27
11-04-77
256 UT 15

MASTER

BDX-613-1634 (Rev.)

DEVELOPMENT OF PLATING THICKNESS STANDARDS
FOR THE PHYSICAL VAPOR DEPOSITION OF
ALUMINUM ON KAPTON

PDO 6985015, Milestone Report

R. J. Russell, Project Leader

Project Team:

G. J. Hale

J. W. Hill

G. A. McWhirt

Published September 1977

Prepared for the United States Energy Research and Development
Administration Under Contract Number EY-76-C-04-0613 USERDA

**Kansas City
Division**

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

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 \$2.25
Paper Copy \$4.~~00~~
50

DEVELOPMENT OF PLATING THICKNESS STANDARDS
FOR THE PHYSICAL VAPOR DEPOSITION OF
ALUMINUM ON KAPTON

Published September 1977

Project Leader:
R. J. Russell
Department 142

Project Team:
G. J. Hale
J. W. Hill
G. A. McWhirt

PDO 6985015
Milestone Report

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Communications Services

**Kansas City
Division**

DEVELOPMENT OF PLATING THICKNESS STANDARDS FOR THE PHYSICAL VAPOR
DEPOSITION OF ALUMINUM ON KAPTON

BDX-613-1634, UNCLASSIFIED Milestone Report, Published
September 1977

Prepared by R. J. Russell, D/142, under PDO 6985015

Reference and working standards for determining nondestructively the thickness of aluminum-on-Kapton plating, unobtainable either commercially or from the National Bureau of Standards, have been developed at Bendix Kansas City. The results from the calibration data of the reference standards indicate that thickness values are within ± 152.4 nm (± 6 microinches).

WPC-bap

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

The Bendix Corporation
Kansas City Division
P.O. Box 1159
Kansas City, Missouri 64141

A prime contractor with the United States
Energy Research and Development
Administration under Contract
Number EY-76-C-04-0613 USERDA

CONTENTS

Section	Page
SUMMARY.	6
DISCUSSION	7
SCOPE AND PURPOSE.	7
ACTIVITY	7
<u>Background</u>	7
<u>Fabrication Process for Standards.</u>	7
<u>Thickness of Reference Standards</u>	9
<u>Thickness of Working Standards</u>	13
<u>Characteristics of PVD Aluminum.</u>	15
<u>Analysis of Calibration Data</u>	19
ACCOMPLISHMENTS.	28
FUTURE WORK.	29
REFERENCES	30
APPENDIX. VACUUM-DEPOSITION PROCEDURE	31
DISTRIBUTION	32

ILLUSTRATIONS

Figure		Page
1	Optical Flats Before Aluminum Deposition and After Deposition (P-93854)	9
2	Deformation Correction Graph for Link Interferometer Using 6.35-mm-Diameter (0.25 Inch) Diamond Stylus for Measuring Aluminum Thickness	14
3	Aluminum-On-Kapton Reference Standards (P-93855).	15
4	Beta Backscatter Calibration for Aluminum Thickness on Kapton, Using Least-Squares Method	17
5	Beta Backscatter Calibration for Aluminum Thickness on Kapton, Using Count-Ratio Method	18
6	Polar Inspection Fixture	21
7	Placement of Kapton Laminated Buttons and Optical Flats in the Vapor-Deposition Fixture.	24
8	Profile Trace of PVD Aluminum Deposited on an Optical Flat	25
9	SEM Photograph Showing an Imperfection in an Aluminum Deposit.	26
10	Typical Set of Data Obtained From Bendix Reference Standards.	27

TABLES

Number		Page
1	Percentage of Copper Concentration Determined Through the Use of an Atomic Absorption Spectrophotometer	10
2	Summary of Aluminum Thickness Standards Data	11
3	Reference Thickness Standards.	13
4	Working Standard Data.	16
5	Working Thickness Standards.	19
6	Density Determination of PVD Aluminum.	20
7	Aluminum-Thickness-Uniformity Data From Three Laminates.	22
8	Pole-To-Skirt Variation in Aluminum Thickness of Three Laminates	23

SUMMARY

Because aluminum-on-Kapton plating-thickness standards are not available from the National Bureau of Standards or any known commercial source, these standards have been developed at the Bendix Kansas City Division. Weight-gain, interferometry, and beta-backscatter methods were used in the development. A set of 13 reference standards was verified by interferometry, certified, and put into service for certifying working standards which are used in the inspection of Bendix products.

All working standards are inspected periodically for damage and compared to a set of reference standards. By this technique, each working standard is either recertified and returned to service or replaced with a certified standard.

The effectiveness of the reference standards was evaluated through a series of statistical tests. These tests were performed on each set of calibration data, and they provided an effective means for determining the merit of the data. From the test results, 90 percent of the count-per-second data points projected to the aluminum-on-Kapton thickness were found to be within ± 152.4 nm (± 6 microinches). This statement did not include variations that might have existed in the acquisition of the counts-per-second data on the working standards or any bias that might have existed in the set of reference standards.

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 the Bendix Kansas City Division. When available, working standards will be purchased from a commercial source.

Weight-gain and interferometry were the techniques chosen to determine the plating thickness of the Bendix reference standards. Both methods provide direct traceability to the National Bureau of Standards and a reliable estimate of the measurement uncertainty. Through the use of beta backscatter or some other nondestructive measuring technique, transfer and working standards will be assigned thickness values by comparison to the reference standards. The working standards will improve test reliability and accuracy, and they will ensure proper traceability.

ACTIVITY

Background

Beta backscatter equipment has been used at Bendix Kansas City for measuring the thickness of aluminum on Kapton substrates for some time. In the past, thin calibrated aluminum foils were placed on a Kapton substrate for use as thickness reference units (standards). These standards, while quite useful, did not exactly simulate the parts that were to be measured. Such variables as density, alloy composition, shape, texture, and adhesion needed to be simulated since they were believed to affect the beta-backscatter count rate.

This report describes the development of a set of reference standards which simulate the production part as closely as possible.

Fabrication Process for Standards

In the development of standards, the fabrication process must be capable of being reproduced at a later time. The following techniques were used to maximize the accuracy of the thickness values and to optimize the reproducibility of the results.

Reference standards were fabricated by vacuum-depositing aluminum on 19-mm-diameter Kapton substrates which were mounted in an aluminum housing. The assembly was called a "button." The thickness of the aluminum on the standards was determined by the following described process.

Three buttons and three optical parallels (or optical flats) were mounted in a deposition fixture. Quartz optical parallels were chosen for this application because their thickness, parallelism, and weight remained constant throughout the fabrication process. The thickness of the aluminum deposits was determined by measuring the thickness of the quartz parallels before and after deposition (Figure 1). The difference in the two measurements was the thickness value assigned to the aluminum.

The deposition fixture, with the optical parallels and buttons, was loaded along with two large substrates (used for making working standards) into a motor-powered planetary system located inside the vacuum chamber. The procedure described in the Appendix was followed, and the aluminum was deposited by rf sputtering.

The process of depositing the aluminum was controlled through the use of a quartz microbalance which was located inside the chamber. When the aluminum had reached the desired thickness, the system was shut down and allowed to cool.

The parallels, buttons, laminates, and a test panel were removed from the chamber and measured. The material composition of the test panel was determined through the use of an atomic absorption spectrophotometer. The results obtained are shown in Table 1.

The thickness of the aluminum on the optical parallels was measured with a Link Gage Block Interferometer; in addition, the deposited aluminum was stripped from one button and its thickness also was measured with the interferometer. This data is shown in Table 2. The excellent agreement in the measurements of film thickness added confidence to the assumption that the remaining two buttons had the same thickness of aluminum as that which had been deposited on the measured flats. The process must be repeated for each additional thickness desired.

The remaining two buttons from each run were put in a set which was designated *reference standards*. Table 3 identifies the reference standards that were fabricated under this process.

Small sections of the aluminum-coated Kapton laminate which had been fabricated with the standards were punched out through the use of a die and were mounted in a fixture. They will be used as reference units (working standards) for beta-backscatter inspection. Using beta-backscatter techniques, the thickness of the

Figure 1. Optical Flats Before Aluminum Deposition (Above) and After Deposition (Below)

working standards is determined by comparison with the reference standards.

A periodic calibration schedule is used to assure the integrity of the reference units. If these working standards should become worn or damaged, they will be replaced with new sections. When the supply of working standards becomes exhausted, the described process must be duplicated to obtain additional reference units.

Thickness of Reference Standards

The thickness of the aluminum deposit on the optical parallels was determined with a Link Gage Block Interferometer by measuring the thickness before and after the deposition of the aluminum. The interferometer has a resolution of 2.54 nm (0.1 microinch) and an estimated measurement uncertainty of less than 50 nm (2 microinches).

Table 1. Percentage of Copper Concentration Determined Through the Use of an Atomic Absorption Spectrophotometer

Identification of Run	Copper Concentration (Percent)
1	0.12
2	0.22
4	0.14
5	0.12
6	0.13
7	0.11
8	0.15
9	0.12
10	0.22

Because the interferometer has the disadvantage of being a contact-type instrument, corrections for stylus deformation are required. Figure 2 shows the correction curves that were developed. These curves were generated by varying the stylus force and measuring the resulting film thickness. The changes in the film thickness versus the stylus force were plotted, and the curve was forced through zero at zero force to produce the correction graph.

To measure the thickness of the aluminum on the plated optical flats, the deformation of the aluminum on a quartz substrate had to be determined. In addition, the deformation of aluminum on the interferometer anvil was determined to permit the measurement of the stripped foil. By proper application of the correction data, measurements within ± 75 nm (± 3 microinches) should be possible.

Table 2 provides comparative data between the aluminum thickness on the optical parallels and the thickness of the stripped foil. The worst-case disagreement was found to be 178 nm (7 microinches); a more typical result was 50 nm (2 microinches). Thus the results shown in Table 2 add confidence to the assumption

Table 2. Summary of Aluminum Thickness Standards Data

Identification of Run	Designation of Optical Flat	Designation of Stripped Button*	Thickness	
			(Microinches)	(μm)
1	1		440	11.18
1	3		441	11.20
2	5		395	10.03
2	6		393	9.98
2	7		394	10.01
2		6	392	9.96
4	13		495	12.57
4	14		496	12.60
4	16		493	12.52
4		16	489	12.42
5	5		497	12.62
5	6		496	12.60
5	9		496	12.60
5		6-3	495	12.57
6	2		(Damaged)	(Damaged)
6	10		397	10.08
6	12		397	10.08
6		14	396	10.06
7	17		527	13.39
7	18		526	13.36

Table 2 Continued. Summary of Aluminum Thickness Standards Data

Identification of Run	Designation of Optical Flat	Designation of Stripped Button*	Thickness	
			(Microinches)	(μm)
7	19		528	13.41
7		18	528	13.41
9	25		457	11.61
9	26		459	11.66
9	27		457	11.61
10	28		450	11.43
10	29		450	11.43
10	30		451	11.46
11	4		358	9.09
11	8		361	9.17
11	31		365	9.27

*Prepared for stripping.

Table 3. Reference Thickness Standards

Reference Standard Designation	Certified Thickness	
	(Microinches)	(μm)
1	440	11.18
2	440	11.18
3	440	11.18
4	394	10.01
5	394	10.01
7	495	12.57
12	495	12.57
13	496	12.60
15	397	10.08
17	397	10.08
20	527	13.39
25	457	11.61
26	457	11.61
27	457	11.61
28	450	11.43
29	450	11.43
30	450	11.43

that the remaining two buttons have the same thickness of aluminum deposited on them as that on the measured optical flats. Both buttons were assigned the thickness that was measured on the optical flats and were put in service as reference standards. Figure 3 shows a set of reference standards that are now in service.

Thickness of Working Standards

The reference standards previously described (Figure 3) were used to calibrate three sets of working standards. The comparison was performed through the use of beta-backscatter thickness-measuring equipment. A least-squares line (counts per second versus thickness) was drawn through the data obtained from the reference standards.

Figure 2. Deformation Correction Graph for Link Interferometer Using 6.35-mm-Diameter (0.25 Inch) Diamond Stylus for Measuring Aluminum Thickness

The thickness of the aluminum on each working standard was calculated from the average counts-per-second data and the equation of the least-squares line of the reference standards. A typical data set is shown in Table 4, and a graphic representation of the results of the least-squares line is shown in Figure 4. All data was collected by recording the time required to accumulate 10^6 counts; this method should assure the same statistical accuracy for each result.

The following ratio was considered to be more meaningful when plotted against thickness than the previously described method because it would standardize the data and assist in system-to-system correlation (Figure 5):

$$\text{RATIO} = \frac{C_T - C_A}{C_\infty - C_A}; \quad (1)$$

where

Figure 3. Aluminum-On-Kapton Reference Standards

C_T = counts per second from standards;

C_A = counts per second from air; and

C_∞ = counts per second from an infinite thickness of aluminum.

This method should compensate for any differences in source activity or background, and additional experience should prove its merit. Table 5 identifies working standards that have been fabricated.

Characteristics of PVD Aluminum

The following described tests were performed to define the material characteristics of the aluminum which was deposited on the standards.

Table 4. Working Standard Data

Working Standard Designation	Counts Per Second*	Computed Thickness	
		(Microinches)	(μm)
AL-6	2726.3	389	9.88
AL-7	2789.4	420	10.67
AL-8	2822.5	436	11.07
AL-9	2852.3	451	11.46
AL-10	2921.4	486	12.34

*Average of three runs (10^6 counts per run);
 Thickness (microinches) = 0.49684 (counts per
 second) - 965.9; Refer to Figure 2.

Copper Concentration

A sheet of Kapton was placed in the vacuum-deposition chamber and coated with aluminum during each standards' run. The deposited material was analyzed for the concentration of copper and other extraneous impurities. Table 1 shows the results of several analyses for copper that were made using an atomic absorption spectrophotometer.

Density

The density of the raw material that was loaded into the crucible before deposition was found to be $2.71 \pm 0.01 \text{ g/cm}^3$. The density of the PVD aluminum was determined by measuring the weight and volume of the deposit. An optical parallel was weighed before and after deposition of the aluminum. The weight was determined through the use of a Mettler Model M5 microbalance. This equipment has a precision of $3 \mu\text{g}$ and an estimated uncertainty of less than $20 \mu\text{g}$.

Area was determined through the use of a SIP Trioptic Measuring Machine. The limiting factor of this measurement was the edge definition of the plated area. The estimated uncertainty in the area measurement was $\pm 0.8 \text{ mm}^2$.

Figure 4. Beta Backscatter Calibration for Aluminum Thickness on Kapton, Using Least-Squares Method

The thickness of the deposit was measured with a Link Gage Block Interferometer. This measurement had an estimated uncertainty of less than $0.13 \mu\text{m}$.

Table 6 lists several independent attempts that were made to determine the density. The average density of the aluminum deposit was found to be 2.58 g/cm^3 . The uncertainty of the density value was determined to be $\pm 0.05 \text{ g/cm}^3$. (The error due to surface topography was not taken into consideration.)

Figure 5. Beta Backscatter Calibration for Aluminum Thickness on Kapton, Using Count-Ratio Method

Uniformity

The small area of aluminum deposited on each standard does not lend itself to an investigation of the uniformity of the aluminum thickness. An investigation of three large laminates on which aluminum had been vacuum-deposited in separate deposition runs therefore was conducted to provide the required data with which

Table 5. Working Thickness Standards

Working Standard Designation	Certified Thickness	
	(Microinches)	(μm)
AL-6	389	9.88
AL-7	420	10.67
AL-8	436	11.07
AL-9	451	11.46
AL-10	486	12.34

to determine the possible aluminum-thickness gradient. The data were collected through the use of beta-backscatter equipment positioned by a polar inspection fixture (Figure 6) and are shown in Table 7. As indicated by the table, a 2σ -aluminum-thickness variation from 0.2 to 0.25 μm (8 to 10 microinches) was found.

Table 8 shows that the largest aluminum-thickness variation was found between the pole and the outer skirt of the laminate. A distance of approximately 100 mm (4 inches) separated the pole and skirt locations and resulted in a gradient of approximately 50 nm (2 microinches) for each 25.4 mm (1 inch). The 19-mm-diameter (0.75 inch) standards were placed in the deposition fixture as shown in Figure 7 with all buttons and optical flats equidistant from the pole. Thus the aluminum deposited on a standard can be expected to be very uniform with an expected variation (the average thickness value determined with a beta-backscatter probe having an aperture diameter of 3.2 mm or 0.125 inch) not exceeding 50 nm (2 microinches).

Figure 8 shows a profile trace of the aluminum deposit using a Clevite 1200 surface analyzer with a 12.7- μm -radius (500-microinch) stylus. The trace shows a typical 100-nm (4-microinch) peak-to-valley variation with occasional large imperfections. Figure 9 is a scanning-electron-microscope (SEM) photograph which identifies an imperfection.

Analysis of Calibration Data

The calibration curve that is used for determining the aluminum thickness on reference units is of the following form.

$$t = BR + C, \quad (2)$$

Table 6. Density Determination of PVD Aluminum

Optical Flat	Optical Flat Weight (g)	PVD Aluminum Volume* (cm ³)	Weight Gain** (g)	Density*** (g/cm ³)
6	7.177344	0.003604	0.009258	2.57
11	7.407876	0.003619	0.009437	2.61
13	7.407634	0.003591	0.009256	2.58
14	7.500950	0.003599	0.009270	2.58

*Typical Area = 2.8561 cm², Uncertainty 0.008 cm²;
 Typical Thickness = 0.00126 cm, Uncertainty 0.000013 cm.

**Typical Weight Gain = 0.009256 g, Uncertainty 0.00006 g.

***Density = 2.58 ± 0.05 g/cm³. Error due to surface topography was not taken into consideration.

where

t = thickness,

B = slope,

R = count rate, and

C = intercept.

The least-squares method of linear regression has provided excellent results within the desired thickness range. The following four formulas are used for this operation.

$$\bar{t} = \frac{1}{n} \sum_{I=1}^n t_I, \quad (3)$$

$$\bar{R} = \frac{1}{n} \sum_{I=1}^n R_I, \quad (4)$$

Figure 6. Polar Inspection Fixture

$$\hat{t} = BR_I + C, \text{ and} \quad (5)$$

$$B = \frac{\sum_{I=1}^n (R_I - \bar{R})(t_I - \bar{t})}{\sum_{I=1}^n (R_I - \bar{R})^2}, \quad (6)$$

where

Table 7. Aluminum-Thickness-Uniformity Data From Three Laminates

Location of Aluminum Deposit* (Degrees)		Thickness Values of Deposit**					
		Laminate 7		Laminate 9		Laminate 10	
θ	ϕ	(Micro- inches)	(μm)	(Micro- inches)	(μm)	(Micro- inches)	(μm)
0	330	488	12.40	437	11.10	429	10.90
0	340	493	12.52	442	11.23	433	11.00
0	350	498	12.65	445	11.30	438	11.13
90	330	492	12.50	440	11.18	425	10.80
90	340	497	12.62	443	11.25	429	10.90
90	350	500	12.70	447	11.35	436	11.07
180	330	492	12.50	438	11.13	422	10.72
180	340	496	12.60	445	11.30	433	11.00
180	350	500	12.70	445	11.30	435	11.05
270	330	488	12.40	436	11.07	432	10.97
270	340	488	12.40	444	11.28	434	11.02
270	350	494	12.55	444	11.28	431	10.95
0	0	498	12.65	448	11.38	439	11.15
	$\bar{x}$	494	12.55	442	11.24	432	10.97
	σ	4.4	0.11	3.8	0.10	4.9	0.12

*Refer to Figure 6 for definition of angles.

**The thickness values are referenced to a set of 0.999-percent-pure aluminum foils placed on a Kapton substrate; the data are obtained from the time, in seconds, required to accumulate 10^6 counts.

Table 8. Pole-To-Skirt Variation in Aluminum Thickness of Three Laminates

Laminate Designation	θ (Degrees)	Thickness Value of Aluminum Deposit at Location ϕ (in Degrees) Indicated*			
		330 (μm)	340 (μm)	350 (μm)	Pole (μm)
7	0	12.40	12.52	12.65	
	90	12.50	12.62	12.70	12.65
	180	12.50	12.60	12.70	
	270	12.40	12.40	12.55	
9	0	11.10	11.23	11.30	
	90	11.18	11.25	11.35	11.38
	180	11.13	11.30	11.30	
	270	11.07	11.28	11.28	
10	0	10.90	11.00	11.13	
	90	10.80	10.90	11.07	11.15
	180	10.72	11.00	11.05	
	270	10.97	11.02	10.95	

*Data are obtained from the time, in seconds, required to accumulate 10^6 counts.

n = number of data points, and

$\hat{t}_I$ = calculated values of thickness from the least-squares regression.

Equations 7 through 10 were used to compare the linearity and precision of the various calibration runs.¹

$$\text{Correlation} = \sqrt{\frac{\sum_{I=1}^n (\hat{t}_I - \bar{t})^2}{\sum_{I=1}^n (t_I - \bar{t})^2}}, \quad (7)$$

Figure 7. Placement of Kapton Laminated Buttons and Optical Flats in the Vapor-Deposition Fixture

$$\text{Mean Square Error} = \sum_{I=1}^n \frac{(t_I - \bar{t}_I)^2}{n-2}, \quad (8)$$

$$\text{Standard Error} = \sqrt{\text{Mean Square Error}}, \text{ and} \quad (9)$$

$$\text{Coefficient of Variation} = \frac{\text{Standard Error}}{\bar{t}}. \quad (10)$$

The coefficient of variation is useful for data sets having different mean values, whereas the standard error is more suitable for data sets having similar baselines.

Figure 8. Profile Trace of PVD Aluminum Deposited on an Optical Flat

The run test, identified as *NR* in Figure 10, involves counting the number of times an exact-fit line crosses the least-squares straight line. This information can be combined with a table of values to determine, for a given confidence interval, whether the data show a nonlinear trend².

Another measure of the merit of the calibration consists of calculating tolerance bands about the slope and intercept values. The following three formulas are used³.

$$\text{Slope Tolerance} = T \frac{\sqrt{\frac{\sum_{I=1}^n (t_I - \hat{t}_I)^2}{n-2}}}{\sqrt{\sum_{I=1}^n (R_I - \bar{R})^2}}, \quad (11)$$

Figure 9. SEM Photograph Showing an Imperfection in an Aluminum Deposit (Original Magnification 3000X)

$$\text{Intercept Tolerance} = T \sqrt{\frac{\sum_{I=1}^n (t_I - \bar{t}_I)^2}{n-2}} \sqrt{\frac{1}{n} + \frac{\bar{R}^2}{\sum_{I=1}^n (R_I - \bar{R})^2}}, \quad (12)$$

and

$$\text{Normalized Intercept Tolerance} = \frac{\text{Intercept Tolerance}}{\bar{t}}, \quad (13)$$

where

T = student T value for a confidence interval of $\alpha/2$ and $n-2$ degrees-of-freedom.

RUN LINEAR			
ENTER X Y PAIRS			
2823.3	440.		
2741.2	394.		
2940.3	495.		
2853.9	450.		
2836.9	440.		
2829.7	440.		
2945.5	496.		
2738.2	397.		
2853.1	450.		
2844.1	450.		
2740.5	397.		
2740.5	394.		
2935.1	495.		
MEAN SQUARE ERROR= 0.56157055E 01			
STANDARD ERROR= 0.23697481E 01			
COEFFICIENT OF VARIATION= 0.0054			
CORRELATION= 0.998222			
FOR A 90 PERCENT CONFIDENCE INTERVAL			
SLOPE= 0.49684075 +/- 0.01606245			
NORMALIZED INTERCEPT TOLERANCE= 0.1031			
INTERCEPT= -965.908936 +/- 45.496643			
RUN TEST RESULTS: N=13 NR= 9			
XB= 2832.4844 YB= 441.3846 T= 1.7959 F= 1.9730			
90% OF THE DATA POINTS FALL WITHIN THE LISTED TOLERANCE 90% OF THE TIME			
X= 2758.9299	Y= 404.8398+/-	5.8106	
X= 2779.6599	Y= 415.1393+/-	5.7164	
X= 2800.3899	Y= 425.4388+/-	5.6494	
X= 2821.1199	Y= 435.7383+/-	5.6139	
X= 2841.8501	Y= 446.0380+/-	5.6123	
X= 2862.5801	Y= 456.3374+/-	5.6446	
X= 2883.3101	Y= 466.6370+/-	5.7087	
X= 2904.0400	Y= 476.9365+/-	5.8005	
X= 2924.7700	Y= 487.2360+/-	5.9137	
X= 2945.5000	Y= 497.5355+/-	6.0395	
\$			

Figure 10. Typical Set of Data Obtained From Bendix Reference Standards

The normalized intercept tolerance is useful for comparing the results obtained.

A more useful measure of the merit of a calibration consists of calculating the tolerance intervals around the calculated values of thickness ($\hat{t}_I$), as determined from the linear-regression equation. The relationships are expressed by the following equations³.

$$t_I = \hat{t}_I \pm K(\text{Standard Error}), \quad (14)$$

$$K = Z_A \left[1 + \frac{2}{N'} - \frac{2Z_A^{-3}}{24(N')^2} \right] \left[\sqrt{F_{1-\gamma}(\infty, n-2)} \right] \text{ and} \quad (15)$$

$$N' = \frac{1}{\frac{1}{N} + \frac{(R_I - \bar{R})^2}{S_R^2(n-1)}}, \quad (16)$$

where

Z_A = a value from the Z distribution corresponding to the probability (P);

$A = (1+P)/2$, with P denoting the percentage of the population covered;

$F_{1-\gamma}$ = a value from the F distribution with γ denoting the percentage of the points falling within the population; and

S_R = the standard deviation corresponding to $n-1$ degrees-of-freedom.

A typical set of data obtained from the Bendix reference standards is shown in Figure 10; at the bottom of the page are shown the tolerance intervals for selected thicknesses. This test provides a quick and effective method of evaluating the data collected during a calibration. It should identify such contributive factors as wear, defects, or poor technique so that corrective action can be taken when necessary.

ACCOMPLISHMENTS

A set of aluminum-on-Kapton reference standards has been fabricated and put into service. This set was used to calibrate

several sets of working standards that now are being used to support the inspection of products.

FUTURE WORK

The Bendix Metrology Laboratory will maintain the aluminum-on-Kapton reference standards from which working standards will be calibrated and scheduled. A calibration history of each set of working standards will be kept on file.

Refinement of both the calibration and data-acquisition processes has been planned.

REFERENCES

¹Murray R. Spiegel, *Statistics*. New York: McGraw-Hill, 1964.

²Julius S. Bendat and Allan G. Piersal, *Random Data: Analysis and Measurement Procedures*. New York: Wiley-Interscience, 1971.

³W. J. Dickson and F. J. Massey, *Introduction to Statistical Analysis*. New York: McGraw-Hill, 1957.

Appendix

VACUUM-DEPOSITION PROCEDURE

- Preclean and vacuum-bake parts at 200°C for 1 hour.
- Rinse parts with ethanol only, and bake at 150°C for a minimum of 2 hours.
- Place parts in physical-vapor-deposition (PVD) chamber and pump the chamber for approximately 1 to 1-1/2 hours to achieve a pressure of less than 1.2 mPa (9×10^{-6} torr).
- Preheat rf-induced crucible for 5 minutes at low power.
- Superheat crucible at high power for 4 minutes.
- Reduce power and open the shutter to plate at a rate of approximately 75 Å/s (750 nm/s) until the indicated thickness is at the target value.
- Close the shutter at the target-value thickness.
- Shut off the power.
- Cool the system for a minimum of 30 minutes before opening the vacuum chamber.
- Open the chamber and remove the parts.
- Submit the parts for thickness measurement.

DISTRIBUTION

	Copy
Robert Bulcock, ERDA-KCAO, 1D49	1
D. F. Wright, LLL	2
A. W. Reger, SLA	3
R. L. Calkins, D/100, 2A30	4
C. L. Walters, D/141, 2B30	5
W. B. Abel, D/142, BQ30	6
J. S. Carson, D/142, BQ30	7
J. F. Hadley, D/142, BN30	8
J. W. Hill, D/142, BQ30	9
G. A. McWhirt, D/142, BN30	10
R. J. Schofield, D/144, FE49	11
J. D. Corey, D/554, BD50	12-13
L. Stratton, D/554, 2C44	14-16
R. P. Frohmberg, D/800, 2A39	17
J. W. Chesney, D/812, 2D31	18
J. C. Culver, D/812, 2D31	19
W. J. Nicholson, D/812, 2D31	20
G. J. Hale, D/814, 2C43	21
D. M. Sherman, D/814, 2C43	22
R. E. Kessler, D/865, 2C40	23-24

BDX-613-1634

PDO 6985015

DEVELOPMENT OF PLATING THICKNESS STANDARDS
FOR THE PHYSICAL VAPOR DEPOSITION OF ALUMINUM
ON KAPTON, R. J. Russell, D/142, UNCLAS
Milestone, September 1977.

Reference and working standards for determining nondestructively the thickness of aluminum-on-Kapton plating, unobtainable either commercially or from the National Bureau of Standards, have been developed at Bendix Kansas City. Test results from the calibration data of the reference standards indicate that thickness values are within ± 152.4 nm (± 6 microinches).

SPECIAL PROJECTS: Nondestructive Testing

DEVELOPMENT OF PLATING THICKNESS STANDARDS FOR
THE PHYSICAL VAPOR DEPOSITION OF ALUMINUM ON
KAPTON, R. J. Russell, D/142, UNCLAS Milestone,
PDO 6985015, BDX-613-1634, September 1977.

Reference and working standards for determining nondestructively the thickness of aluminum-on-Kapton plating, unobtainable either commercially or from the National Bureau of Standards, have been developed at Bendix Kansas City. Test results from the calibration data of the reference standards indicate that thickness values are within ± 152.4 nm (± 6 microinches).

DEVELOPMENT OF PLATING THICKNESS STANDARDS FOR
THE PHYSICAL VAPOR DEPOSITION OF ALUMINUM ON
KAPTON, R. J. Russell, D/142, UNCLAS Milestone,
PDO 6985015, BDX-613-1634, September 1977.

Reference and working standards for determining nondestructively the thickness of aluminum-on-Kapton plating, unobtainable either commercially or from the National Bureau of Standards, have been developed at Bendix Kansas City. Test results from the calibration data of the reference standards indicate that thickness values are within ± 152.4 nm (± 6 microinches).