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PLATING THICKNESS STANDARDS

Milestone Report

R. J. Russell, Project Leader

Project Team:
G. J. Hale
G. A. McWhirt
D. M. Sherman

Published August 1978

Prepared for the United States Department of Energy Under Contract Number EY-76-C-04-0613.

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Communications Services Bendix Kansas City Division

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Standards which are unavailable commercially or from the National Bureau of Standards have been developed to support the nondestructive measurement of plating thickness. Reference and working standards of copper on Kapton and aluminum on Kapton have been put into service. Their fabrication, measurement, certification, and calibration recall are discussed.

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A prime contractor with the United States Department of Energy under Contract Number EY-76-C-04-0613

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SUMMARY

Because aluminum-on-Kapton and copper-on-Kapton plating-thickness standards are not available from the National Bureau of Standards or any known commercial source, standards have been developed and fabricated at Bendix Kansas City. Interferometry and weight-gain methods were used to determine the metal thickness on each of the reference standards. The reference standards were used to certify the thickness of several sets of working standards by comparison using beta-backscatter methods for aluminum on Kapton and four-point resistance measurements for copper on Kapton.

All working standards have been placed on a calibration schedule for periodic inspection for damage and comparison with a set of reference standards. By this technique, each working standard either will be recertified and returned to service or replaced with a certified standard.

The precision of the reference standards was evaluated by a series of statistical tests that were performed on each set of calibration data. These tests provided an effective means for determining the quality of the data and estimating the measurement uncertainty. The measurement uncertainty of the thickness values assigned to the working standards should not exceed $\pm 0.26\ \mu m$ for aluminum on Kapton and $\pm 0.43\ \mu m$ for copper on Kapton.

DISCUSSION

SCOPE AND PURPOSE

This project was initiated to develop plating-thickness measurement standards for use with nondestructive thickness-measuring equipment. These standards, unavailable from the National Bureau of Standards or any commercial sources, are required to support the inspection of products at Bendix Kansas City and the Los Alamos Scientific Laboratories (LASL). When available, working standards will be purchased from a commercial source.

The development and fabrication of thickness standards and the resulting plating-thickness standards will benefit both Bendix Kansas City and LASL by making possible the nondestructive measurement of plating thickness on products with a high degree of precision.

This report describes the methods used to verify the thickness of the plating that was deposited on the reference standards together with those used for the fabrication, calibration, and calibration-recall scheduling of the working standards.

ACTIVITY

Background

A variety of plating-thickness standards have been fabricated at Bendix Kansas City.[1] As a result of the experience gained, Bendix was requested by LASL to fabricate plating-thickness standards of aluminum on Kapton and copper on Kapton. Two sets of working standards of each material were fabricated to support LASL's immediate needs and, in addition, two sets of working standards were furnished to the Bendix Kansas City Nondestructive Testing Laboratory. All working standards will be scheduled for calibration by Bendix Metrology Laboratory personnel (Figure 1).

Fabrication of Standards

The fabrication of plating-thickness standards begins with the preparation of the substrate. Each different type of standard fabricated requires its own special substrate preparation. For example, the optical flats which provide a stable reference surface for the deposit must be measured for thickness and mass. They are cleaned with solvent and placed in a holding fixture similar to the one in Figure 2.

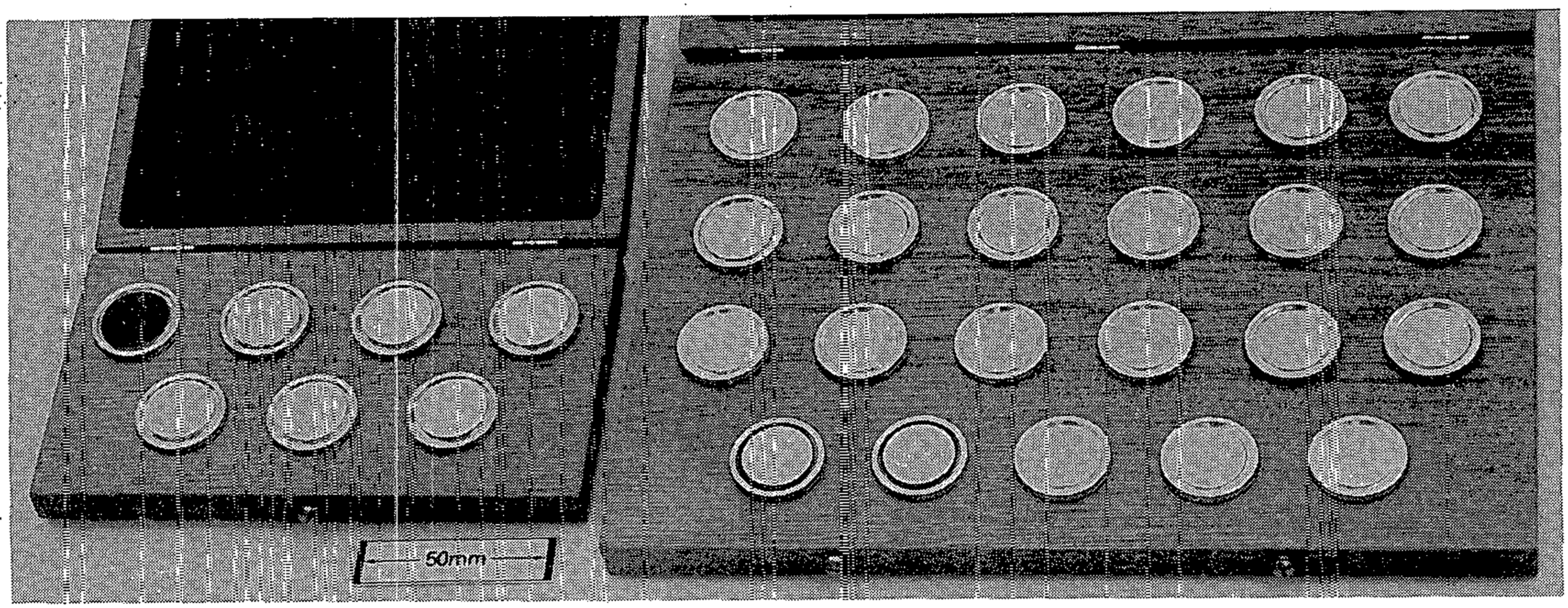

Figure 1. Working Plating-Thickness Standards (Left) and Reference Standards (Right) of Aluminum on Kapton

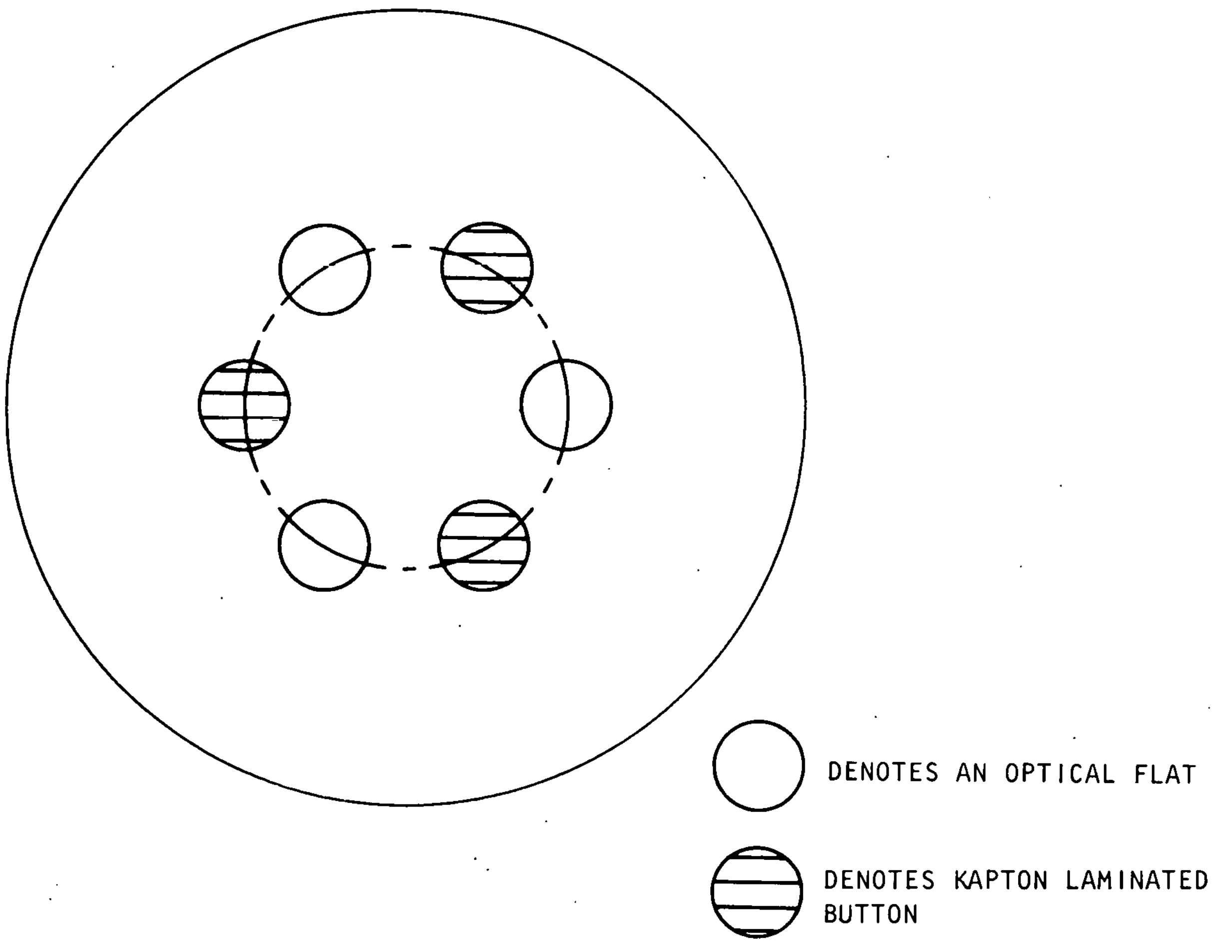

Figure 2. Placement of Kapton Laminated Buttons and Optical Flats in the Vapor-Deposition Fixture

The substrates for working standards were fabricated from two flat pieces of Kapton laminated together and cut to size (55 by 280 mm). These substrates were used for the fabrication of the aluminum working standards and for the first five runs of copper working standards. However, because of the high deposition temperature of copper, the laminated material had a tendency to delaminate. The subsequent six runs therefore were changed to a single layer of 0.127-mm Kapton. The described strips of Kapton were mounted in curved fixtures which allowed the metal to be uniformly deposited. Two fixtures were used for each run.

The substrates for the reference standards were fabricated by punching 19-mm discs from a Kapton sheet which was identical in composition to those used for the working standards. After these discs had been laminated to aluminum housings, the assemblies, called *buttons*, were cleaned and mounted in the holding fixtures along with an equal number of optical flats (Figure 2).

The fixtures were loaded into a motor-powered planetary system inside the vacuum chamber, and they were rotated as the metal was being deposited. The deposition rate was controlled through the use of a quartz microbalance inside the chamber. When the metal had attained the desired nominal thickness, the system was shut down and allowed to cool before the optical flats, buttons, and the material for the working standards were removed.

The thickness of the metal on the optical flats was measured with a Link Gage Block Interferometer, and the determined average thickness value was assigned to the three buttons that had been fabricated during the deposition. Comparative measurements indicated that the difference between the thickness of the metal deposited on the optical flats and the thickness of the metal deposited on the buttons was insignificant.[2] The buttons from this deposition were placed in a set which was designated *reference standards*. Tables 1 and 2 identify the reference standards that were fabricated.

The working standards necessary to support production measurements were fabricated by bonding small die-punched sections of the metal-coated Kapton sheets to aluminum housings. Beta-backscatter techniques were used to compare the thickness of the aluminum working standards with that of the reference standards. A four-point resistance measurement was used to compare the thickness of the copper working standards.

A schedule for periodic calibration-recall assures the continued integrity of the working standards. If the standards become worn or damaged, they will be replaced with new sections. When the available supply of working-standard material has been exhausted, the described fabrication process must be duplicated to obtain additional working standards.

Thickness of Reference Standards

The thickness of the metal deposited on the optical flats was determined with a Link Gage Block Interferometer by measuring the thickness before and after deposition (Figure 3).[1] The interferometer has a resolution of 3 nm and an estimated measurement uncertainty of 50 nm.

Because the interferometer has the disadvantage of being a contact-type instrument, corrections for stylus deformation are required. Figure 4 shows the correction curves that were developed. These curves were generated by varying the stylus force of

Table 1. Aluminum-Thickness Reference Standards

Reference-Standard Designation	Certified Thickness (μm)
AL-FL-1	10.67
AL-FL-2	10.67
AL-FL-3	10.67
AL-FL-4	10.11
AL-FL-5	10.11
AL-FL-6	10.11
AL-FL-7	9.53
AL-FL-8	9.53
AL-FL-9	9.53
AL-FL-10	11.38
AL-FL-11	11.38
AL-FL-12	11.38
AL-FL-16	11.76
AL-FL-17	11.76
AL-FL-18	11.76
AL-FL-19	9.12
AL-FL-20	9.12
AL-FL-21	9.12
AL-FL-22	10.57
AL-FL-23	10.57
AL-FL-24	10.57

the instrument and measuring the resulting film thickness. The changes in the film thickness versus the stylus force were plotted, and the curves were forced through zero deformation at zero force to produce the correction graph.

Table 2. Copper-Thickness Reference Standards

Reference-Standard Designation	Certified Thickness (μm)
CU-FL-4	7.21
CU-FL-5	7.21
CU-FL-10	11.81
CU-FL-11	11.81
CU-FL-12	11.81
CU-FL-16	10.41
CU-FL-18	10.41
CU-FL-25	9.78
CU-FL-26	9.78
CU-FL-27	9.78
CU-FL-28	8.76
CU-FL-29	8.76
CU-FL-30	8.76
CU-FL-31	11.05
CU-FL-32	11.05
CU-FL-33	11.05

The thickness values that were determined from the plated optical flats were corrected through the use of the appropriate curve. The resulting value is the thickness measured to the peak of the deposited material at zero load. The thickness values determined from the three optical flats were averaged and the averaged value was assigned to each of the buttons, as shown in Tables 1 and 2. The buttons then were placed in a set containing the reference standards that are now in service.

Thickness of Working Standards

The previously described reference standards were used to calibrate three sets of working standards. The thickness comparisons were performed through the use of beta-backscatter measuring equipment for aluminum on Kapton, and a four-point resistance measurement for copper on Kapton (Table 3).[3]

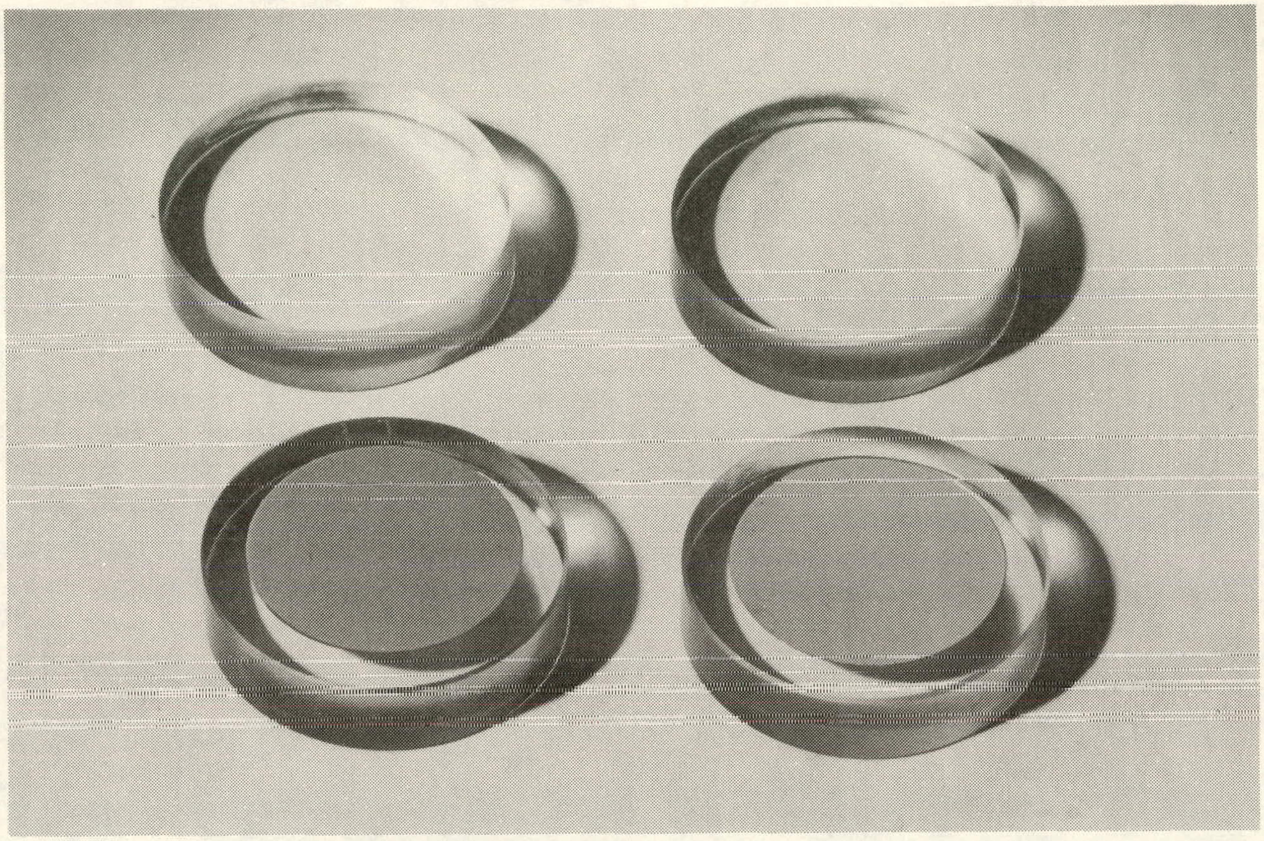

Figure 3. Optical Flats Before Deposition (Above) and After Deposition (Below)

The reference standards were measured, the instrument output was plotted with respect to thickness, and a least-squares linear regression was used to determine the equation of the line. Several statistical tests also were performed on the data simultaneously with the execution of the regression; these results are shown in Figures 5 and 6.

The thickness of the working standards is calculated by substituting the instrument output into the equation developed from the reference standards. An estimate of the measurement precision can be obtained from the calculated statistical tolerance limit shown in Figures 5 and 6. Graphic representations of the calibration curves are shown in Figures 7 and 8.

The working standards have been assigned thickness values and scheduled for routine, periodic calibration recall. At the end of the specified time interval, the standards will be inspected and recalibrated. If acceptable, they will be put back into service. An unacceptable standard will be replaced before the set is returned to the user.

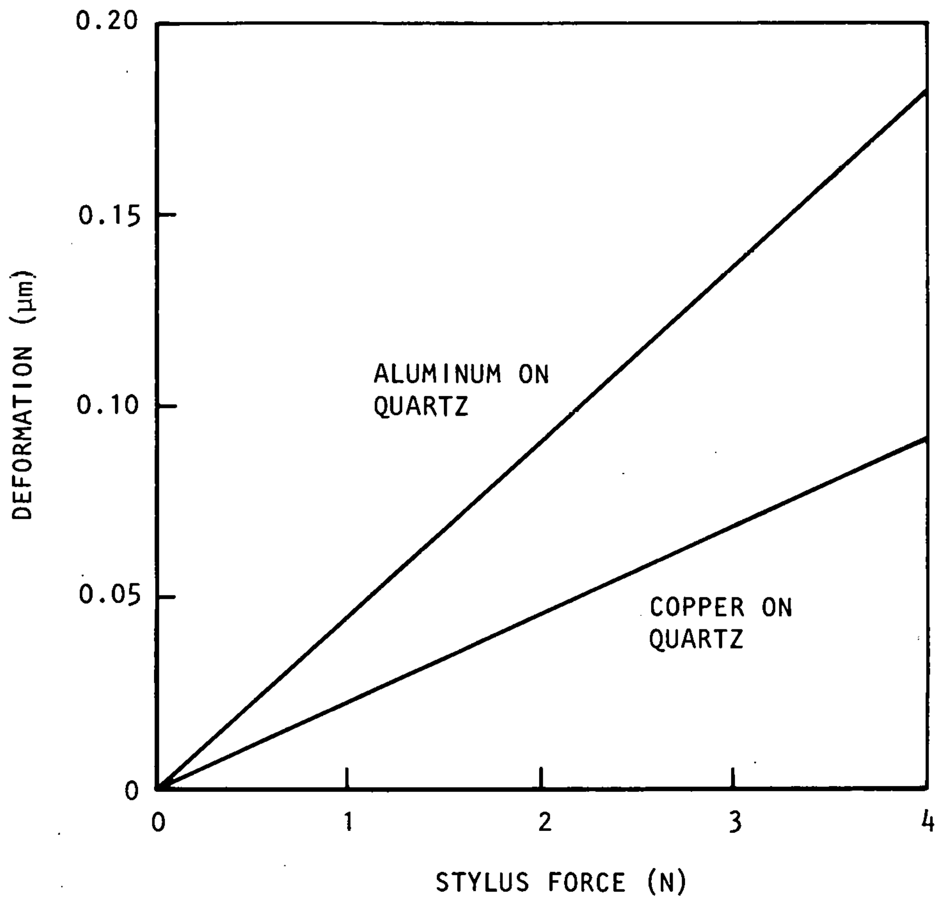

Figure 4. Deformation-Correction Graph for the Link Gage Block Interferometer

Deposit Characteristics

The following described tests were performed to assure satisfactory characteristics of the deposited metal.

Thickness Uniformity

Because the small area of metal that is deposited on each reference standard does not permit an investigation of the plating-thickness uniformity, flat strips of Kapton (Figure 9) were fabricated for this purpose. Prior to the deposition of the metal, the strips were placed in a curved fixture to provide a uniform distribution of the deposit. Upon completion of the deposition, the strips were removed from the fixture and allowed to return to their original flat configuration before the plating-uniformity measurements were made.

Table 3. Data for Least-Squares Regression

Aluminum/Kapton		Copper/Kapton	
(Counts/s)	Thickness (μm)	Conductance (S)	Thickness (μm)
2798.3	10.57	1536	7.214
2671.7	9.12	1520	7.214
2713.6	9.53	1984	8.763
2805.7	10.57	1972	8.763
2863.4	11.38	1976	8.763
2675.0	9.12	2165	9.779
2806.9	10.57	2198	9.779
2673.7	9.12	2203	9.779
2895.1	11.76	2356	10.414
2893.2	11.76	2331	10.414
2765.7	10.11	2564	11.049
2715.9	9.53	2532	11.049
2865.7	11.38	2577	11.049
2762.7	10.11	2681	11.811
2713.4	9.53	2762	11.811
2759.5	10.11	2732	11.811
2864.1	11.38		
2898.9	11.76		

The thickness of the metal was measured across the surface of the strips at 25.0-mm intervals, the locations of which are shown in Figure 9. Variations in the measured thickness are shown in Tables 4 and 5.

The deposits were found to be thickest at the center of the strips, and progressively thinner as the edges were approached. The largest variation was observed in the measurements made adjacent to the upper and lower edges. By eliminating these measurements from the data accumulated from the aluminum deposits, a thickness gradient of approximately 0.008 $\mu m/mm$ was obtained. The standard deviation of the thickness of the aluminum deposited in this area was found to be 0.13 μm.

```
RUN LINEAR

ENTER X Y PAIRS
2798.3     10.57
2671.7     9.12
2713.6     9.53
2805.7     10.57
2863.4     11.38
2675.0     9.12
2806.9     10.57
2673.7     9.12
2895.1     11.76
2893.2     11.76
2765.7     10.11
2715.9     9.53
2865.7     11.38
2762.7     10.11
2713.4     9.53
2759.5     10.11
2864.1     11.38
2898.9     11.76

MEAN SQUARE ERROR=  0.24861500E-02
STANDARD ERROR=  0.49861308E-01
COEFFICIENT OF VARIATION=       0.0048
CORRELATION=     0.998752

FOR A 90 PERCENT CONFIDENCE INTERVAL
SLOPE=   0.01199274   +/-   0.00026171
NORMALIZED INTERCEPT TOLERANCE=      0.0700
INTERCEPT=   -22.996437   +/-      0.729052
RUN TEST RESULTS:N=18   NR=10
XB= 2785.6948         YB=     10.4117        T=      1.7459        F=      1.7188

90% OF THE DATA POINTS FALL WITHIN THE LISTEDTOLERANCE 90% OF THE TIME
X=    2694.4199         Y=       9.3170+/-        0.1140
X=    2717.1399         Y=       9.5895+/-        0.1124
X=    2739.8599         Y=       9.8620+/-        0.1112
X=    2762.5798         Y=      10.1345+/-        0.1104
X=    2785.3000         Y=      10.4069+/-        0.1101
X=    2808.0200         Y=      10.6794+/-        0.1104
X=    2830.7400         Y=      10.9519+/-        0.1111
X=    2853.4600         Y=      11.2244+/-        0.1124
X=    2876.1799         Y=      11.4968+/-        0.1140
X=    2898.8999         Y=      11.7693+/-        0.1159
$
```

Figure 5. Aluminum-On-Kapton Data Reduction

In the case of the copper deposit, a thickness gradient of approximately 0.005 μm/mm was obtained, and the standard deviation of the thickness of the copper deposited in this area was found to be 0.08 μm.

```
RUN LINEAR

ENTER X Y PAIRS
1536.0      7.214
1520.0      7.214
1984.0      8.763
1972.0      8.763
1976.0      8.763
2165.0      9.779
2198.0      9.779
2203.0      9.779
2356.0      10.414
2331.0      10.414
2564.0      11.049
2532.0      11.049
2577.0      11.049
2681.0      11.811
2762.0      11.811
2732.0      11.811

MEAN SQUARE ERROR=  0.14659235E-01
STANDARD ERROR=  0.12107533E 00
COEFFICIENT OF VARIATION=        0.0121
CORRELATION=     0.996952

FOR A 90 PERCENT CONFIDENCE INTERVAL
SLOPE=  0.00385079  +/-  0.00014184
NORMALIZED INTERCEPT TOLERANCE=      0.0321
INTERCEPT=      1.280682  +/-      0.319921
RUN TEST RESULTS:N=16   NR= 9
XB= 2255.5625      YB=      9.9664        T=      1.7613        F=      1.7980

90% OF THE DATA POINTS FALL WITHIN THE LISTEDTOLERANCE 90% OF THE TIME
X=    1644.2000        Y=       7.6121+/-       0.2930
X=    1768.4000        Y=       8.0904+/-       0.2866
X=    1892.6000        Y=       8.5687+/-       0.2811
X=    2016.8000        Y=       9.0470+/-       0.2769
X=    2141.0000        Y=       9.5252+/-       0.2744
X=    2265.2000        Y=      10.0035+/-       0.2736
X=    2389.3999        Y=      10.4818+/-       0.2746
X=    2513.6001        Y=      10.9600+/-       0.2775
X=    2637.8000        Y=      11.4383+/-       0.2819
X=    2762.0000        Y=      11.9166+/-       0.2876
$
```

Figure 6. Copper-On-Kapton Data Reduction

From the test results, the uniformity of the metal deposit should be adequate to fabricate working standards capable of supporting present production activity.

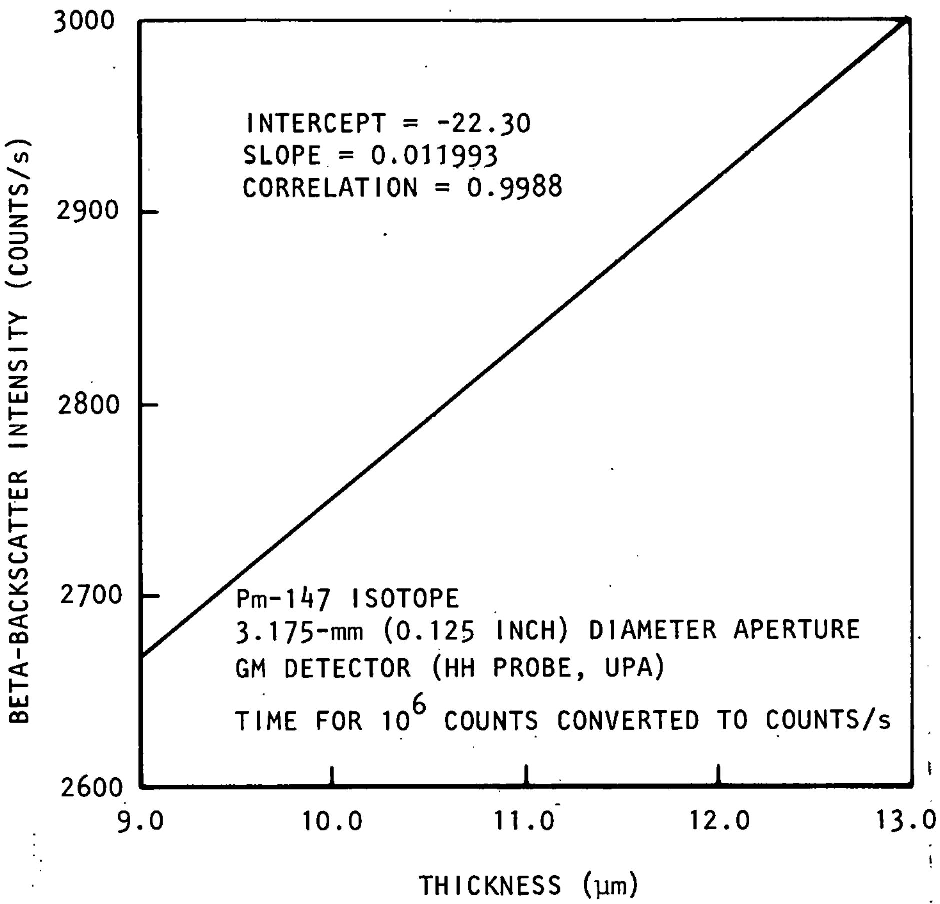

Figure 7. Beta-Backscatter Least-Squares Calibration Curve for Physical-Vapor-Deposited Aluminum on Kapton

Density

The density of each material deposited was determined by measuring the mass and volume of the plating. The measured volume required corrections for nonuniformity of diameter, taper at the edge of the plating (caused by fixture-shadowing), and surface roughness.

Table 6 shows an average value of 9.07 g/cm^3 for the density of physical-vapor-deposited copper; the estimated uncertainty in the determination was ±3 percent. The density value shown for aluminum (2.6 g/cm^3) has not been corrected for surface roughness.

Composition of Copper Deposit

The appearance of the deposited copper was observed to vary from bright and metallic to a reddish matte. The reddish color is caused by surface roughness which is produced by the high deposition temperature.

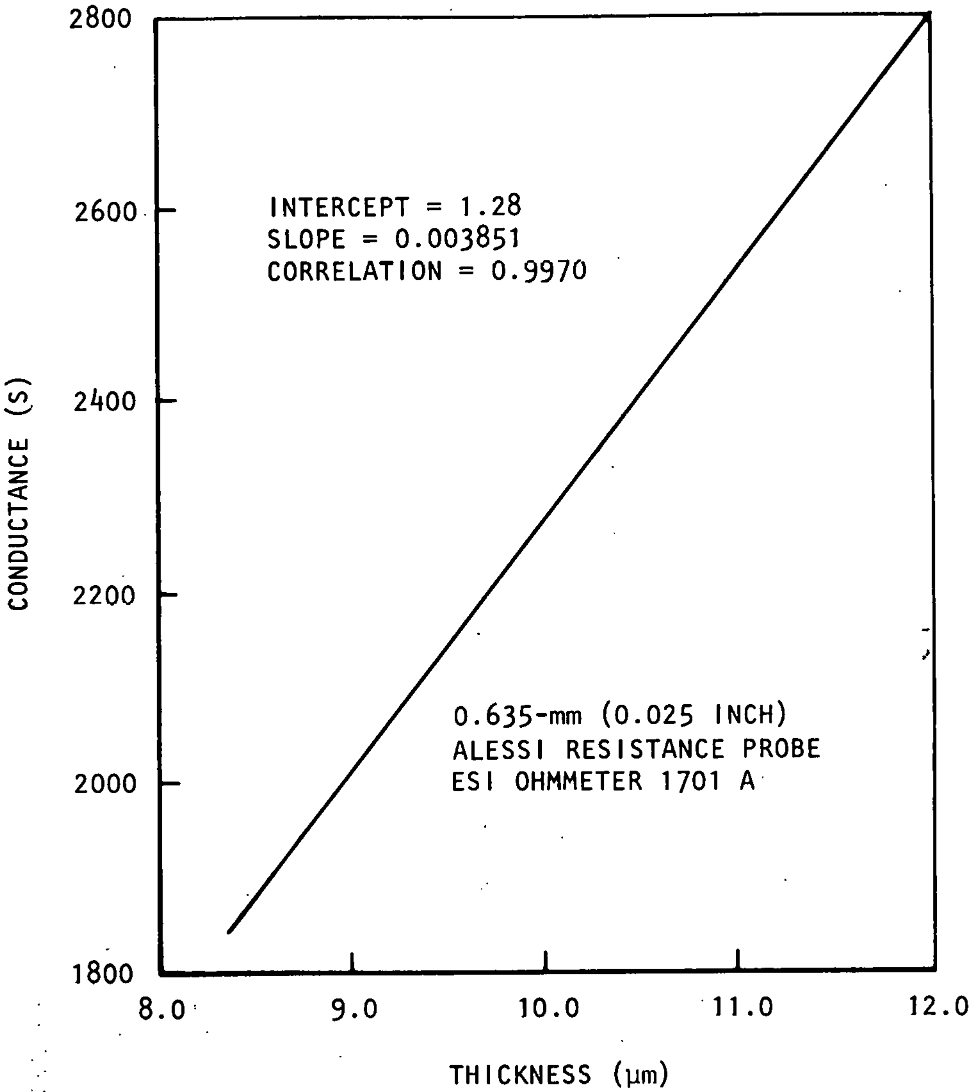

Figure 8. Four-Point Resistance Least-Squares Calibration Curve for Physical-Vapor-Deposited Copper on Kapton

An Auger-Electron-Spectrometer examination of the rough surface indicated the presence of oxygen, carbon, and chlorine (all common surface elements). The surface then was sputtered with argon to remove approximately 20 nm of material, and the analysis was repeated. The second analysis revealed the presence of only copper, thus indicating a very pure deposit.

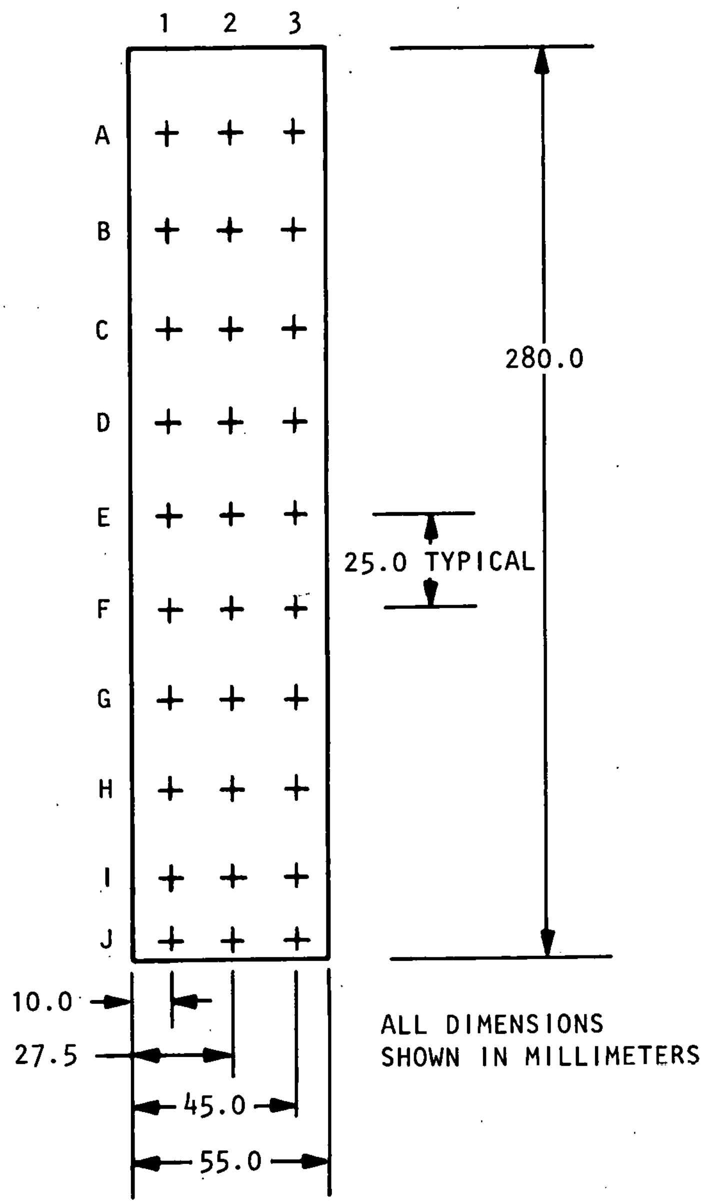

Figure 9. Probe-Location Coordinates on Kapton Strips Used for Plating-Thickness-Uniformity Determination

ACCOMPLISHMENTS

Reference thickness standards of copper on Kapton and aluminum on Kapton have been fabricated and put into service. These reference standards were used to calibrate working standards which, in

Table 4. Thickness Uniformity of Aluminum-On-Kapton Deposits

Part	Vertical Coordinate*	Thickness for Horizontal Coordinate Indicated* 1 (μm)	2 (μm)	3 (μm)
1**	A	9.42	9.60	9.47
	B	9.65	9.73	9.68
	C	9.86	9.93	9.93
	D	9.98	9.98	9.98
	E	10.01	10.08	10.01
	F	9.98	10.08	9.98
	G	9.93	9.91	9.75
	H	9.53	9.60	9.50
2***	A	9.35	8.99	8.74
	B	9.73	9.73	9.73
	C	9.86	9.91	9.83
	D	9.91	9.98	9.91
	E	9.88	9.93	9.88
	F	9.93	9.93	9.88
	G	9.70	9.75	9.73
	H	9.50	9.58	9.60

*Refer to Figure 9 for measurement locations.
**$\bar{x}$ = 9.82; σ = 0.21. Values for vertical coordinates A and H were eliminated from the calculations since they were outside the useful area of the part: $\bar{x}$ = 9.91; σ = 0.13.
***$\bar{x}$ = 9.71; σ = 0.30. Values for vertical coordinates A and H were eliminated from the calculations since they were outside the useful area of the part: $\bar{x}$ = 9.84; σ = 0.09.

turn, are used to support product inspection. Two sets of working standards of each material have been sent to LASL, and they periodically will be returned to Bendix Kansas City for calibration in accordance with a recall schedule specified by Bendix calibration personnel.

Table 5. Thickness Uniformity of Copper-On-Kapton Deposits

Part	Vertical Coordinate*	Thickness for Horizontal Coordinate Indicated* 1 (μm)	2 (μm)	3 (μm)
2**	A	9.80	9.86	9.92
	B	9.82	9.92	9.90
	C	9.92	10.00	9.88
	D	9.92	10.05	10.00
	E	9.96	10.10	10.02
	F	9.92	10.02	10.02
	G	9.84	10.00	9.96
	H	9.92	9.96	9.96
	I	9.82	9.90	9.88
	J	9.77	9.86	9.80
10***	A	8.81	8.85	8.87
	B	8.88	8.96	8.99
	C	8.93	8.81	8.96
	D	8.90	9.05	8.93
	E	8.91	8.94	8.97
	F	8.90	8.91	8.96
	G	8.85	8.87	8.93
	H	8.84	8.85	8.81
	I	8.90	8.87	8.84
	J	8.88	8.91	8.87

*Refer to Figure 9 for measurement locations.
**$\bar{x}$ = 9.92; σ = 0.08.
***$\bar{x}$ = 8.90; σ = 0.06.

Table 6. Density of Physical-Vapor-Deposited Copper and Aluminum

Metal	Optical Flat	Density (g/cm^3)
Copper*	4	9.18
	19	9.01
	25	9.02
Aluminum**	46	2.63
	17	2.57

*Estimated uncertainty does not exceed ±3 percent.
**Values obtained from D. M. Sherman, 2/16/77; corrections for surface roughness have not been applied; upon correction, a value of 2.71 g/cm^3 can be anticipated.

FUTURE WORK

The Bendix Metrology Laboratory will maintain the reference thickness standards of aluminum on Kapton and copper on Kapton. These standards will be used to calibrate working standards. A calibration history of each set of working standards will be kept on file and updated upon each calibration recall.

Refinement of both the calibration and data-acquisition processes has been planned. A comparison between flat physical-vapor-deposited aluminum standards and curved 0.15-percent-copper-doped aluminum is under way. This should determine whether additional physical-vapor-deposited aluminum standards should be fabricated.

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[3]Richard Sard and others, editors, *Properties of Electrodeposits; Their Measurement and Significance*. Princeton, New Jersey: The Electrochemical Society, 1975 (Library of Congress No. 74-84702).

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SPECIAL PROJECTS: Nondestructive Testing

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