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BDX-613-2032 (Rev.)

PLATING THICKNESS STANDARDS

By R. J. Russell

Published January 1979

Final Report

Prepared for the United States Department of Energy
Under Contract Number DE-AC04-76-DP00613.

**Kansas City
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Printed in the United States of America

Available From the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161.

Price:	Microfiche	\$3.00
	Paper Copy	\$4.00

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By R. J. Russell

Published January 1979

Final Report
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A variety of standards that have been developed and fabricated for the nondestructive measurement of plating thickness are listed. The development and fabrication of TiB_2 -on-steel thickness standards are described. Pure-copper-foil reference units were provided for use in the four-point resistance measurement of copper-on-epoxy-board plating thickness.

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A prime contractor with the United States
Department of Energy under Contract Number
DE-AC04-76-OP00613

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SUMMARY

This report identifies the reference standards that have been developed for nondestructive measurement of plating thickness. These standards are retained in the Bendix Metrology Laboratory for use in the calibration of transfer and working standards.

The development and fabrication of TiB_2 -on-steel plating thickness standards are described. The vapor-deposition of TiB_2 on the face of tool-steel molds used in the manufacture of parts from abrasive material will significantly increase the life of the molds.

Efforts to simplify the reproduction, decrease the cost, and increase the durability of plated-through-hole reference units through the development of synthetic stainless-steel hole samples have been abandoned. Copper reference units continue to be used since the stainless steel did not provide a favorable balance between contact-probe force and hole-material ductility.

Pure-copper-foil standards of known thickness were found satisfactory for use as reference units in the four-point resistance measurement of copper-on-epoxy-board plating thickness.

DISCUSSION

SCOPE AND PURPOSE

This project was initiated to develop standards for 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 have been 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, weight loss, interferometry, and profile measurements. Each method allows 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 backscatter or four-point resistance measurements.

PRIOR WORK

The techniques of fabrication, measurement, certification, and control of the plating-thickness standards developed and fabricated have been discussed in previous reports.^{1,2,3,4}

High-precision beta-backscatter equipment was developed to support the calibration of plating-thickness standards.²

ACTIVITY

Reference Standards in Service

A variety of plating-thickness reference standards are retained in the Metrology Laboratory for use in the calibration of transfer and working standards. Tables 1 and 2 list the standards that have been developed or obtained as a result of this effort.

The thickness of the reference standards is measured by a minimum of two independent techniques, whenever possible, and the assigned thickness value is determined by averaging the measured values. Such techniques as weight gain, weight loss, interferometry, step-height, and interference spectroscopy have been used. Beta backscatter is the most prominent method now used for both product inspection and the calibration of working standards.

Table 1. Beta-Backscatter Reference Standards
Provided to the Nondestructive-Testing
(NDT) Laboratory

Plating	Substrate	Nominal Plating Thickness Range (μm)
0.15-Percent Copper-Doped PVD Aluminum	Kapton (Du Pont Corporation)	8.7 to 12.5
Copper	Kapton	7.0 to 12.0
PVD Aluminum	Kapton	3.0 to 12.0
TiB ₂	Steel	0.5 to 3.5

Table 2. Beta-Backscatter Standards Provided
to Product-Inspection Departments

Plating	Substrate	Nominal Plating Thickness Range (μm)
Cadmium	Copper	2.0 to 25.0
	Steel	3.8 to 25.0
Copper	Aluminum	1.8 to 9.0
	Epoxy	5.0 to 100.0
Gold	Ceramic	2.3 to 10.0
	Kovar*	0.5 to 7.5
	Nickel	0.5 to 10.0
	Silver	0.5 to 15.0
Rhodium	Copper	0.7 to 3.0
	Nickel	0.5 to 3.2
Silver	Copper	2.5 to 22.5
	Steel	2.5 to 22.5
TiB	Steel	0.5 to 3.5
Tin	Steel	1.5 to 20.0

*Westinghouse Electric Corporation

TiB₂-On-Steel Plating Thickness Standards

At the present time, Bendix Kansas City uses tool-steel molds to form parts made from abrasive material. The molding process quickly wears the mold to an out-of-tolerance condition which requires the manufacturing of a new mold. A vapor-deposition process now is being developed to deposit TiB₂ on the face of the molds. TiB₂ is highly resistant to wear, and it will significantly increase the life of the molds.

The development of the TiB₂ vapor-deposition process requires a method for determining the thickness of the deposited coating. By this means, variables such as wear resistance and adhesion can be related to the coating thickness. Beta backscatter was selected as the measurement method to be used.

Beta backscatter is a comparative measurement process which requires the use of plating thickness standards. The standards employed were fabricated to meet a design requirement for measuring the coating thickness within ± 10 percent. The reference standards were fabricated by the following described method.

A set of tool-steel wafers was machined to a diameter of 25 mm and a thickness of 6 mm. Each sample face was lapped flat to within 0.25 μ m. The samples then were cleaned by the same process that will be used on the molds, after which they were placed in the deposition chamber and sputtered with TiB₂. When the desired thickness had been attained, the system was shut down and allowed to cool.

The samples were removed from the deposition chamber, and their thickness was measured with a surface analyzer, using the profile mode. Two measurements, 90 degrees apart, were performed on each sample. Because the measurement traverses the sample, a step-height is recorded at each edge; a typical trace is shown in Figure 1.

The standards that have been fabricated by the described process are listed in Table 3. These standards were used to calibrate a set of reference units for development activity. The thickness values of the reference units and the standards were compared by beta backscatter, using a C₁₄ source. The reference units that have been put into service are listed in Table 4.

Reference Units for Plated-Through Holes

Sheets of copper-clad epoxy are used at Bendix Kansas City for fabricating printed circuit boards. Many of these boards have copper cladding on both sides, and the etched circuit patterns are interconnected, where necessary, by the use of plated-through holes.

Figure 1. Typical Calibration Trace of TiB_2 -On-Steel Plating
Sample Obtained From a Surface Analyzer

The inspection of plated-through holes is performed with a tester that was developed in the Bendix Advanced Inspection Engineering department.⁵ This tester can be used for making direct measurements of the thickness of the copper in the hole walls. The method used consists of acquiring and processing resistance data from the test hole to obtain an average value of wall thickness of the plating around the circumference of the hole.

The probe system used with the tester has eight individual contacts: four on the top side of the hole, and four on the bottom. To determine the resistance of a hole, a constant current is introduced from a top probe contact to the corresponding bottom contact. A voltage measurement then is made between the similar set of contacts that is located 180 degrees from the current contacts. The voltage measurement obtained is converted to resistance by a digital resistance-measuring system whose output is converted to thickness by a microprocessor.

The plated-through-hole tester requires a periodic check to assure that the probe tips are functioning correctly, and that the results obtained accurately indicate the hole-wall thickness. This function is performed through the use of reference units.

Table 3. TiB_2 -On-Steel Plating
Thickness Standards
Fabricated by
Sputtering

Standard Designation	Plating Thickness (μm)
14694-1	1.19
14694-2	1.27
14694-3	2.67
14694-4	3.43
14694-5	1.52
14694-6	2.44
14694-7	0.66
14694-8	2.29

Table 4. TiB_2 -On-Steel
Reference Units
in Service

Reference- Unit Designation	Plating Thickness (μm)
R9B5-1	0.97
R9B4-7	1.65
R7B2-1	2.46
R6B5-7	3.12
R9B1-1	4.01

The reference units consist of plated-through-hole samples that exhibit the same resistance value as that of a sample whose thickness has been determined by measuring a cross section of the hole wall under a microscope.

The reference units that are now used do not provide either the durability or ease of reproduction that is desired. In an effort to attain these goals, a set of synthetic plated-through-hole samples were fabricated from stainless-steel tubing having the same inside diameter as the plated-through hole. (Stainless-steel tubing was selected because it is durable, readily available, and relatively low in cost.) Several cylindrical slices of the 1.5-mm-thick tubing were measured for thickness using the plated-through-hole tester. The resistance values that were obtained proved to be excessively high with some degree of variability between successive measurements.

Copper plating then was deposited on the outside of the stainless-steel tubing to a thickness of 0.013 mm, and samples were prepared as before. Subsequent testing revealed that the resistance values had dropped significantly, but variability in successive measurements had increased. Careful investigation showed that the stainless steel was not deforming enough to allow the probe to make good contact. While the problem could be minimized by increasing the contact force, this action would cause permanent damage to the probe contacts.

The search for a more durable reference-unit material was not pursued further because of the belief that progress must come through the selection of a material that is more ductile than stainless steel. Again, this might solve the contact problem, but the durability of the new material might prove to be no greater than that of the copper reference units that are currently in service. Thus the work to develop plated-through-hole reference units having greater durability has been abandoned.

Copper-On-Epoxy-Board Thickness Standards

Four-point resistance-measuring techniques are used to determine the thickness of copper foil and copper plating (thin films) on printed-circuit-board material. This method consists of measuring the sheet resistance as a function of a specified distance on the surface of the copper. Resistance values are converted to conductance values to take advantage of the linear relationship between the conductance and the corresponding material thickness. The thickness of sample parts is obtained from the reference-unit thickness-versus-conductance relationship, as determined by calibration.

Figure 2 shows a four-point probe that is used for this type of measurement. As indicated, the probes are arranged in a straight line. The two outside tips are the current leads, and the two inside tips are used for measuring the voltage drop. The resistance is measured with an ohmmeter.

Each measuring-instrument-and-probe combination has slight differences which require the development of a separate calibration curve. Since copper plating, copper foil, and a combination of both materials will be measured, an experiment was performed to determine whether independent calibration curves would be required for each material.

Specimens of copper foil and copper plate were measured with a four-point resistance-measuring system, after which they were sectioned at the point where the resistance measurement had been taken, and the thickness was determined with a calibrated microscope. The results that were obtained are shown in Tables 5 and 6. The resistance values are converted to conductance and plotted as a function of thickness in Figure 3. As indicated in the illustration, both the copper foil and the copper plate lie along a least-squares line. A least-squares line was fitted through the data, since a linear relationship exists between the conductance values and the corresponding material thicknesses.⁶

The conclusion was reached from the graphical data that copper foil and copper plate can be used interchangeably within the range of 0.01 to 0.1 mm without introducing significant error. Pure-copper-foil standards of known thickness therefore can be used as reference units.

Four-point resistance measurements were performed on samples from a set of reference units having a known thickness. The results obtained are shown in Table 7, and a plot of the results is shown in Figure 4. The following equation represents the least-squares best-fit line that was determined by a calibration of the equipment used:

Thickness (in mm) = $4.789 \times 10^{-6} \times \text{Conductance (in S)} - 0.0016$.

The resulting correlation coefficient of this fit was 0.9992.

The equation of the least-squares line fitted through the data obtained will be used to determine product thickness.

ACCOMPLISHMENTS

Several sets of standards have been fabricated and put into service to support the nondestructive measurement of plating thickness. A systematic procedure for manufacturing and verifying plating thickness standards has been developed and implemented.

Figure 2. Four-Point Probe Used for Resistivity Measurements of Thin Films

TiB₂-on-steel plating thickness standards have been developed and fabricated. The vapor-deposition of TiB₂ on the face of tool-steel molds used in the manufacture of parts from abrasive material will significantly increase the life of the molds.

Pure-copper-foil standards of known thickness have been found satisfactory for use as reference units in the four-point resistance measurement of copper-on-epoxy-board plating thickness.

FUTURE WORK

The Bendix Metrology Laboratory will provide maintenance and calibration service for plating-thickness standards. New standards will be developed, fabricated, and put into service by the Metrology Laboratory using techniques that have been developed.

Table 5. Thickness-Measurement Data From Copper Clad and Foil

Material Designation	Microscopically Measured Thickness (μm)	Resistance ($\text{m}\Omega$)	Conductance (kS)
0.0005 Foil*	12.4	0.298	3.356
0.0008 Foil**	20.7	0.184	5.420
Thin Clad A	06.5	1.239	0.807
Thin Clad B	06.5	1.320	0.758
1-mil A***	26.6	0.169	5.923
3-mil A†	72.5	0.063	15.830
3-mil A-A	64.6	0.069	14.49
3-mil A-B	63.5	0.070	14.29
5-mil A††	122.1	0.037	26.790
5-mil A-A	115.3	0.030	26.32
5-mil A-B	115.3	0.039	25.60

*0.0005 in. = 12.7 μm .

**0.0008 in. = 20.3 μm .

***1 mil = 25.4 μm .

†3 mils = 76.2 μm .

††5 mils = 127.0 μm .

Table 6. Thickness-Measurement Data From Copper Plate

Material Designation	Microscopically Measured Thickness (μm)	Resistance ($\text{m}\Omega$)	Conductance (kS)
598918-5A	100.1	0.040	25.11
598918-5A-1	104.4	0.040	25.00
598918-5B-1	106.4	0.039	25.6
598918-5C	105.2	0.040	25.00
598919-13E	35.5	0.121	8.26
598920-5A	76.5	0.058	17.24
598920-5B	74.7	0.058	17.24

Figure 3. Comparison Between Copper-Foil and Copper-Plate Thickness Specimens Using Four-Point Resistance Testing

Table 7. Data Obtained From Copper Plate
on 0.006-mm Copper-Clad Epoxy
Using 0.0625 Probe*

Sample	Determined Thickness (μm)	Conductance (kS)
1	6.8	1.148
2	13.6	2.342
3	21.2	4.484
4	32.3	6.623
5	41.1	8.772
6	60.5	13.513
7	82.6	18.519
8	139.2	29.412
9	175.5	37.037
10	215.4	47.619
11	404.9	83.333

*0.0625 (in.) = 1.5875 mm.

Figure 4. Conductance Versus Thickness for Copper Plate on 0.006-mm Copper-Clad Epoxy Using 0.0625 (1.5875 mm) Probe

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BDX-613-2032 (Rev.)

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