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BDX-613-1709 (Rev.)

DEVELOPMENT OF PLATING THICKNESS
STANDARDS

Milestone Report

R. J. Russell, Project Leader

Project Team:
G. A. McWhirt
M. G. Ulitchny

Published January 1978

Prepared for the United States Department of Energy
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DEVELOPMENT OF PLATING THICKNESS STANDARDS

BDX-613-1709 (Rev.), UNCLASSIFIED Milestone Report,
Published January 1978

Prepared by R. J. Russell, D/424

Standards which are unavailable from the National Bureau of Standards have been developed to support the nondestructive measurement of plating thickness. Their fabrication, measurement, certification, and calibration-recall schedule are discussed. Reference standards that have been put into service include aluminum/Kapton, silver/copper, tin/steel, gold/silver, cadmium/Kovar, silver/iron, rhodium/copper, and gold/ceramic.

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SUMMARY

Plating thickness standards which are unavailable from the National Bureau of Standards have been developed to support the nondestructive measurement of plating thickness for products manufactured by Bendix Kansas City. Their fabrication, measurement, certification, and calibration-recall schedule are briefly discussed. This information is provided as a guide for the future fabrication of other plating thickness standards.

Reference standards that have been fabricated and put into service since September 25, 1975 include aluminum/Kapton (E. I. du Pont de Nemours & Co.), silver/copper, tin/steel, gold/silver, cadmium/Kovar (Westinghouse Electric Corporation), silver/iron, rhodium/copper, and gold ceramic.

Working standards will be purchased commercially, when possible, and their thickness will be verified by comparison to the reference standards.

DISCUSSION

SCOPE AND PURPOSE

This project was initiated to develop standards to support the nondestructive measurement of plating thickness. These standards, unavailable from the National Bureau of Standards (NBS), are required for the inspection of Bendix Kansas City products. Working standards will be purchased from a commercial source, when available.

The techniques that were chosen to determine the plating thickness of the Bendix reference standards consisted of weight gain, interferometry, and profile measurements. Each method allows direct traceability to the NBS and a reliable estimate of the measurement uncertainty. Working standards are assigned thickness values by comparison to the reference standards through the use of a nondestructive measurement method such as beta back-scatter.

ACTIVITY

Background

Nondestructive plating thickness measurements, supported by the use of standards supplied by the manufacturer of the measuring equipment, have been performed at Bendix Kansas City for some time. To insure traceability to NBS and to provide confidence in the working thickness standards, reference standards had to be fabricated. This report describes several methods that were used to verify the thickness of the plating that was deposited on the reference standards.

Fabrication of Standards

The selection of a stable substrate is of the utmost importance in the development of plating thickness standards. The base material that was chosen was machined, annealed, and lapped to conform to Grade II gage-block tolerances for flatness and parallelism. These substrates then were plated by either vacuum deposition or electroplating.

Figures 1 and 2 show the masks and plating fixtures that were fabricated and used to define the area upon which the plating was deposited. This procedure allowed the thickness of the deposit to be measured by several independent methods. Photolithography techniques were used to etch a desired area pattern for the gold-on-ceramic thickness standards.

Figure 1. Plating Mask

Plating-Thickness Measurements

The techniques that were chosen to determine the plating thickness consisted of weight gain, interferometry, and profile measurements. A minimum of two methods of measurement were used, when possible, to provide independent methods of evaluating the measurement reliability. The techniques used are described in the following paragraphs.

Figure 2. Plating Fixtures

Weight Gain

A stable substrate was selected and cleaned before plating. The mass of each sample was determined before and after deposition of the plating by a Mettler Model M-5 microbalance which has a precision of $3 \mu\text{g}$ and an estimated uncertainty of less than $20 \mu\text{g}$. As will be shown, the error contributed by this equipment was found to be minimal.

The area of the deposited material was defined by a mask. (Photolithography techniques, which also have been used to define the plated area, require etching of the deposit.) The area then was measured with a SIP Trioptic Measuring Machine. The limiting factor of this measurement was the edge definition of the plated area; the estimated uncertainty was $\pm 0.008 \text{ cm}^2$ on the gold-on-ceramic thickness standards.

The density of the deposit was assumed to be the theoretical density of the bulk material. This assumption must be verified by experiment to minimize a large possible source of error. The thickness was determined by the following equation.

$$t = \frac{m}{dA}, \quad (1)$$

where

t = plating thickness,

m = plating mass,

d = plating density, and

A = plating area.

The following equation is used to determine the maximum resulting error.

$$\frac{\Delta t}{t} = \frac{\Delta m}{m} + \frac{\Delta d}{d} + \frac{\Delta A}{A}. \quad (2)$$

For gold on ceramic, this equation is used to perform the following calculation.

$$\frac{\Delta t}{t} = \frac{3}{15000} + \frac{0.5}{19.3} + \frac{0.008}{2.85},$$

$$\frac{\Delta t}{t} = 0.03 = 3 \text{ percent.}$$

Interferometry

A Link gage block interferometer was used to make direct thickness measurements (Appendix A). The substrate, which had been lapped flat and parallel within $0.05 \mu\text{m}$ (2 microinches), was measured for thickness using the interferometer. The substrate then was masked and the plating material was deposited. After the resulting composite thickness (substrate plus plating) had been measured, the plating thickness was obtained by subtracting the substrate thickness from the composite thickness.

The interferometer has the disadvantage of being a contact-type of instrument. The readings obtained therefore require corrections for stylus deformation. Correction curves were developed by varying the force of the stylus and measuring the resulting film thickness. The changes in measured film thickness versus the stylus force were plotted, and the curve was forced through a

force value of zero at zero deformation to produce a correction graph. This type of graph must be generated for each material that is measured.

The estimated uncertainty in the thickness determination would be $\pm(0.05 \mu\text{m} + \text{deformation correction error})$ or $\pm(2 \text{ microinches} + \text{deformation correction error})$. For aluminum on quartz, this would be $\pm 0.08 \mu\text{m}$ ($\pm 3 \text{ microinches}$).

Profile

A Clevite Model 1200 Surface Analyzer was used to make the profile measurements. By this method, the specimen was measured after the plating had been deposited by moving a stylus across the substrate and onto the plated material. As the transition was made from the substrate to the plating, a step, proportional to the thickness of the plating, was shown in the output of the recorder. By applying a proportionality constant, determined through the use of a lever-arm calibrator, the thickness then was calculated. The estimated uncertainty of this measurement is $\pm(0.05 \mu\text{m} + \text{estimated error due to surface roughness})$ or $\pm(2 \text{ microinches} + \text{estimated error due to surface roughness})$.

Certification of Reference Standards

Reference standards are measured by a minimum of two independent techniques, and their thickness is determined by averaging the measured values. The standards then are certified for thickness and put on a calibration-recall schedule. Upon recall, they are inspected for visual damage and either recertified or replaced.

Every five years (or some other interval of time approved by the calibration engineer), one thickness standard is remanufactured and compared by a nondestructive method such as beta backscatter to the standards in service. If the certified thickness of this sample agrees with the thickness obtained by beta-backscatter comparison, the set of reference standards is recertified and put back into service; if unacceptable, it must be rejected and a new set must be manufactured. Figure 3 shows members of a set of reference standards that are now in service, and Tables 1 and 2 show typical sets of data that were obtained during the measurement of the standards indicated.

Certification of Working Standards

Reference standards are used to calibrate the working standards. The comparison is performed with beta backscatter or some other acceptable nondestructive thickness-measuring equipment. The equation of the calibration line is determined by using the data obtained from the reference standards (counts/second versus thickness, using beta backscatter).¹ This equation is applied to

Figure 3. Reference Thickness Standards: Silver on Copper (Above); and Gold/Chromium/Ta₂N on Ceramic (Below)

the data obtained from the working standards to determine their thickness. The working standards then are certified and put into service. Periodic calibrations are performed to maintain the integrity of the working standards by making certain that they are replaced when they become worn or damaged.

Data Analysis and Certification

The following technique is used to assure a uniform method of dealing with the data obtained during the recalibration of working standards.

Table 1. Thickness Measurements of Gold-On-Silver Thickness Standards

Thickness Standard Serial Number	Thickness Measured by Method Indicated*				
	Link Interferometer		Weight Gain		Difference (Percent)
	μm	Microinches	μm	Microinches	
2	3.53	139	3.38	133	4.3
3	1.19	47	1.14	45	4.4
4	4.06	160	4.01	158	1.3
5	8.20	323	7.92	312	3.4
6	2.31	91	2.24	88	3.3
7	0.69	27	0.66	26	3.7
8	13.72	540	13.34	525	2.9
9	11.53	454	11.13	438	3.5
10	5.54	218	5.69	224	2.7

*Original measurements were in microinches; $\rho_{\text{Au}} = 19.3 \text{ g/cm}^3$. Thickness is determined by averaging the measured values. Estimated uncertainty does not exceed ± 5 percent of assigned thickness.

Table 2. Thickness Measurements of Gold/Ta₂N/Chromium-On-Ceramic Thickness Standards

Thickness Standard	Thickness Measured by Method Indicated*					
	Weight Gain		Profile		Difference	
	μm	Microinches	μm	Microinches	μm	Microinches
#1	1.47	58	1.40	55	0.08	3
#6	1.88	74	1.83	72	0.05	2
#11	2.21	87	2.34	92	0.13	5
40C2-5	2.21	87	2.26	89	0.05	2
60C1-5	3.25	128	3.20	126	0.05	2
80C2-7	4.37	172	4.42	174	0.05	2
100C1-6	5.31	209	5.41	213	0.10	4

*Original measurements were in microinches; Ta₂N thickness = 50 nm (500 Å); Cr thickness = 20 nm (200 Å). Thickness is determined by averaging the measured values. Estimated uncertainty does not exceed ±5 percent of assigned thickness.

Data are collected from each of the reference standards. In addition to estimating the relationship between parameters for the purpose of prediction, certain inferences to be made concerning the calibration line also are of interest.

In the case of linear curves, the slope, intercept, and general quality of the regression line are of importance. A computer program is available to perform this evaluation. The resulting equation is used to determine the thickness of each working standard. If the determined thickness is within established limits (Appendix B), it is averaged with the results of all previous calibrations and the average is reported. If the determined thickness is not within the established limits, the standard is replaced and the set is recertified.

A similar technique is used for nonlinear curves. Because a computer routine for nonlinear curves has not been implemented, the calibration engineer must determine the merit of the calibration data, the adequacy of the data fit, and the quality of the calibration performed. The results are used to determine whether the standards are replaced or recertified.

ACCOMPLISHMENTS

Several sets of plating thickness standards have been fabricated and put into service. A systematic routine for manufacturing and measuring the standards has been developed and implemented. The reference standards shown in Table 3 have been fabricated or acquired since September 25, 1975.

FUTURE WORK

Cadmium-on-copper and copper-on-aluminum plating thickness standards must be fabricated and certified.

Additional plating thickness standards will be fabricated and certified as the need arises to support the nondestructive inspection of new products.

Table 3. Reference Plating Thickness Standards Fabricated
or Acquired Since September 25, 1975

Reference-Standard Materials	Range	
	μm	(Unit Indicated)
Aluminum/Kapton	9.4 to 12.7	370 to 500 Microinches
Silver/Copper	2.5 to 12.7	100 to 500 Microinches
Tin/Steel	1.5 to 19.1	60 to 750 Microinches
Gold/Silver	0.5 to 10.2	20 to 400 Microinches
Cadmium/Kovar	2.5 to 25.4	100 to 1000 Microinches
Silver/Iron	3.8 to 22.9	150 to 900 Microinches
Rhodium/Copper	0.5 to 3.2	20 to 125 Microinches
Gold/Ceramic	3.0 to 10.0	30,000 to 100,000 Å

REFERENCES

¹ R. J. Russell, *Development of Plating Thickness Standards for the Physical Vapor Deposition of Aluminum on Kapton* (Milestone Report). UNCLASSIFIED. Bendix Kansas City: BDX-613-1634, September, 1976 (Available from NTIS).

² R. E. Walpole and R. H. Myers, *Probability and Statistics for Engineers and Scientists*. New York: Macmillan, 1972.

Appendix A

LINK INTERFEROMETER

The Link interferometer, manufactured by Link Division General Precision, Inc., Binghamton, New York, is a combination interferometer and bidirectional digital counter. By using the optics and light source shown in the optical schematic (Figure A-1), the interferometer converts displacement into interference fringes. The system employs an automatic electronic counting circuit to count the interference fringes and to display the result on a digital counter.

A mechanical schematic of the Link interferometer (Figure A-2) and a photograph of the interferometer anvil and stylus (Figure A-3) follow the optical schematic.

Figure A-1. Optical Schematic of Link Interferometer

Figure A-2. Mechanical Schematic of Link Interferometer

Figure A-3. Link Interferometer Anvil and Stylus

Appendix B

DETERMINATION OF DEVIATION LIMIT FOR A MEASURED THICKNESS

To determine an acceptable limit for the deviation of a measured thickness from the certified thickness value, the following equation was used and the indicated calculations were performed.²

$$\epsilon^2 = \frac{\left(\frac{Z_\alpha}{2} + Z_B\right)^2 \sigma^2}{n} \quad (B-1)$$

Assuming a normal distribution where

ϵ = maximum allowable change from nominal,

α = Pr (Type I error) = 0.05,

$Z_{\alpha/2}$ = 1.96,

B = Pr (Type II error) = 0.05,

Z_B = 1.645,

σ^2 = population variance, and

n = sample size,

then for $n = 4$,

$$\epsilon^2 = \frac{(1.96 + 1.645)^2 \sigma^2}{4},$$

and

$$\epsilon = 1.8 \sigma.$$

For aluminum on Kapton, and using Probe 25649,

$$\epsilon = 1.6(4) = 6.4 \text{ microinches} = 162.56 \text{ nm}.$$

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