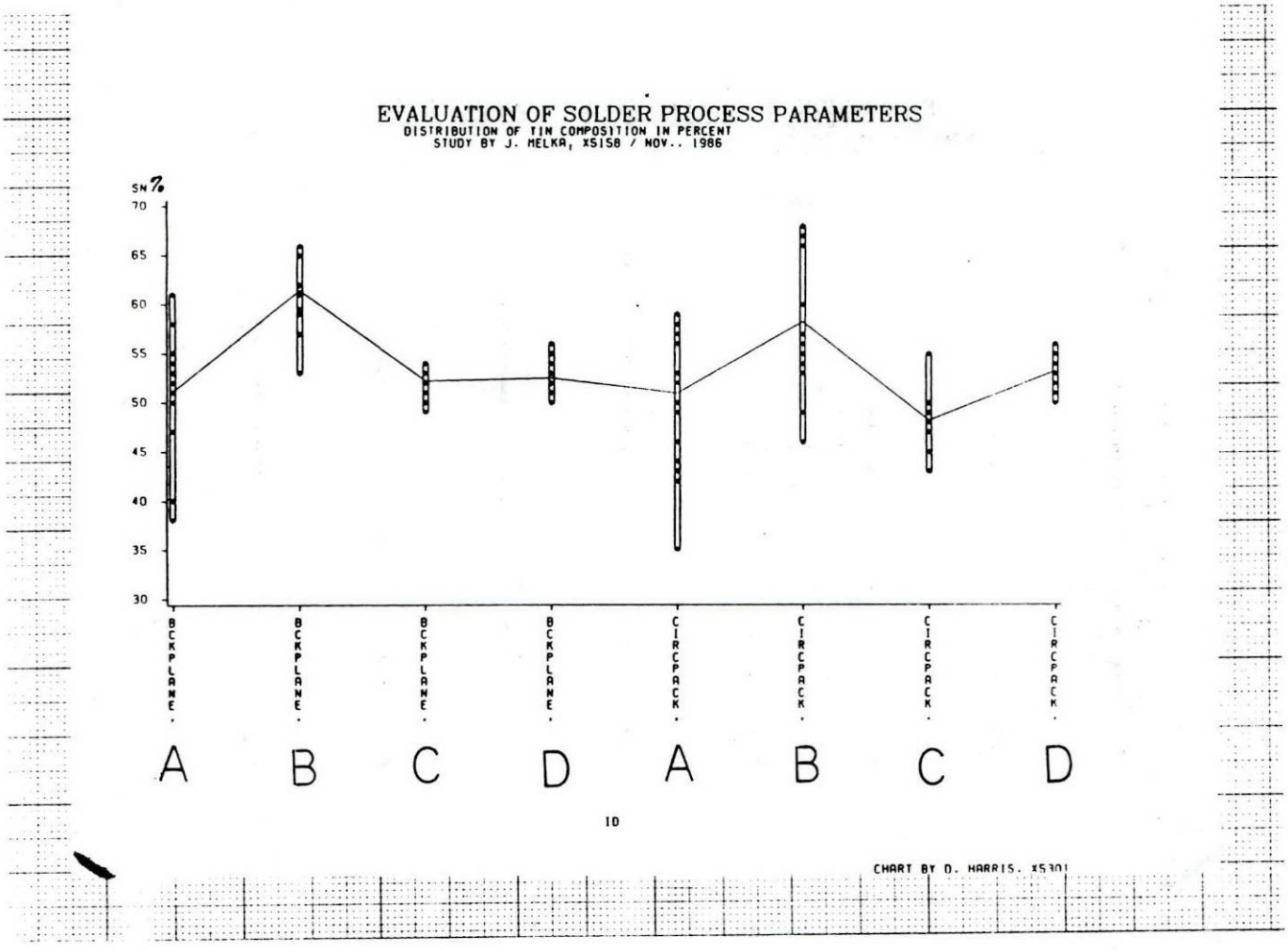

FIGURE 8

Distribution Plot of Tin Content for the Two Types of Test Q-Panels from the Four Baths

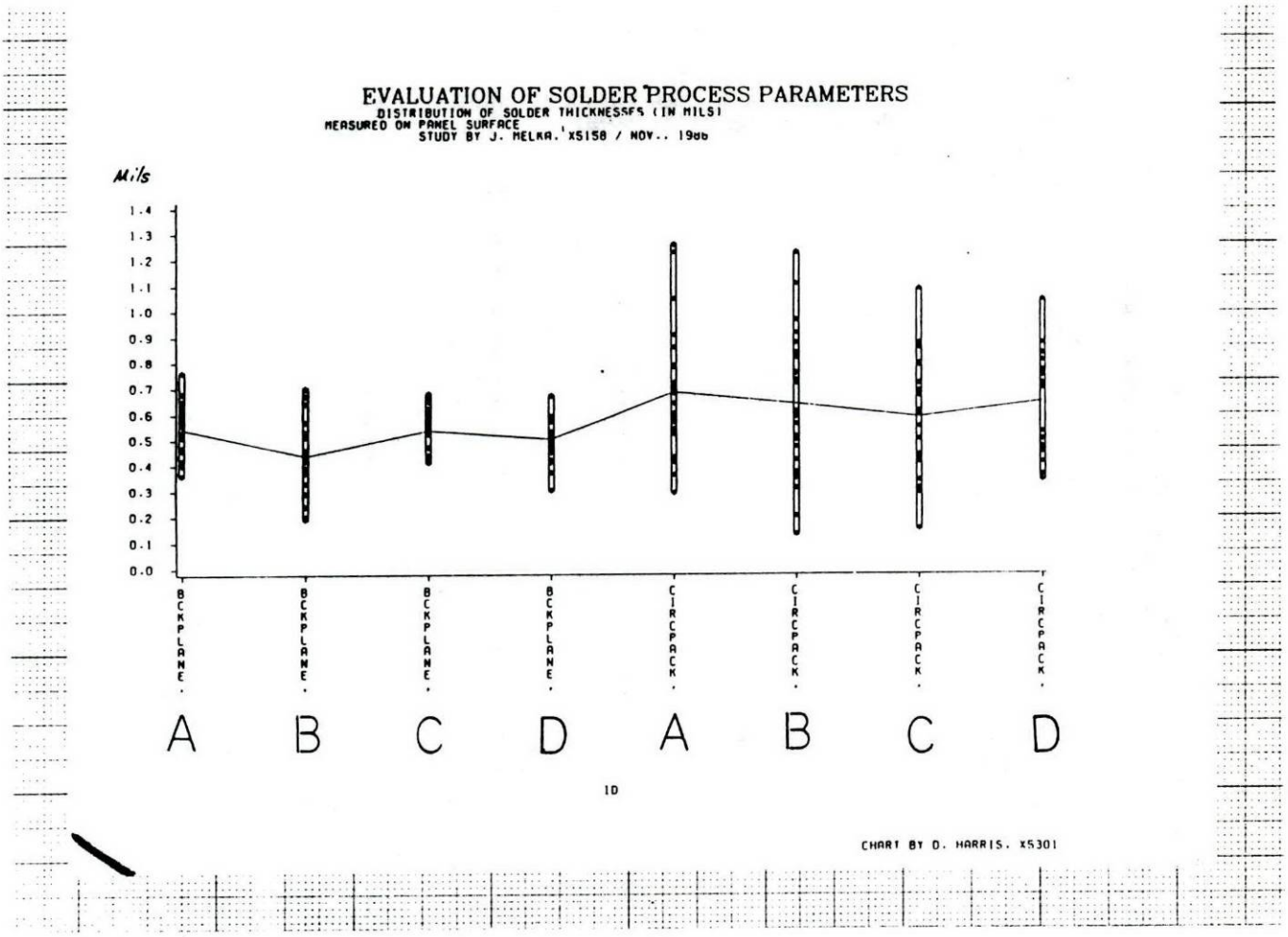

FIGURE 9

Distribution Plot of Surface Electrodeposit
Thickness for the Two Types of Test
Q-Panels from the Four Baths

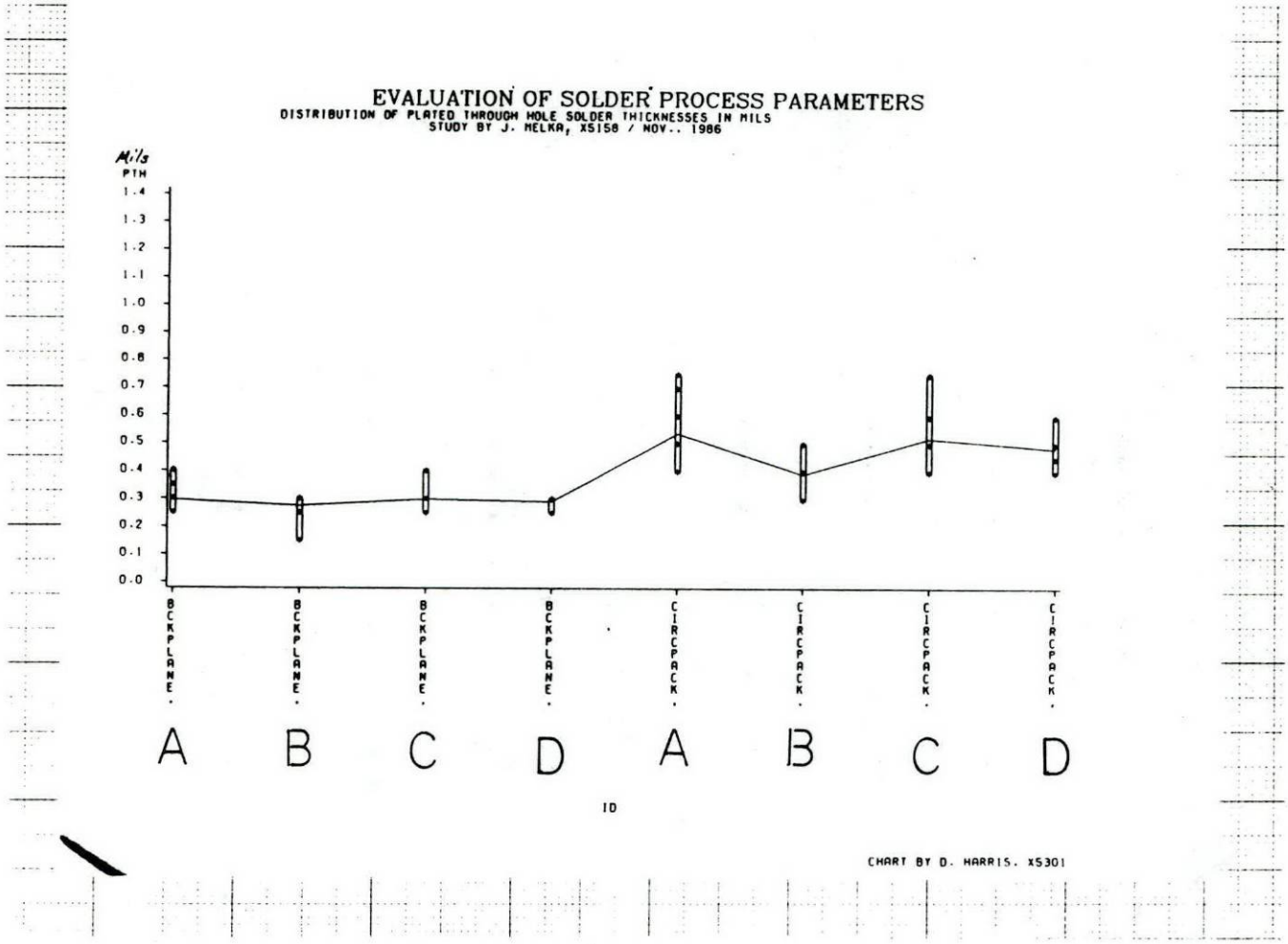

FIGURE 10

Distribution Plot of Hole Electrodeposit Thickness for the Two Types of Test Q-Panels from the Four Baths

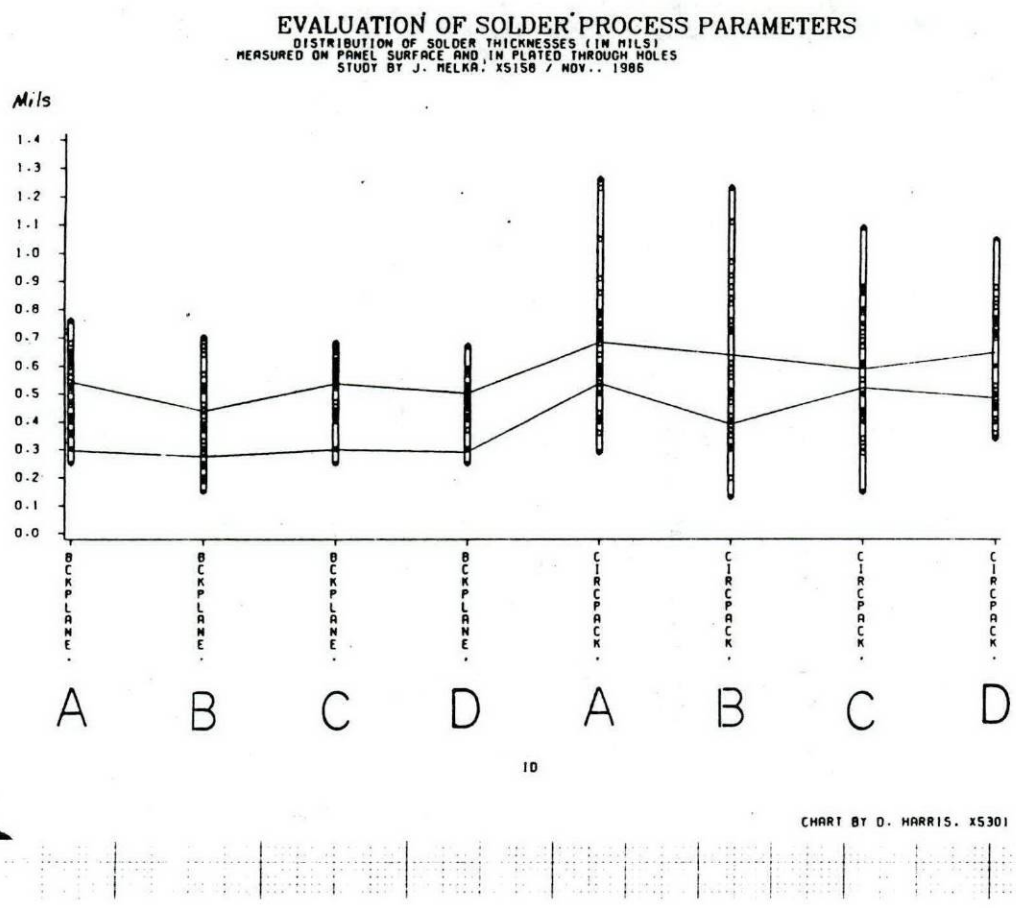

FIGURE 11

Combined Distribution Plot of All Electro-deposit Thickness for the Two Types of Test Q-Panels from the Four Baths

E-3

Electronics II
Session E

Tin and Tin-Lead Alloy Electroplating in the Electronics Industry

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TIN AND TIN-LEAD ALLOY ELECTROPLATING IN THE ELECTRONICS INDUSTRY

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Tin and tin-lead alloy electrodeposits are an integral part of a majority of electronic components and subassemblies such as connector blocks and printed circuit boards.

The fundamental properties of tin and tin-lead alloys, low melting point, low cost corrosion resistance and ductility, make them the natural choice of the design engineer to effect a permanent soldered interconnection during component assembly and to allow for ease of component replacement when necessary.

The increased demand for and complexity of electronic components make process selection a crucial step in establishing an operation which is both cost effective and capable of producing a tin or tin-lead alloy finish to meet the quality requirements of the final product.

Quality Requirements and End Use Specifications

In today's production environment the importance of quality control in predicting reliability of performance is a major factor in a company's ability to remain competitive.

Tin and tin-lead alloy electrodeposits are subject to some of the most stringent quality checks to substantiate reliability. These include solderability testing, both before and after accelerated aging, porosity and corrosion tests, and naturally, thickness and alloy checks. Many of the tests are the subject of both national and military specifications.[1-6] In the United States the most commonly applied specification is the Military Standard Mil-Std-202F.

Since the final assembly of a component may take place following prolonged periods of storage, accelerated aging tests designed to simulate storage in various climatic conditions have been studied extensively, in order to predict the useful life of tin and tin-lead alloy finishes.

As of April 1, 1987, Mil.-Std. 202F Method 208F has been updated in this regard, the duration of the steam aging test being increased to 4 - 8 hours from the 1 hour previously required. Ackroyd[7] reports that although resistance to 8 hours steam age correlates well with 6 months shelf life, a 24 hour steam age is required in order to distinguish the variation in solderability performance between deposit thicknesses in the range of 2 - 8 microns (80 - 320 microinches). Other accelerated aging tests used in the industry include dry heating at 155°C for 16 hours,[8,9] claimed to be equivalent to the effect of 12 months' normal storage.

Many of the findings of the accelerated aging studies are incorporated by individual manufacturers to take account of their customer's requirements and improve reliability of performance after storage.

The ultimate requirement for any tin or tin-lead finish is solderability either by fusing or by dipping in molten solder. The components are evaluated by the uniformity of coverage or "wetting" of the solidified solder coating. The temperature at which the molten solder test is conducted also varies from specification to specification. Currently the Mil-Std. 202F Method 208F requires a temperature of 245°C, recently increased from 232°C, whereas other specifications call for a temperature of up to 260°C.

In order to meet minimum specifications, the electrodeposits must have the following basic properties:

Uniformity of thickness and alloy composition

Fine grained crystal structure

Low or zero porosity.

Process Selection

The processes used to achieve these deposit characteristics are evaluated on the basis of:

Finish required

- matte or bright
- alloy composition
- deposit thickness

Ease of operation

- control requirements
- availability of analytical control methods
- consistency
- ease of handling
- ease of treatment and disposal of effluent

Economy of use

- cost of raw materials
- cost of maintenance and equipment
- yield versus cost

Productivity

- yield versus time

The vast majority of electrolytes currently used for the high volume tin and tin-lead alloy electroplating of electronic components are based on acid solutions of soluble stannous tin Sn II and lead Pb salts.

Acid solutions have become the choice of the electronics industry despite their inherent corrosive nature because of the high rates of deposition achievable from these systems. The high solubility of the divalent metal salts in these matrices allows the formulation of electrolytes to achieve the high current densities required for efficient production in modern high speed reel to reel electroplating equipment.

There are three electrolytes commonly in use:

1. **Sulfuric acid based**
2. **Fluoboric acid based**
3. **Organic sulfonic acid based.**

The specific formulation of the electrolyte in terms of metal ion and acid concentrations will vary, dependent on the process, the particular application and the production equipment. However, since the electrolyte type will impact all subsequent decisions related to process selection, thorough consideration should be given to the relative merits of the alternative electrolytes available before proceeding.

Sulfuric Acid Based Electrolytes

Due to the insolubility of lead sulfate, $PbSO_4$, the sulfuric acid based electrolytes are restricted to the electrodeposition of pure tin. However, because of the low initial cost of sulfate based chemicals, the system is still used despite its limitations.

Fluoboric Acid Based Electrolytes

Fluoborate based systems came into prominence due to their ability to electrodeposit both pure tin and a wide range of tin-lead alloys at extremely high current densities.

Both tin and lead are added as liquid concentrates of tin fluoborate $Sn(BF_4)_2$ and lead fluoborate $Pb(BF_4)_2$.

Organic Sulfonic Acid Based Electrolytes

Electrolytes based on organic sulfonic acids are relative newcomers to the electroplating industry. Introduced commercially in the early 1980's, the systems were well received due to their inherent advantages with respect to ease and flexibility of operation and have successfully established themselves as the preferred process for the high speed reel to reel tin and tin-lead alloy electroplating of electronic components.

The tin and lead are added as liquid concentrates of tin organic sulfonate and lead organic sulfonate.

Since all the sytems are acidic and contain stannous tin Sn II salts, there are inherent and common problems associated with their use. These are the corrosive nature of the electrolytes and the tendency for the divalent stannous tin to oxidize to tetravalent stannic tin Sn IV.

Corrosive Nature of the Electrolytes

By far, the most corrosive electrolyte is the fluoborate based system. The corrosive nature of fluoboric acid itself is compounded by the formation of the fluoride ion during electrolysis which mandates the use of the most acid resistant materials of construction to avoid premature degradation of plant and equipment. It also precludes the use of silica based materials such as silica sheathed heaters, glass electrodes and silica based filter aids.

Another unique negative feature of the fluoborate system in this regard is the tendency for the electrolyte to "creep" over hardware, leaving highly corrosive films on surfaces, even in areas remote from the electroplating equipment.

The sulfuric acid and organic sulfonic acid based electrolytes, although corrosive by definition, are much less severe on plant and equipment and are generally easier to handle than the fluoborates.

The organic sulfonate electrolytes are the least corrosive of the electrolytes under comparison which not only allows a wide choice in hardware but also minimizes attack on plant, decreasing both maintenance and capital equipment costs.

Oxidation of Divalent Tin

The oxidation of divalent stannous tin to tetravalent stannic tin caused by atmospheric and anodic oxidation leads to various problems in process control. The loss of stannous tin, if uncorrected, will ultimately affect both the high current density range and cathode efficiency of the process, the net effect of which is a loss in depositon rate and productivity.

In tin-lead alloy electrolytes, the loss of stannous tin will decrease the ratio of divalent tin to lead in solution and hence alter the electrodeposited alloy composition. In practice, the stannous tin can be replenished according to analysis before the above problems compromise process control and product quality. However, the frequency with which the analysis must be performed and the subsequent amounts of stannous tin replenished have a direct effect on the overall cost of the process and final product.

A more visual effect of the oxidation is the build-up of sludge produced from the precipitation of stannic tin compounds. The precipitates can cause rough deposits which compromise product quality, producing rough deposits, and ultimately the accumulated sludge must be removed, a costly and time consuming procedure. The sulfate electrolyte is the most prone to sludge build-up.

Although most proprietary processes contain anti-oxidants to minimize the oxidation of the divalent tin, only the organic sulfonates have an inherent ability in this regard. In fact, the organic sulfonic acid based processes are sufficiently resistant to oxidation to allow the use of insoluble anodes, a classic source of highly oxidative nascent oxygen.

Based on a comparison of the corrosive nature and stability of stannous tin alone, the organic sulfonic acid based electrolytes show clear advantages over either the sulfuric or fluoboric acid based systems.

The organic sulfonates' resistance to oxidation is particularly beneficial in high speed operations where the necessary use of vigorous solution agitation in combination with high current densities of operation produce highly oxidative conditions.

The systems are the least prone to sludge build-up and generally give longer service than either the fluoborate or sulfate systems.

The properties of the organic sulfonic acid based electrolyte allow flexibility in material selection for equipment design which improves process control and product quality. The use of anode pieces in titanium baskets makes for ease of anode maintenance and therefore helps ensure a consistent anode area and cell efficiency. The use of insoluble anodes allows the use of specific cell designs with very short anode to cathode distances where the ultimate in speed and profile are required.

Finally, the effluent from the organic sulfonate system can be rendered innocuous by simple neutralization and filtration procedures.

Proprietary Processes and Additive Packages

Regardless of the type of electrolyte selected, some form of organic additive is usually required to produce electrodeposits with the necessary properties to meet the quality requirements of the final product. It is at this stage that specialty suppliers of proprietary chemicals are normally consulted and the choice of process package made. The decision making process should take into account not only the initial requirements but also the quality and continuity of ongoing support available from the supplier.

Different additive packages are used, dependent on the cosmetic finish required, matte, semibright or full-bright. However, common to all applications and particularly important for high speed electroplating conditions is the need for a primary grain refiner and an anti-oxidant. The anti-oxidant, as previously discussed, is designed to inhibit the oxidation of divalent tin.

The primary grain refiner inhibits the tendency for dendritic or outward deposit growth, which produces rough, powdery and poorly adherent deposits of little practical value. This effect is often referred to as "treeing" when observed at high current densities.

Although the detailed mechanism of grain refinement has never been fully elucidated, surface adsorption of the organic additives certainly plays a role in the modification of the electrogrowth process.

In systems designed to produce full-bright deposits, secondary organic additives which further modify the growth process to produce finishes with specular reflectance are incorporated.

The effect of proprietary additives, often wrongly classified simply as brighteners, on the solderability of tin and tin-lead alloy electrodeposits has been and continues to be the subject of considerable debate and discussion. An area of primary focus is the amount of organic matter from the additives and their breakdown products occluded in the electrodeposit. As a general rule, it is believed that the higher the occluded organic content of the deposit, the more solderability and fusing problems will be encountered. This has led to a greater emphasis being placed on the control of organic

additive in the process in order to maximize their beneficial effects without compromising the solderability properties of the deposit. Analytical methods such as Ultra Violet Spectroscopy and High Pressure Liquid Chromatography, coupled with various detection techniques for the separated organic species, are being utilized to effect this improvement in process control. Cyclic voltammetry is also being applied to process control. This technique is based on electrokinetic measurements and has the advantage of detecting materials having a dynamic effect on the electrodeposition process. This makes it a powerful tool, not only for process control but also in additive development.

Research Work

A great deal of previous work has been reported on factors which influence solderability.[7, 10-13]

The research work presented in this paper was initiated to compare the influence on solderability, both before and after accelerated aging of different proprietary additives designed to produce bright electrodeposits from an organic sulfonic acid based system.

Included in the study was the influence of underplate type and finish, as well as thickness of the tin-lead electrodeposits.

One premise of the comparison was that the generalization of higher occluded organics being detrimental to solderability may not always apply, since it does not take account of either the type of organic material occluded or the nature of its dispersion through the electrodeposit.

Experimental

Substrates and Underplates

Previous solderability experiments conducted using polished brass Hull cell panels cut into strips as test coupons had been shown to be unsuitable for an evaluation of solderability of tin-lead alloy electrodeposits. Variations in tin-lead alloy thickness between the center and edge of the panels, coupled with the observation that dewetting tended to be associated with the sheared edge of the panels ruled out this configuration as nonreproducible.

It could, of course, be argued that these are the real variables experienced in stamped and formed components. However, it was felt that a standard reproducible configuration was a prerequisite in order to ensure that the solderability performance of the tin-lead finishes were compared rather than component effects.

It was for this reason that 1/4 inch diameter copper rods of the type shown in Fig. 1 were chosen as the standard test coupons. This configuration has three distinct advantages:

1. There are no edge effects.
2. The test coupons can be electroplated in a rotating cathode fixture[14] Fig. 2, which allows the researcher to duplicate the flow characteristics of a high speed electroplating cell and ensures a uniform thickness profile over the entire surface.
3. The experimental technique is easily reproducible.

The 1 x 3 inch flat brass coupons were, however, used for the electrographic porosity testing of the tin-lead finishes.

In many cases, an initial nickel coating of between 1.25 and 5 microns (50 - 200 microinches) is electrodeposited to improve long term corrosion resistance and prevent copper diffusion into the functional final finishes. For tin and tin-lead coatings, copper diffusion is a particular problem since the copper-tin intermetallics are brittle compounds and can lead to failure of a solder joint through fracture. The intermetallics are also more difficult to wet with solder and are therefore undesirable in the electroplated components.

The introduction of an underplate introduces a new variable for solderability testing. Electrodeposited nickel can be produced in a range of cosmetic appearances from dull/matte to full-bright, and again organic additives are used in many of the processes.

In this work, three undercoats were studied, dull nickel, bright nickel and bright acid copper. The electrolytes used and thicknesses of deposits produced are listed in Table 1.

Table 1

Electrolyte Formulations for Each Underplate Finish Studied

Dull Nickel		Bright Nickel		Bright Acid Copper	
Nickel Sulfamate	320 g/l	Nickel Sulfate	270 g/l	Copper Sulfate	75 g/l
Nickel Chloride	40 g/l	Nickel Chloride	20 g/l	Copper Metal	20 g/l
Nickel Metal	85 g/l	Nickel Metal	80 g/l		
Boric Acid	45 g/l	Boric Acid	50 g/l	Sulfuric Acid	100 ml/l
Additives	None	Additives	Primary & Secondary	Additives	Yes
Temperature	57°C	Temperature	57°C	Temperature	24°C
Thickness	4.0 µm	Thickness	4.0 µm	Thickness	12.5 µm

Solderability

90% tin 10% lead electrodeposits were chosen for this study due to the prevelant use of this alloy composition in the connector industry. Full bright finishes, Fig. 1, were used in order to exaggerate the effects of occluded organics. However, as previously discussed, the type of organic may be as important as the quantity occluded. Therefore, in the present study, electrodeposits produced using primary grain refiners of different chemical types were compared.

One set of matte, 6 µm thick deposits, Fig. 1, produced using the primary grain refiners only, was included for control and comparison. Only bright nickel substrates were used for the matte deposits.

The electrolytes used were of the organic sulfonic acid type. The specific formulations are given in Table 2.

Table 2

Tin-Lead Electrolytes

Electrodeposit Type	A	B	C	D
Tin Concentration	40 g/l	40 g/l	40 g/l	40 g/l
Lead Concentration	5 g/l	5 g/l	5 g/l	5 g/l
Organic Sulfonic Acid	50 ml/l	50 ml/l	50 ml/l	50 ml/l
Primary Grain Refiner	"A"	"B"	"A"	"B"
Secondary Grain Refiner	40 ml/l	40 ml/l		

In order to evaluate the influence of tin-lead deposit thickness, samples were plated at thicknesses of 3.0, 6.0 and 12.0 microns.

Electroplating Cycle

The following cycle was used for all samples:

Cathodic Alkaline Electroclean
Double Rinse
10% Sulfuric Acid
Double Rinse
Underplate
Double Rinse
20% Organic Sulfonic Acid
Tin-Lead Finish
Double Rinse
Deionized Water Rinse
Hot Air Dry

Rods to be tested for solderability were electroplated at a current density of 100 ASF using an AFASR rotator[14], Fig. 2, at a rotation speed of 2000 rpm.

The 1 x 3 inch brass coupons used for porosity testing were electroplated at a current density of 100 ASF using stir bar agitation and reciprocal movement of the part.

Testing of Deposits

The deposits were evaluated as follows:

1. Thickness and alloy check
2. Percent Occluded Organic
3. Percent Occluded Carbon
4. Porosity
5. Solderability

Test Procedures

Thickness and alloy composition of all tin-lead electrodeposits were determined by X-ray fluorescence.

The percent occluded organic was determined by measuring the weight loss after fusing of electrodeposited tin-lead foils.[11]

The carbon content of the electrodeposited foils was measured by a pyrolitic technique[15], the carbonaceous materials being converted to and measured as carbon dioxide.

Electrographic porosity testing[16] was performed on a selection of samples, which exhibited poor solderability after steam aging, in an attempt to correlate poor performance with the degree of porosity. 1 x 3 inch flat brass test coupons with 12.5 μm of a bright acid copper undercoat were used for the test.

Conditions of Test

Electrolyte:	100 g/l Sodium Bicarbonate Solution
Detection Method:	Cadmium Sulfide Papers
Current:	650 mA
Time:	60 sec

Solderability testing was carried out in accordance with Mil-Std 202F Method 208F both before and after the following accelerated aging tests:

1. Eight hours exposure to steam according to Mil-Std 202F.
2. Sixteen hours exposure to dry heat at 155°C.

All systems were prepared and tested in triplicate to improve statistical confidence.

Solderability performance was evaluated by examination of each soldered sample under 10X magnification. Defects such as dewetting, nonwetting and pinholing were assigned an index value corresponding to the degree of defect:

	Index Value
Less than 1% of surface area exhibited defects.	0
1 - 3 % of surface area exhibited defects.	1
4 - 6% of surface area exhibited defects.	2
Greater than 6% of surface area exhibited defects.	3

Examples of the soldered rods showing good solderability and typical defects are shown in Fig. 3.

Results

Codeposited Organic Matter

Electrodeposit Type	Occluded Organic Content	Occluded Carbon Content
A	0.41%	0.156%
B	0.42%	0.280%

Porosity

No porosity was detected in any of the samples tested.

Solderability

The solderability performance of the two full-bright tin-lead electrodeposits A and B are presented in Tables 3 and 4, respectively. The tables are compiled using the measured index values and compare the influence of tin-lead thickness, accelerated aging and underplate.

The individual effects of tin-lead thickness, aging and underplate on solderability performance are presented graphically in Figs. 4, 5 and 6.

The results of the matte electrodeposits C and D are presented in Fig. 7.

Fig. 8 compares the effects of aging on the solderability performance of electrodeposits A, B, C and D.

Table 3

Electrodeposit A

	As Plated			Steam Aged			Heat Aged		
Underplate	3 μm	6 μm	12 μm	3 μm	6 μm	12 μm	3 μm	6 μm	12 μm
Dull Nickel	0	1	1	0	2	1	1	1	0
Bright Nickel	0	0	1	3	2	2	2	3	1
Bright Acid Copper	0	0	1	2	2	2	2	3	1

Table 4

Electrodeposit B

	As Plated			Steam Aged			Heat Aged		
Underplate	3 μm	6 μm	12 μm	3 μm	6 μm	12 μm	3 μm	6 μm	12 μm
Dull Nickel	0	0	0	1	1	0	1	1	0
Bright Nickel	0	0	0	0	1	1	1	0	1
Bright Acid Copper	0	0	0	1	1	0	0	0	1

Conclusions

1. The total occluded carbon of the Type B electrodeposit is higher than that of Type A. This difference is not apparent from the organic content of the deposits.

2. The influence of the underplates is most evident with the Type A electrodeposit, (Fig. 6). The beneficial effect of a dull nickel underplate is highlighted by these results.

3. Thickness of the tin-lead electrodeposits, in the range 3 - 12 μm tested, has little influence on the solderability performance, (Fig. 4). This is supported by the porosity test results.

4. Although the use of primary grain refiner "B" resulted in tin-lead electrodeposits with higher occluded carbon content, their solderability performance is superior overall to tin-lead electrodeposits produced with primary grain refiner "A".

   This result is confirmed by the solderability performance of the matte tin-lead electrodeposits, Type C and D, (Fig. 7).

   The difference is particularly evident following accelerated aging of the electrodeposits, (Figs. 5 and 8).

Summary

A review of the major problems encountered by the process engineer and materials scientist in meeting production demands and end use specifications for electronic components with tin-lead finishes has been presented.

The importance of the composite finish and electroplating processes used, to achieve these requirements has been demonstrated.

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Figure 1

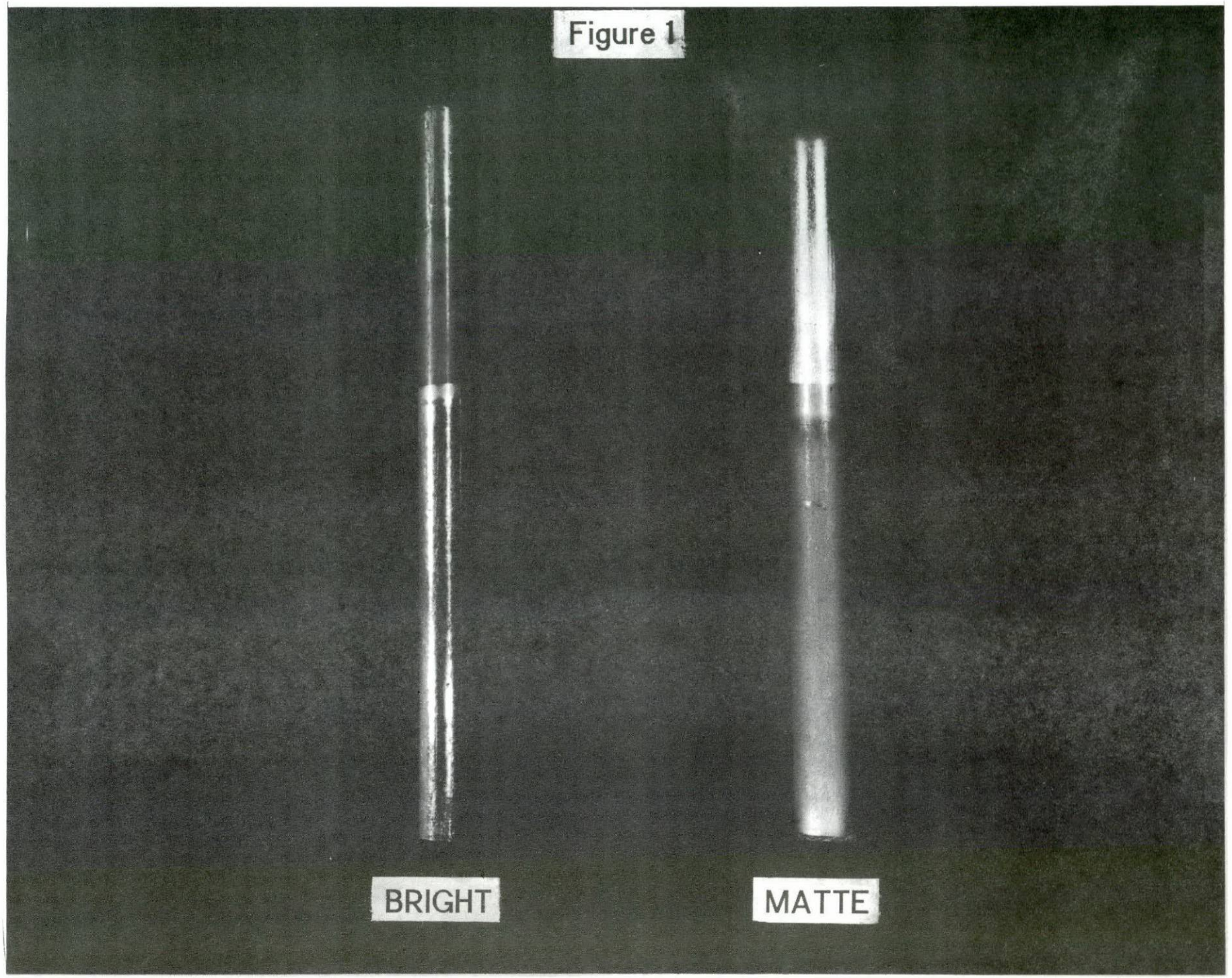
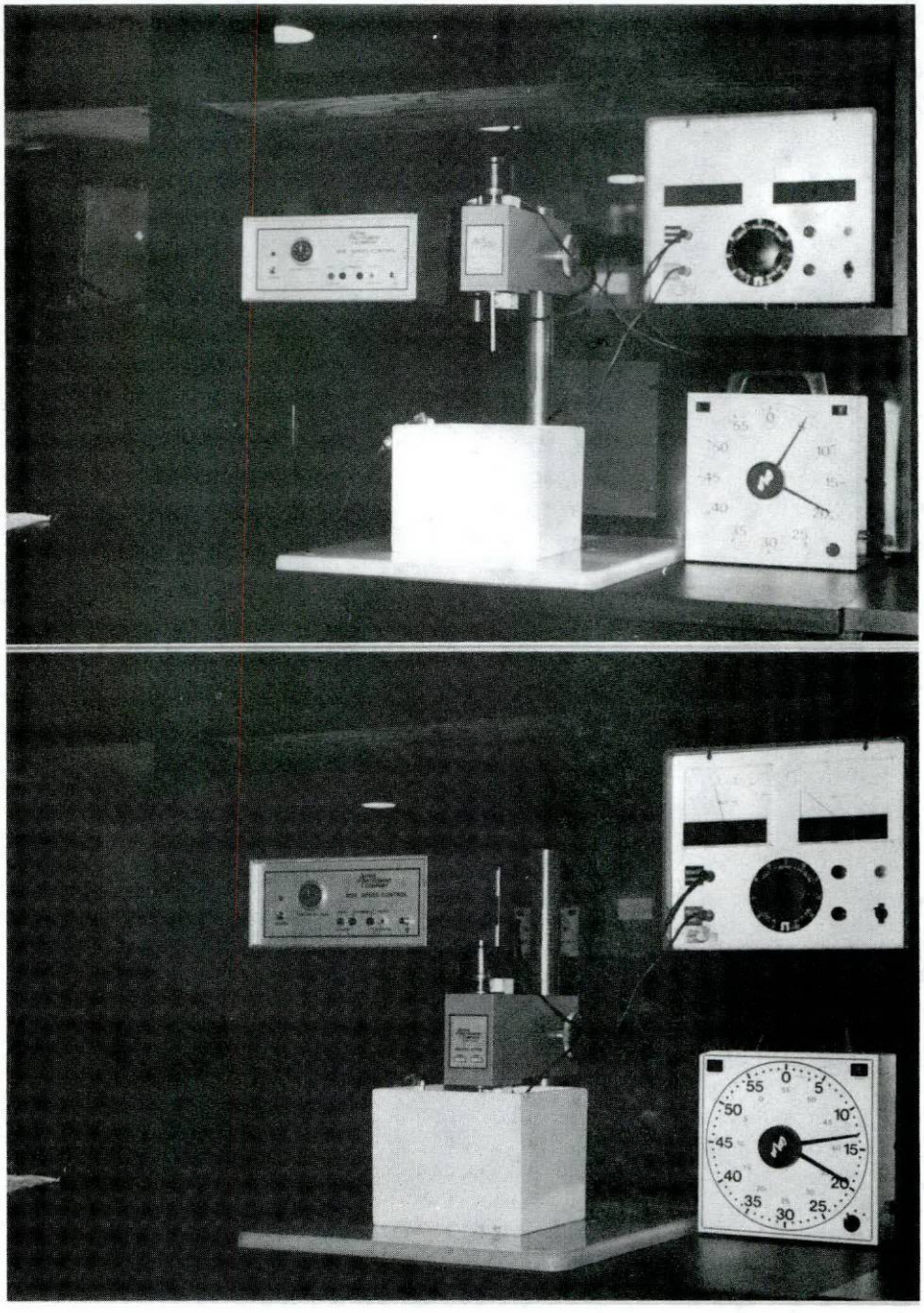


FIGURE 2



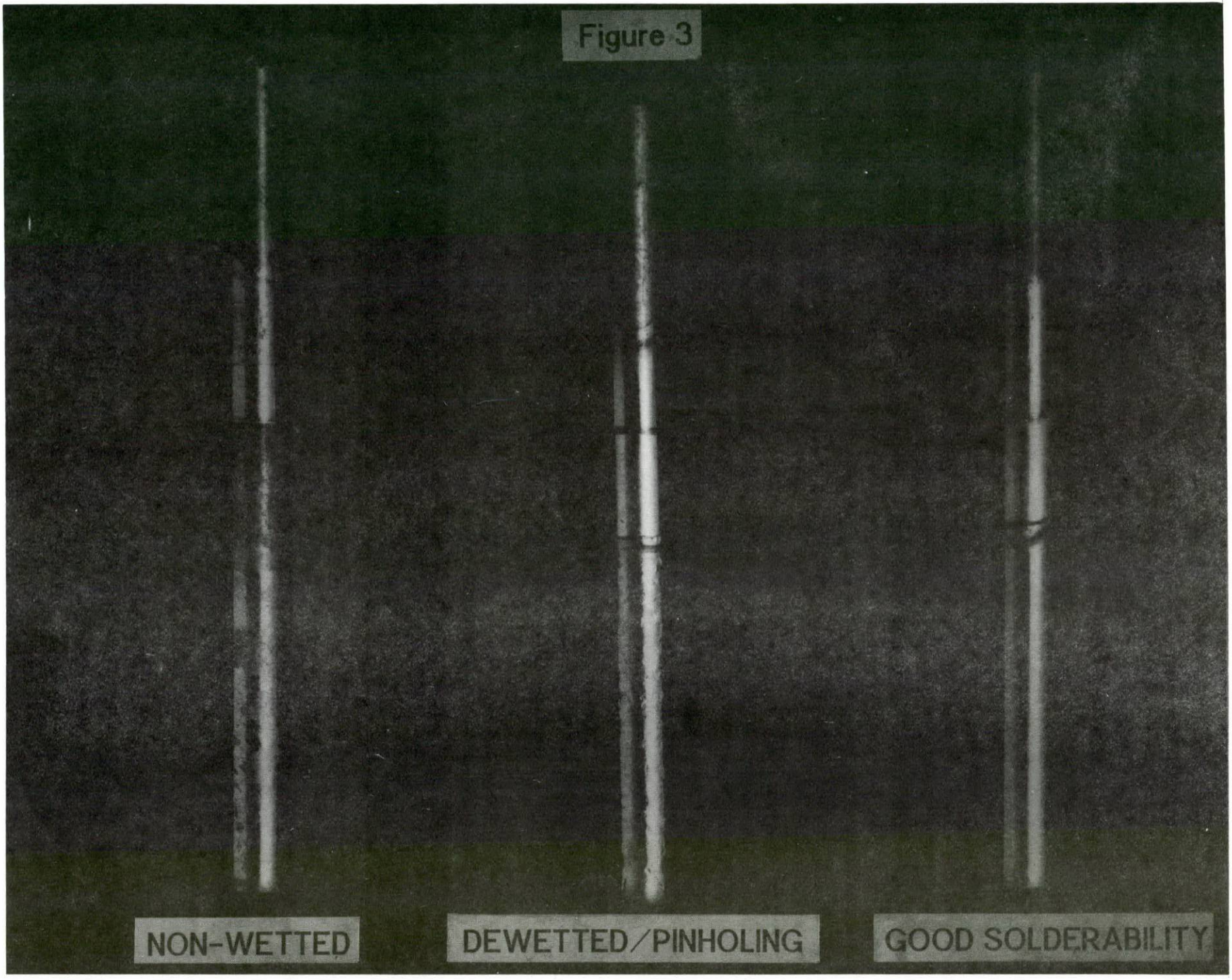


Figure 3
NON-WETTED
DEWETTED/PINHOLING
GOOD SOLDERABILITY

FIGURE 4

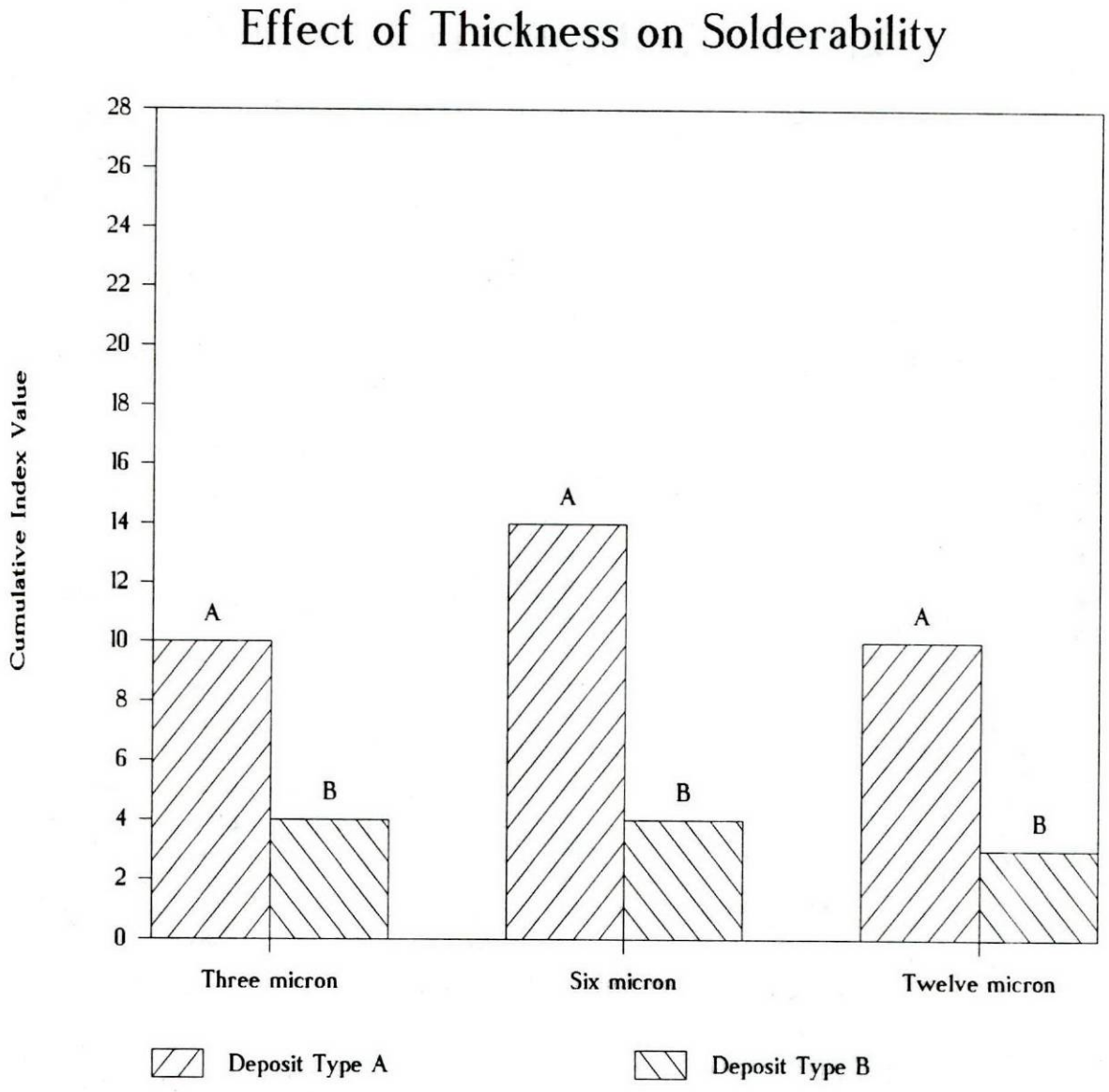

Effect of Thickness on Solderability
Cumulative Index Value
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
A
B
A
B
A
B
Three micron
Six micron
Twelve micron
Deposit Type A
Deposit Type B

FIGURE 6

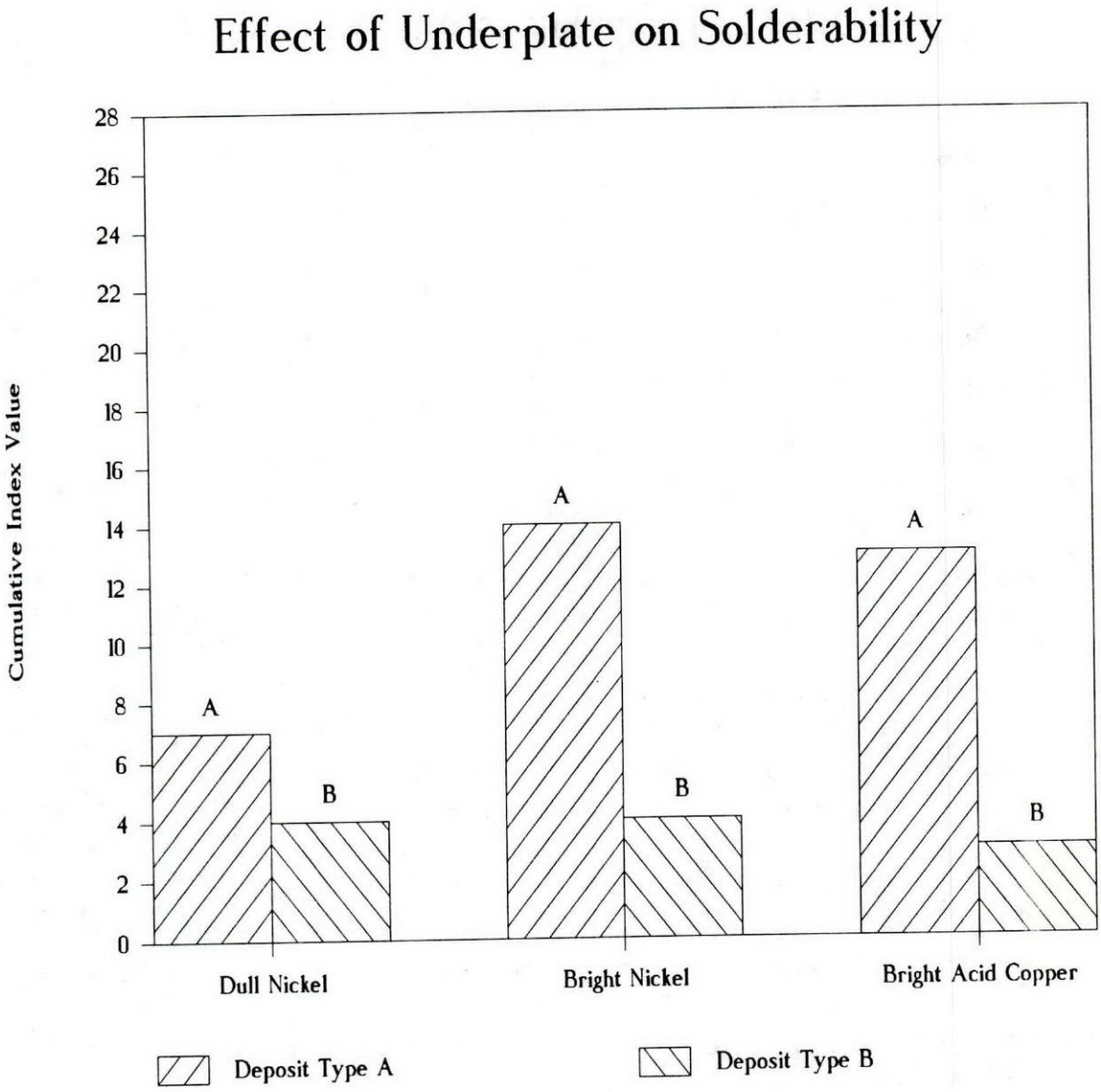

Effect of Underplate on Solderability
Cumulative Index Value
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
A
B
A
B
A
B
Dull Nickel
Bright Nickel
Bright Acid Copper
Deposit Type A
Deposit Type B

FIGURE 7

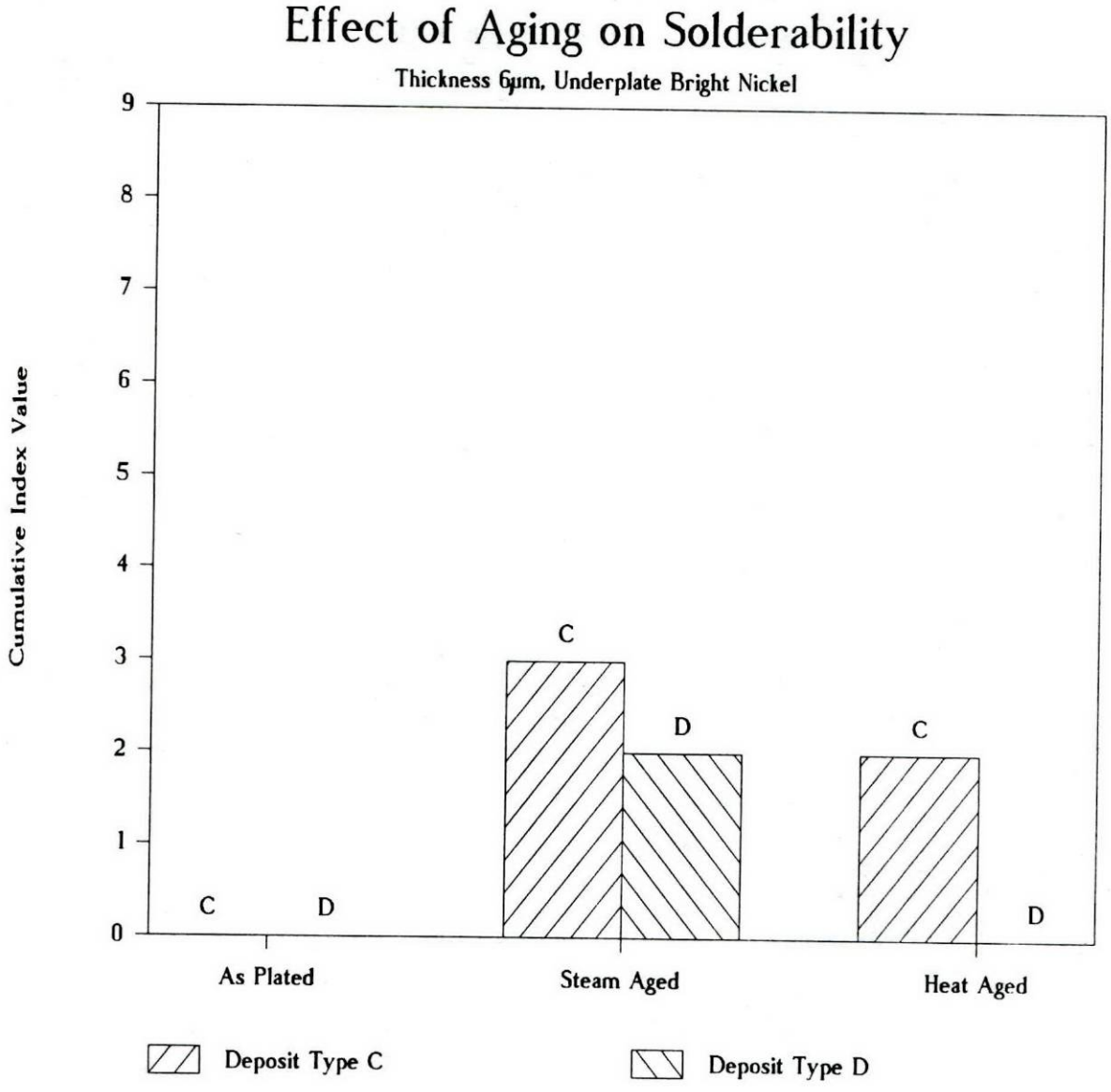

Effect of Aging on Solderability
Thickness 6µm, Underplate Bright Nickel
Cumulative Index Value
9
8
7
6
5
4
3
2
1
0
C
D
C
D
C
D
As Plated
Steam Aged
Heat Aged
Deposit Type C
Deposit Type D

FIGURE 8


Effect of Aging on Solderability
Thickness 6µm, Underplate Bright Nickel
Cumulative Index Value
9
8
7
6
5
4
3
2
1
0
A
B
C
D
A
B
C
D
A
B
C
D
As Plated
Steam Aged
Heat Aged
Type A
Type B
Type C
Type D

E-4

Electronics II
Session E

A New Pure Palladium Electrodeposit for Electronic Applications

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A NEW PURE PALLADIUM ELECTRODEPOSIT FOR ELECTRONIC APPLICATIONS

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Abstract

The physical properties of deposits obtained from a new palladium electroplating bath are discussed. Data on ductility, contact resistance, corrosion resistance, hydrogen uptake, and preferred orientation of the deposits are presented and compared to those obtained using a traditional palladium plating formulation.

Introduction

Electrodeposited palladium, both pure and alloyed, over-plated with a thin layer of gold has proved to be a suitable contact finish for many electronic applications (1, 2, 3, 4).

Its acceptance by design and production engineers, however, has been slow because of several factors. Hard gold electrodeposits work well; they have been an industry standard for many years and, although expensive to produce, their costs have been acceptable even though gold has been valued at $300 - $400 per troy ounce. These factors combined with poor traditional palladium plating processes, deposits that have been stressed, and palladium's reputation for catalytic activity have worked against the acceptance of this metal as a contact material.

As reliable electrolytes are formulated and data on the properties of these electrodeposits are accumulated, palladium will increasingly be recognized as a viable connector material. The object of this paper is to describe an electrolyte which has been formulated specifically for high speed electroplating of connectors or stripstock, and to add to the information base on Palladium as a deposit.

The Plating Process

Throughout the paper we will compare the electrolyte operation and deposit properties of this novel palladium process (5) with a traditional formulation based on palladium diammine dinitrite (6).

A comparison of operating parameters is given in Table 1. The traditional electrolyte is composed of a solution of ammonium sulfamate and depends upon the NH_3/NH_4^+ buffer for pH stability. The excess ammonium ions also stabilize the palladium tetraammine complex, particularly at the anode. Under rack plating conditions the process is adequate provided that frequent additions of ammonium hydroxide solution are made. At higher deposition speeds and the high-aeration conditions of most reel to reel plating lines ammonia is driven off at a rapid rate. This causes pH instability, palladium salt precipitation on the anodes and operator discomfort.

In designing a high speed palladium plating solution this shortcoming was overcome by eliminating the offending buffer system and replacing it with a combination of a stable palladium complex, a simple conducting salt which does not hydrolyze and a salt buffer which is especially effect at inert anode surfaces where local pH tends to fall dramatically due to destruction of hydroxide ions. The end result is a solution which can be operated over a wide pH and temperature range without instability or offence to the operator's sense of smell.

Deposition speed is important if a plating process is to be applied in a selective deposition mode in, for example, the connector industry. Typical hard gold plating solutions deposit at rates of up to 10 microinches per second (15 micrometers/minute) in suitable equipment. Although even higher speeds are in use, it is usual to operate at approximately 3 microinches/second (4.5 micrometers/minute).

Traditional pure palladium electrolytes cannot even approach these speeds. However, the new formulation under discussion here can comfortably match 3 microinches/second at 180 to 200 A/ft^2 (19.4 to 21.6 A/dm^2) with reasonable agitation in order to provide good mass transfer. This result indicates a cathode efficiency of about 85% or 28 mg/Amp. min. Efficiency remains largely constant over a wide range of pH values and current densities.

The Deposit Properties

Hydrogen Content, Ductility and Stress

One of the major factors limiting use of palladium deposits has been their tendency to crack after plating is completed. This is caused by codeposited hydrogen leaving the deposit. This phenomenon has been well documented and measurement of the hydrogen content of deposits has been established and reported (7, 8) for both pure and nickel alloyed palladium deposits. Reasonable ductility is a requirement for any contact finish that might be subjected to mechanical or thermal deformation during subsequent manufacturing operations or in its service life. Deposit cracks might expose the substrate materials to corrosive environments and lead to high contact resistance in the connector assembly.

The measured hydrogen content of these deposits are reported in table 2, and the ductility, internal stress and microhardness are compared with those for a typical hard gold deposit in table 3.

Ductility has been measured by elongating deposits of 50 microinch (1.25 micrometer) thickness on a soft copper foil substrate. The integer percentage elongation prior to the appearance of cracks in the deposit is the figure quoted. Under no test condition was the elongation of the high speed formulation deposit less than 4% and most results fall into the range of 6-8%. The traditional formula and the typical hard gold gave at best 2% and 3.5% respectively. An observation made during testing was that the P -salt deposits cracked abruptly at a given elongation, indicative of a brittle material. Deposits from the new palladium formulation developed small cracks which slowly expanded on further elongation. (figures 1, 2 and 3).

In the light of these results it might be expected that internal stress of the deposits would be ranked inversely. There was evidence during testing that the deposits produced in the traditional palladium solution were stress relieving themselves during plating (figure 4). Hard gold is well-known for its relatively poor ductility which often necessitates the use of soft gold pre-plate layers as a hard gold foundation.

Porosity And Contact Resistance

The porosity of these deposits (a property often referred to as corrosion resistance) was evaluated using copper substrates and a moist sulfur dioxide atmosphere. It is noteworthy that the traditional nitric acid vapor test is not appropriate for testing palladium since this metal is soluble in nitric acid. The method employed was similar to that of Leeds and Clark (10).

Panels 3 inches by 1 inch (7.5 cm x 2.5 cm) were plated with 40 microinch (1 micrometer) and 80 microinch (2 micrometer) thick deposits of palladium or gold. Pores and cracks through to the substrate copper were rendered visible by the copper salts of the corrosion product on the precious metal surface. A summary of the results is given in table 4.

The test conditions are quite severe as shown by the porosity in the hard gold standard. The traditional palladium corroded badly, probably due to cracking more than classical porosity. The excellent result for the new palladium agrees well with Updegraff's (11).

Contact resistance was measured on flat test panels using a modification of ASTM B539-80 which has been reported previously (12). A minimum of 25 readings per square centimeter were taken automatically at a load of 100 grams. Results are expressed in table 5 and show no anomolies.

Crystal Orientation

X-ray diffraction patterns were obtained for both types of palladium deposit and compared to that from finely divided palladium powder which can be considered to have random crystal orientation. The peaks chosen for analysis were (200) at 46.6 degrees, (220) at 68.2 degrees and (222) at 86.6 degrees. Diffraction intensities are reported in table 6. From these figures it is possible to calculate relative intensities; relative to the powder sample, that is. Also by expressing the relative intensity of each peak as a fraction of the total intensity for each sample we can more clearly see any preferred orientation that might exist. A normalized intensity, arrived at this way, which exceeds 0.33 indicates a preferred orientation.

It is clearly illustrated by these results that the traditional P-salt electrolyte produces a deposit with virtually random orientation. Also the new formulation produces deposits the crystals of which are strongly oriented in the (220) plane (edge-up), at the expense of the (200) plane (face-up). The (222) plane (corner-up) is not present at all.

Conclusions

An electroplating solution has been developed which will operate at high deposition speeds without excessive maintenance or operator discomfort.

The deposit produced by this process shows a definitive crystal orientation and low hydrogen content leading to excellent ductility, freedom from porosity and good contact resistance properties.

Acknowledgement

The authors wish to recognize Dr. W. O. Mc Sharry and Dr. J. E. Phillips of the Materials Characterization Laboratory, Electronic Products Group, Engelhard Corporation for their work in producing the x-ray data and analysis.

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Table 1. Comparison of operating conditions the Palladium plating processes.

	High Speed	P-Salt
Pd. metal , g/L	25	15
Conducting and buffering salts, g/L	50	100
pH	8.5	8.0
Temp. , °C	50	35
Test C.D., A/ft^2 (A/dm^2)	60 (6.5)	10 - 20 (1.1 - 2.2)
Production C.D., A/ft^2 (A/dm^2)	$\leq$ 350 ($\leq$ 37.8)	10 - 20 (1.1 - 2.2)

Table 2. Comparison of the hydrogen content of Palladium deposits.

	High Speed	P-Salt
H/Pd atomic ratio	0.004	0.02
Weight percent	0.0038	0.019
NmL H_2/g Pd	0.42	2.1

Table 3. Ductility, internal stress and microhardness of two types of pure palladium deposits and a typical hard gold deposit.

	High Speed Pd	P-Salt Pd	Hard Au
Ductility (elongation, %)	4 - 8	<2	<3.5
Internal Stress (N/mm^2) (all tensile) *	255	225	190
Microhardness (Knoop, 25g)	200	275	175

* Dvorak and Vrobel method (9).

Table 4. Summary of porosity counts for palladium and gold. Substrate was pure copper foil, the atmosphere was moist SO_2 for 24 hours at 25°C.

Deposit thickness (average)	Average pore count/in^2		
	High Speed Pd	P-Salt Pd	Hard Au
40 microinches	3	*	>100
80 "	3	*	>100

* Too corroded to count pores accurately.

Table 5. Contact resistance of palladium and hard gold deposits.

All deposits were 40 microinches (1 micrometer) average thickness and were tested on the "as plated" condition. Load 100g.

	High Speed Pd	P-Salt Pd	Hard Au
Contact resistance, mΩ	2.0 - 4.5	2.0 - 3.7	1.8 - 2.5

Table 6. X-ray Diffraction Data for palladium powder and the two deposits.

	Crystal Plans	High Speed Pd	P-Salt Pd	Pd Powder
Relative Intensities (%)	(200)	23.8	54.3	63.2
	(220)	76.2	34.2	29.4
	(222)	-0-	11.4	7.3
Relative Orientation	(200)	0.36	0.81	1.00
	(220)	2.71	1.32	1.00
	(222)	-0-	1.38	1.00
Normalized Preferred Orientation	(200)	0.12	0.23	0.33
	(220)	0.88	0.37	0.33
	(222)	-0-	0.39	0.33

E-5

Electronics II
Session E

Palladium Electroplating

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PALLADIUM ELECTROPLATING

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Introduction

All electronic devices contain interconnects that enable an electrical signal—and hence a message—to be transmitted from one component system to another, and any such component must provide a stable pathway for such transmission. This is true for hardware as simple as a wall socket or printed wiring board fingers in a complex system such as a circuit pack in an electronic switching system. In the case of electronic switching systems, computer hardware and most telecommunication equipment, high reliability and low-noise requirements are the determining factors in designing interconnects and selecting electrical contact materials.

Several criteria are used to determine the suitability of contact materials. Contacts and the associated contact materials must be conductive and withstand a wide variety of environmental conditions while maintaining stable contact resistance. In addition, contacts must withstand being connected to and disconnected from other contacts without appreciable physical deterioration, which could lead to unstable electrical resistance. The contact material must therefore be corrosion and wear resistant. Additional functional requirements are imposed on specific connector systems taking into consideration the design and geometry of the connector, the particular contact pressure, the cycles of operation, the ambient conditions to which it is exposed and the expected lifetime.

Thus, while copper and silver are the most conductive metals available, they are not acceptable contact materials in most electronic applications because they corrode easily and show poor wear performance.

On the other hand, gold, while not as conductive as silver or copper, is still a good electrical conductor and is extremely resistant to corrosion. In addition, high-percentage gold alloys exhibit excellent bondability, solderability, low contact resistance and wear properties, and are easily electroplated to form high-quality, stable contact surfaces. Thus, the use of thick electroplated Au films for contact surfaces has been the standard in the electronics industry.[1–3]

Reliance on gold-based contact technology is not without a price, however. In 1972, gold sold for $59 a troy ounce. By 1980, it hit a high point of about $800 a troy ounce and averaged roughly $500. Also in 1980, the United States used about 22.5 metric tons—724,000 troy ounces worth $362 million—of gold in electronic devices. The estimated gold usage in the entire western world was roughly 60 metric tons worth approximately one billion dollars. About 56 per cent of this gold was used in connector contacts and 23 per cent went into printed wiring board fingers.

The highly competitive nature of the electronics market demanded a less-costly, reliable contact finish as a substitute for gold, and research in this area became extremely active.[3–6] Non-precious metals were an area of interest.[7–12] For example, plain solder contacts were acceptable in some consumer electronic applications with design lives of only five years. However, for high-reliability applications and the bulk of telecommunications component systems, platinum-group metals were considered as the most likely alternatives. Of the pure, non-alloyed metals examined, only ruthenium and palladium were serious contenders because they cost much less than gold. For example, recently the price of gold was $460 per troy ounce whereas palladium cost $130 per troy ounce.

Ruthenium was examined for use in relay switches,[13–15] but for separable connectors its use was precluded because of poor wear resistance and insurmountable problems with high-speed electrodeposition processes.

Palladium (Pd), a platinum group metal, has been viewed as a suitable alternative. The lower price of Pd metal (Figure 1) coupled with its lower electroplated density (Pd $\sim$ 10 g/cm^3 vs. Au $\sim$ 17.3 g/cm^3) implies considerable cost savings on utilizing Pd as a contact material (Figure 2). However, despite these economic advantages there has not been an appreciable demand for Pd electrodeposition. This is due to the relative difficulty of electrodepositing Pd with satisfactory physical properties, paucity of contact reliability data and little impetus to develop such a process due to dependence on the well established and reliable Au electroplating technologies.

The recent acceptance of palladium and gold flashed palladium (GFPd) as contact materials reflects the resolution of these technical problems[16–24] and the availability of cost effective reliable electrodeposition processes.

Palladium Electrodeposition

The electrochemical deposition of Pd has many difficulties. The most prominent is the evolution and incorporation of H_2 which is a reaction that can occur simultaneously with Pd deposition and leads to H_2 embrittlement (Figure 3 and 4). This codeposition of H_2 and Pd leads to the formation of palladium hydride alloys (PdH_2).[25]

Palladium and palladium hydrides have what is known as a face centered cubic, or "FCC" structure, and in palladium hydrides that FCC structure occurs in an alpha and a beta phase. Pure palladium, at 3.889 angstroms, and alpha palladium hydride, at 3.893 angstroms, have similar lattice constants and are both mechanically stable.[26–27, 29]

However, the beta-palladium hydride, which contains significantly higher levels of hydrogen, has an FCC structure whose lattice, at 4.04 angstroms, is greater than pure palladium.[28, 29] The hydrogen embrittlement problem, then, stems from the thermodynamic instability of the beta-palladium hydride alloy—which spontaneously evolves hydrogen and decomposes to the alpha-palladium hydride alloy. This phenomenon is accompanied by corresponding lattice contractions and changes in the mechanical properties of the palladium films, resulting in highly-stressed and cracked deposits.[25] The extent of hydrogen incorporation and embrittlement, therefore, depends on the extent of codeposition during palladium electroplating.

Codeposition is determined by the relative reduction potential of palladium and hydrogen—a function of the thermodynamic stability of the palladium-complex, the kinetics of reduction, the overpotential of hydrogen on palladium and the operating conditions of the system.[30]

Many palladium electroplating chemistries are highly susceptible to hydrogen embrittlement, and this severely limits their effectiveness. In addition, many have a tendency to dissolve the base metal used as a substrate for the contact, and in the worst cases the contact disintegrates and contaminats the plating solution with other metals. Some chemistries are also plagued by the presence of volatile components, the formation of precipitates, and chemical or electrochemical degradation of solution constituents[30] (Figure 5).

Overall, these difficulties cannot be easily controlled in a large-scale manufacturing environment. Thus, there was a need to develop a palladium electroplating chemistry for mass production. By 1980 a systematic investigation of palladium electrochemistry at AT&T Bell Laboratories led to the development of a palladium electrodeposition process.[30–36]

This process produces palladium electrodeposits with the desired physical characteristics under a wide range of conditions. It is a simple chemistry employing only three chemical constituents: the palladium complex, a phosphate buffer, and a palladium replenishing compound to replace the metal removed by the deposition process (Figure 6).

Materials Properties

An important aspect of any electrodeposition technology is understanding the relationship between the operating parameters of the electrodeposition process and the material properties of the deposited films. Extensive studies have shown that while palladium electroplated with this process is harder than gold, it is slightly more ductile. Palladium electroplated with this chemistry — which is also less dense than

electroplated hard gold — is slightly purer in composition and thermally more stable (Figure 7). In short, this process provides palladium deposits whose physical properties are comparable to gold.[30, 31, 36]

Gold Flashed Palladium

Palladiumm is a catalyst for many organic reactions, and early research showed that in ambients of organic gases, frictional polymerization on the surfaces of vibrating contacts could form polymers, which act as non-conducting films.[37, 38] Palladium is also more susceptible to corrosion than gold,[18, 19] especially in high-chloride environments,[22, 23] such as chemical manufacturing and seashore areas.

To overcome these potential problems, a thin gold layer of 1 to 2 microinches is deposited over the palladium surface, resulting in what is known as "gold-flashed" palladium.[24] This very thin gold film (Figure 8) on one or both mating contacts eliminates polymerization, improves corrosion resistance and acts as a lubricant which reduces friction and improves wear.[16, 17, 24] Finally, the gold-plated palladium looks very much like gold, making the finished product more appealing to the customer.

Contact Reliability

An extensive series of environmental and wear tests has been performed on AT&T connector hardware with gold-flashed, palladium-plated contacts. These contacts have been exposed to a variety of tests designed to simulate a 20- to 40-year service life span. In all cases, gold-flashed palladium performs as well as or slightly better than comparable gold contact finishes (Figure 9).[24] As a result of this testing, gold-flashed palladium is now being used as a contact material for numerous AT&T products.

Technology Transfer

The ultimate test of any process, no matter how successful it is in the laboratory, is its performance in a manufacturing environment. The manufacturing potential of this particular technology has been demonstrated by numerous successful engineering trials, and its subsequent incorporation into the manufacturing process at several AT&T Technologies plants (Figures 10-13), and non AT&T Manufacturting locations.[36, 39] This palladium electrodeposition process requires no special facilities, and no extraordinary plating techniques are used: the gold bath in an existing plating line is simply replaced by palladium. This facilitates the transition from gold to palladium, since capital plant expenditures required for the conversion are minimal.

Since its introduction to commercial use in 1981, system designers have begun to specify gold-flashed palladium in their products. Printed wiring board fingers, loop plant transmission equipment, backplane interconnections hardware, and telephone handset parts such as modular cord jack spring contacts are among the products now made with gold-flashed palladium.

PALLADIUM vs GOLD
YEARLY AVERAGES

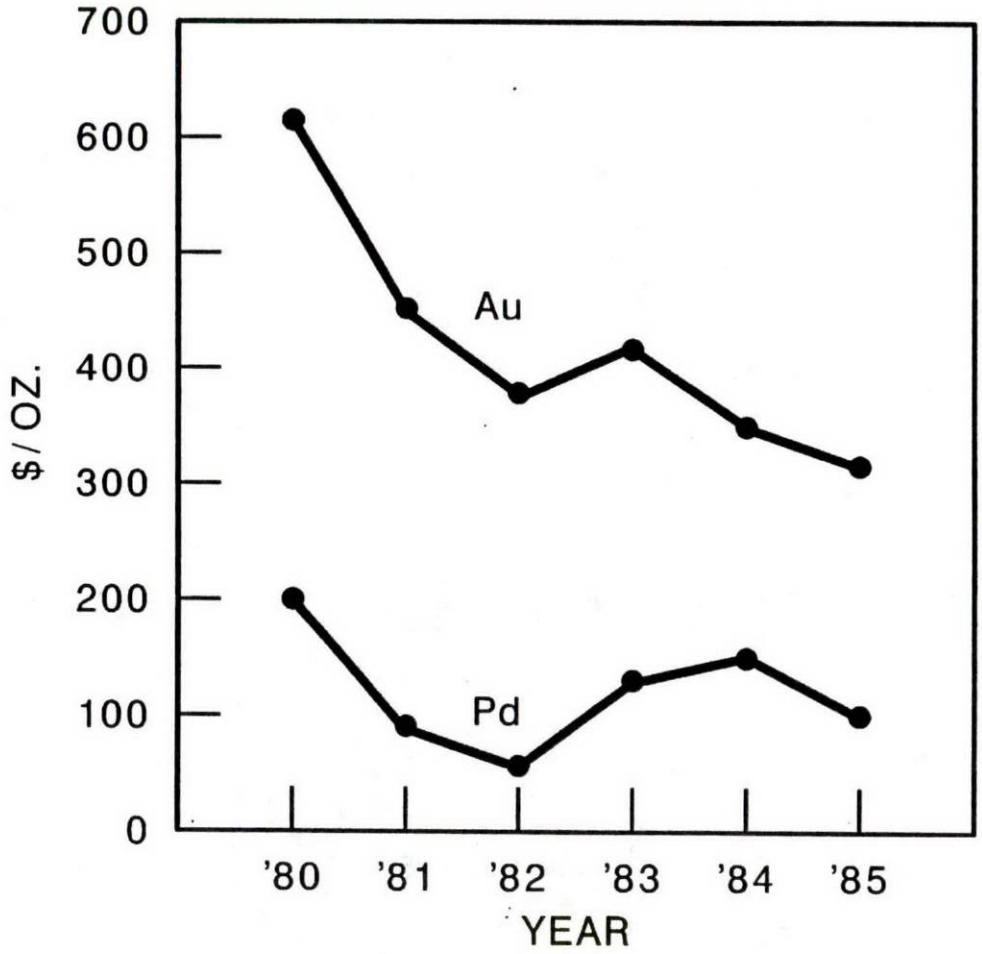

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FIGURE 1. PALLADIUM vs GOLD YEARLY AVERAGES (1980-1985) DOLLARS PER TROY OUNCES.

SOURCES OF COST SAVINGS

- THE COST OF PALADIUM IS SUBSTANTIALLY LESS THAN THAT OF GOLD
- THE DENSITY OF PALLADIUM IS 60% THAT OF GOLD; THEREFORE, IT TAKES FEWER TROY OUNCES TO PLATE TO A GIVEN THICKNESS WITH PALLADIUM THAN GOLD
- THE DENSITY OF THE AT&T PALLADIUM PLATE IS APPROXIMATELY 85% OF THE THEORETICAL, WHICH IS AN ADDITIONAL MATERIAL SAVINGS

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FIGURE 2. SOURCES OF COST SAVINGS ON REPLACING Au WITH Pd.

HYDROGEN EMBRITTLEMENT PROBLEM

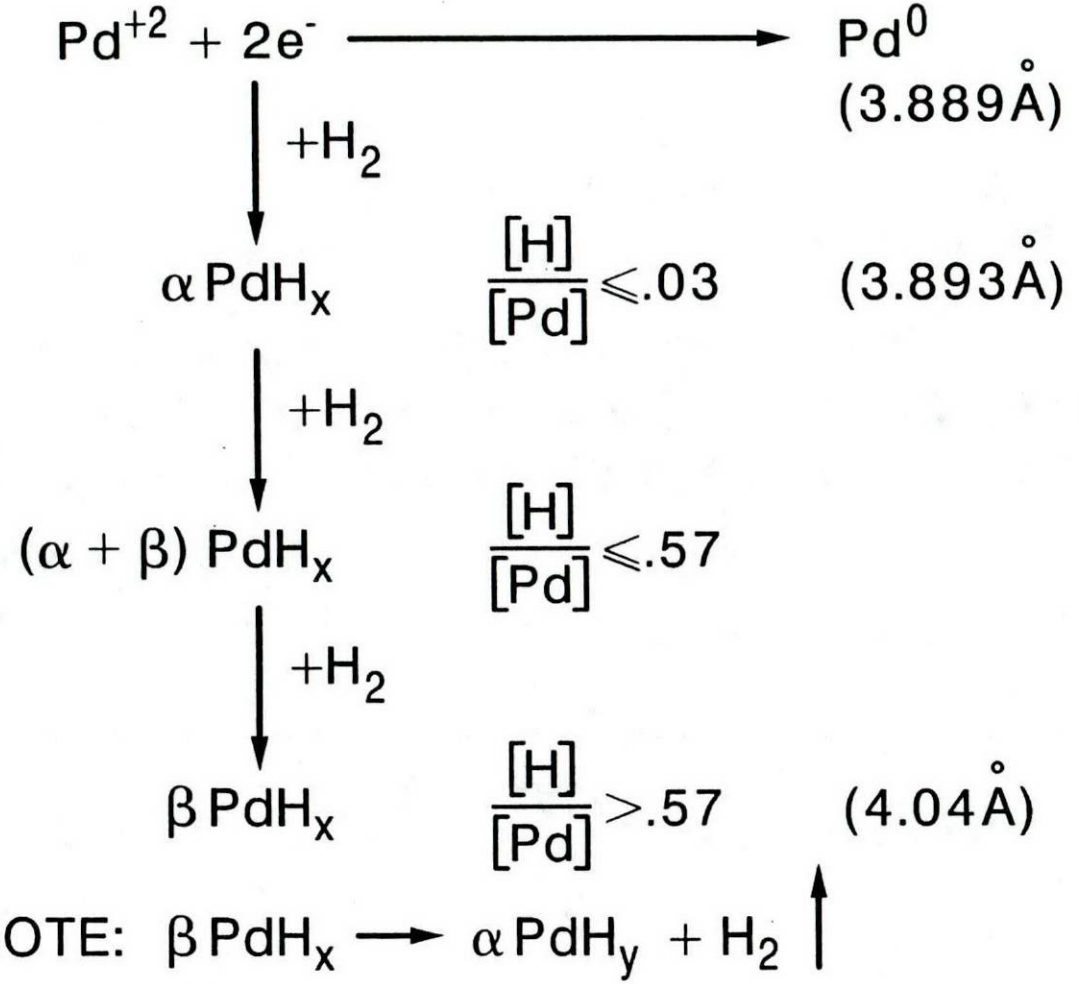

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FIGURE 3. THE HYDROGEN EMBRITTLEMENT PROBLEM.

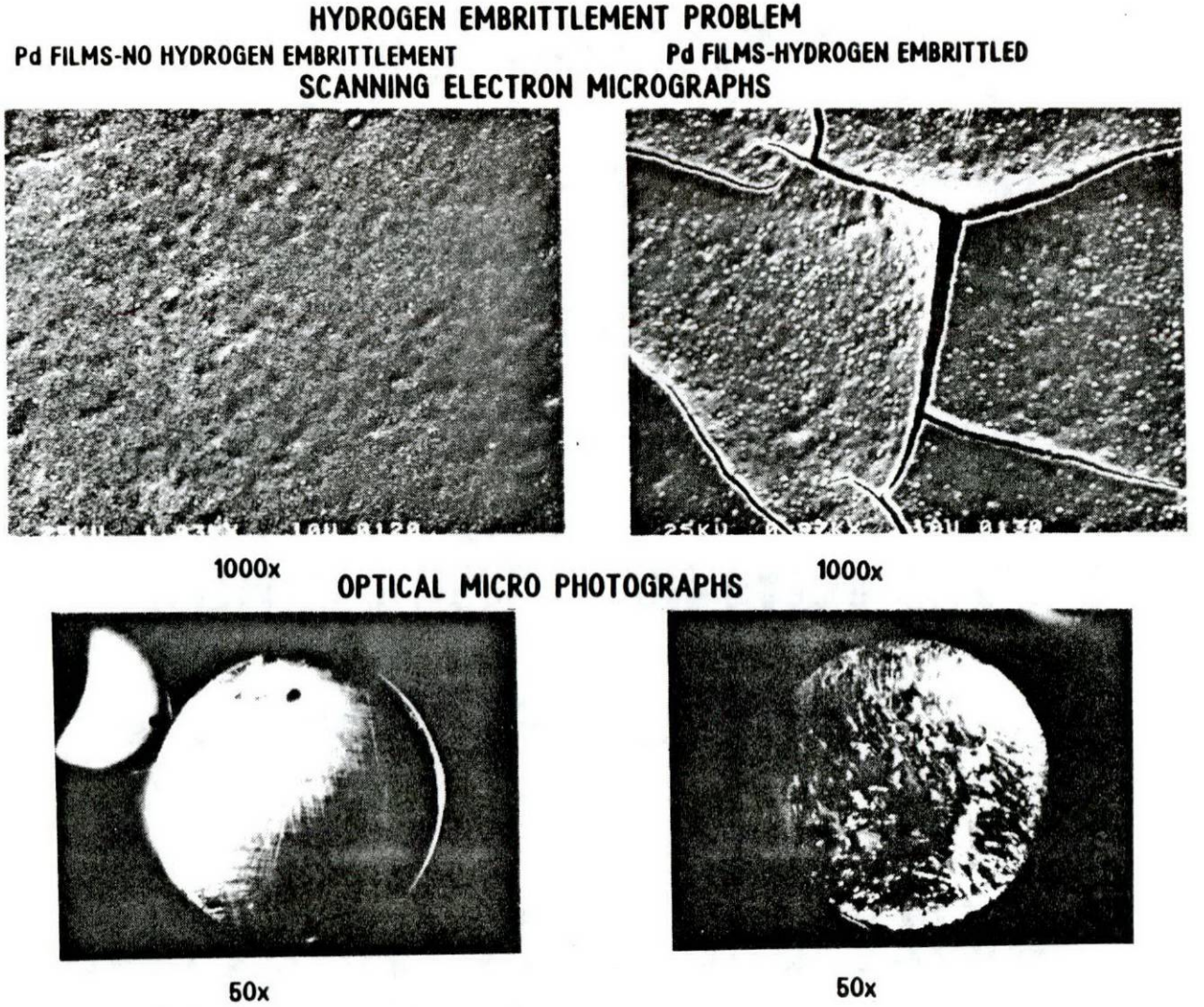

FIGURE 4. SCANNING ELECTRON MICROGRAPHS OF Pd DEPOSITS WITH AND WITHOUT HYDROGEN EMBRITTLEMENT.

PROBLEMS ENCOUNTERED WITH PALLADIUM ELECTROPLATING

- HYDROGEN EMBRITTLEMENT
- LIMITED DEPOSITION RATES
- UNSTABLE CHEMICAL SYSTEMS
- UNSTABLE BUFFER SYSTEMS
- CHEMICAL ATTACK OF BASE METAL
- LIMITED PROCESSING WINDOWS

FIGURE 5. LIMITATIONS OF PALLADIUM PLATING CHEMISTRIES.

PALLADIUM ELECTRODEPOSITION CHEMISTRY

- PALLADIUM CHELATE COMPLEX
- ALKALINE PHOSPHATE BUFFER
- SIMPLE REPLENISHMENT SYSTEM

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FIGURE 6. CONSTITUENTS IN THE AT&T Pd PLATING CHEMISTRY.

COMPARISON OF FILM PROPERTIES

PALLADIUM VERSUS HARD GOLD

	PALLADIUM	HARD GOLD
HARDNESS (KHN_{25})	250-350	140-200
HARDENING AGENTS	NONE	Co,Ni
GRAIN SIZE (Å)	30-100	200-250
DENSITY	9.5-10.5	~17.3
DUCTILITY (% ELONGATION AT 50 µin)	3.5-5.0	2.5-3.5
VOLATILE IMPURITIES (WT%)	<0.5	$\leqslant 1.5$
METALLIC IMPURITIES (WT%)	$<<1.0$	$\leqslant 1.0$
THERMAL STABILITY (^0C)	$\geqslant 360$	$\leqslant 150$

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FIGURE 7. COMPARISON OF FILM PROPERTIES OF PALLADIUM VERSUS HARD GOLD.

WHAT IS THE FUNCTION OF THE GOLD CAP?

- LUBRICANT
- REDUCES POTENTIAL FOR FRICTIONAL POLYMER FORMATION
- MINIMIZES PALLADIUM CHLORIDE FORMATION
- GIVES APPEARANCE OF GOLD

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FIGURE 8. REASON FOR PLATING Au CAP ON Pd.

PALLADIUM CONTACT FINISH EVALUATION

RESULTS

- IN ALL TESTS, FOR ALL PRODUCTS EVALUATED, 25 µin PALLADIUM OVERPLATED WITH 1 µin GOLD FINISHES PERFORMED EQUAL TO OR BETTER THAN STANDARD GOLD FINISH
- NO DATA EVEN SUGGESTS ANY CONCERN THAT WOULD PRECLUDE IMMEDIATE IMPLEMENTATION

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FIGURE 9. SUMMARY OF CONTACT RELIABILITY STUDIES PERFORMED ON GFPd.

LOW SPEED BARREL PLATING PARAMETERS AND CONTROLS

PHYSICAL & CHEMICAL PARAMETERS

• [Pd]	0.75 - 1.5g/l
• [pH]	11.5 - 12.2
• T (°C)	47 - 53°C
• [LIGAND]	0.1 - 0.5M
• SPECIFIC GRAVITY	1.15 - 1.30
• [PHOSPHATE]	~ 0.5M

DAILY CONTROLS

- [Pd]
- [Cu]
- pH
- T (°C)
- [LIGAND]
- SPECIFIC GRAVITY
- PLATING EFFICIENCY - WEIGHT GAIN ANALYSIS
  THICKNESS MEASUREMENTS

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FIGURE 10. OPERATING CHARACTERISTICS AND DAILY PROCESS CONTROLL PARAMETERS FOR BARREL PLATING OPERATION.

LOW SPEED BARREL PLATING
Summary

- PLATED CONTINUOUSLY > 2 YEARS
- PLATED ~190 MILLION PARTS/BATH
- PLATED > 200 BATH TURNOVERS
  - LITTLE MAINTENANCE
  - NO CARBON TREATMENT

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FIGURE 11. SUMMARY OF BARREL PLATING OPERATIONS.

REEL TO REEL HIGH SPEED PLATING PARAMETERS AND CONTROLS

PLATING PARAMETERS

- CONTROLLED DEPTH PLATING CELL
- CELL LENGTH ~ 6 FT.
- LINE SPEED 20 FT/ MIN
- PLATING SPEED 80 - 120 ASF
- PALLADIUM THICKNESS ~ 25 μ"; X-RAY FLUORESCENCE

DAILY PROCESS CONTROLS

- CONCENTRATION - Pd, Cu, Ni, LIGAND
  pH, SG, T(°C), CD (ASF)
- MICROCRACKING - SEM 3000X
- THICKNESS
- ADHERENCE
- ELECTROGRAPHIC POROSITY

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FIGURE 12. OPERATING CHARACTERISTICS AND DAILY PROCESS CONTROL PARAMETERS FOR HIGH SPEED REEL TO REEL PLATING.

SUMMARY OF PRODUCTION DATA
High Speed Reel to Reel Plating

- PLATED CONTINUOUSLY FOR 7 MONTHS
- PLATED 3659 REELS/ 182,950,000 PINS
- PLATED 8.5 - 9.0 BATH TURNOVERS
  WITH LITTLE MAINTENANCE
  WITH NO CARBON TREATMENT

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FIGURE 13. SUMMARY OF HIGH SPEED REEL TO REEL OPERATIONS.

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