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DEPOSITION OF ALUMINUM-COPPER ALLOY ON
LAMINATED POLYIMIDE SUBSTRATES FROM AN
RF INDUCTION SOURCE

By G. J. Hale

Published November 1978

Topical Report

Prepared for the United States Department of Energy
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**Kansas City
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Topical Report
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DEPOSITION OF ALUMINUM-COPPER ALLOY ON LAMINATED POLYIMIDE
SUBSTRATES FROM AN RF INDUCTION SOURCE

BDX-613-1993 (Rev.), Topical Report, Published November 1978

Prepared by G. J. Hale, D/814

A physical vapor deposition process with an rf induction vapor source has been developed to deposit 11.2- μ m aluminum-copper alloy on polyimide laminate substrates. The total thickness values can be repeated within a ± 0.5 - μ m range with a 90-percent yield. The average thickness uniformity (standard deviation divided by average thickness) of the deposit acquired by beta backscatter for 100 substrates was 1.2 percent. The aluminum-0.3 weight percent copper vapor charge results in a copper content in the deposit of approximately 0.12 weight percent. The copper shows no preferential deposition pattern, as related to the surface of the laminate and its dispersion throughout the deposit. The resistivity of the aluminum-copper deposits is 3 $\mu\Omega$ -cm and is stable from one deposition run to another. Also, the resistivity did not appear to be related to the geometry of the domed substrate. The density of the 11.2- μ m-thick aluminum-copper film was within 1 percent of theoretical, and its adhesion to the polyimide exceeds that required for postdeposition operations and handling. Physical vapor-deposited aluminum-copper films for beta backscatter thickness standards were provided and were instrumental in achieving the reported uniformity values and determining the beta backscatter measurement uncertainty.

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SUMMARY

A physical vapor deposition (PVD) process, using a radio frequency (rf) induction source, has been developed for routinely producing 11.2- μm aluminum-0.12 weight percent copper alloy deposits on polyimide laminates. The normal operating vacuum during deposition is 2.6 mPa (2×10^{-5} torr), and the optimized deposition rate is 5 nm/s. The resulting substrate temperature for the 5-nm/s rate is 190°C.

Residual gas analysis (RGA) during deposition shows the normal hydrogen, oxygen, and water but an increased carbon-monoxide and nitrogen (28 amu) contribution as a result of outgassing of the composite BN-TiB₂ crucible.

The uniformity of the aluminum-copper films at the 11.2- μm thickness is 1.2 percent. When given a ± 0.5 - μm tolerance on the target thickness, a repeatability of 90 percent is consistently achieved.

Thickness standards for the aluminum-copper alloy on polyimide were developed to provide baseline thickness and thickness uniformity data. In addition, standards for pure copper were completed for future applications.

Some defects observed in the deposited film appear inherent in the PVD process. However, with appropriate substrate preparation before deposition it is possible to reduce noticeably the quantity of these defects. The vapor source material is an aluminum-0.3 weight percent copper alloy. The resulting deposit contains approximately 0.12 weight percent copper. Electron microprobe analysis indicates little copper variation in the cross section of the deposit. Four-point-probe electrical resistivity measurements on typical deposits recorded values of 3.0 $\mu\Omega\text{-cm}$. The density of the deposits 11.2 μm thick was within 1 percent of theoretical, and the adhesion of the deposits on the polyimide substrate was found to meet post-deposition processing requirements. A pull test of the aluminum-copper film was developed. The resulting adhesion values were found to be independent of the deposition parameters, and the films withstood subsequent processing and handling.

DISCUSSION

SCOPE AND PURPOSE

The objective of this project is to establish a plating technique to deposit 6.3 to 12.7 μm of aluminum, or other selected metals, with less than 2 percent thickness variation, onto metallic and nonmetallic substrates. Physical vapor deposition (PVD) is the technique chosen.

ACTIVITY

Description of Deposition Equipment

The aluminum-0.3 weight percent copper alloy vapor source is deposited on polyimide substrates, using a modified ion plating system which consists of a vacuum system with a 254-mm oil diffusion pump and liquid nitrogen baffling (Figure 1). With this system, a flat-topped stainless steel bell jar, 610 mm in diameter by 762 mm high, houses a vapor deposition source and a planetary system (Figure 2) for mounting the substrates.

The vapor source is a radio frequency, induction-heated source powered by a 5-kilowatt, 450-kilohertz radio frequency generator connected to a five-turn induction coil. The vapor source is mounted inside the induction coil and consists of a boron nitride-titanium diboride composite crucible held by dense alumina insulators. The planetary system rotates the substrates above and around the centrally-located vapor source. The thickness of the deposit is monitored continuously during depositing by either of two rate-and-thickness monitors.

Improvements have been made in the original vapor source and the rate thickness monitoring technique to provide more stability and reliability in the general deposition process. The vapor source (Item E, Figure 2) was improved to provide repeatable crucible-to-coil and crucible-to-planetary setup geometries. The new coil-crucible design included the removal of most of the insulator material, which caused excessive outgassing during initial crucible heatup. In addition, a larger BN-TiB₂ crucible is used to increase the source charge from 20 to 40 grams. This permits a smaller net volume of charge to be used for a typical deposition run and results in a more repeatable aluminum-copper alloy composition.

Figure 1. RF Vapor Deposition System

Figure 2. Vapor Source and Planetary System

addition, a larger BN-TiB₂ crucible is used to increase the source charge from 20 to 40 grams. This permits a smaller net volume of charge to be used for a typical deposition run and results in a more repeatable aluminum-copper alloy composition.

By removing the insulator material, the critical distance from the top edge of the crucible to the coil was kept more constant. Correct positioning of the crucible to the coil is vital in maintaining the top edge of the crucible at a temperature that will completely vaporize the aluminum. Any shift in this crucible-to-coil relationship results in wicking or spillover of the molten aluminum to the surrounding crucible support system, thus requiring its replacement. The present vapor source system is designed to maintain this optimum setpoint and thus minimize the crucible system replacement. Also, the new design permits the crucible to be removed and replaced without disrupting the rf coil and dense Al₂O₃ heat shield.

A new thickness monitor (Item A, Figure 2) was installed with a slimline design sensor located near the vapor axis. This unit provides a more reliable thickness control than does the larger sensor (Item C) used initially. This thickness monitor also interfaces with a new automatic shutter (Item B) to provide better process reliability.

Substrate Composition

The substrates are made from laminated polyimide (Kapton) and aluminum foil. Two layers of polyimide, 0.127 mm thick, and one layer of aluminum, 0.102 mm thick, are formed into domed segments which are then bonded together with Pyralux adhesive. The aluminum foil acts as an outer backing layer for the polyimide.

Recognizing that the polyimide laminate substrates are a source of contamination, they must be thoroughly cleaned and thermally treated before depositing the aluminum-copper alloy. Dirt particles, lint, fingerprints, and films from the Kapton/Kapton/aluminum foil bonding-forming operation, as well as other contaminants, must also be removed to acquire good quality deposits with acceptable adhesion. A preliminary chemical cleaning procedure does not remove all of these sources of contamination. Additional substrate cleaning before deposition is accomplished by rinsing the laminates with ethyl alcohol, blowing them dry with nitrogen, and baking them at 150°C in atmosphere for 2 hours. The selection of 150°C was based on mass spectrometric analyses of laminated substrates exposed to various temperatures and conditions. These data indicated that a 2-hour bake-out between 140 and 150°C removed most of the water and air from the organic laminates.

Process and Parameter Characterization

General Process Description

Aluminum-0.12 weight percent copper films are deposited under vacuum on the polyimide laminate, using a physical vapor deposition process. After the three laminates have been mounted on the planetary fixtures (Figure 2), and have been cleaned and baked, they are mounted in the planetary system and rotated concentrically about the axis of the vapor source.

The three curved laminated substrates and the vapor source are located on a sphere with a radius equal to the radii of the laminates (Figure 3). This vapor source-to-substrate distance is defined as the zero position.

The general deposition process is graphically illustrated (Figure 4) and relates rf deposition power to the vapor source, vacuum chamber pressure, and process time. As shown, after the vacuum chamber is roughed down to 9 Pa (50 microns) in 12 minutes, the diffusion pump is valved in to take the chamber down to 0.13 mPa (10^{-6} torr). At this pressure, the heatup of the vapor source begins. Normally, the vapor source is under continuous power (15 percent) to reduce temperature cycling of the BN-TiB₂ crucible.

During Soak 2 (at 35 percent power), the temperature of the crucible is brought up to a red stage; at that point, a preheat is initiated at 95-percent power for 4 minutes. The rf power is then reduced to approximately 80 percent for 1 minute, and the shutter is opened to begin deposition of the aluminum-0.3 weight percent copper charge. Power is then adjusted to achieve the desired rate of deposition. Typically, a rate of 5 nm/s is used for total thickness requirements of 11.2 μ m. When the target thickness is reached, the shutter is automatically or manually activated to terminate the deposition. Power is reduced in Soak 3 to the Soak 1 level (15-percent power), and a 25-minute cooling cycle begins. After sufficient cooling of the coated substrates, the system is vented, and the three planets containing substrates are removed. The system can then be prepared for another deposition cycle. It should be noted that before the actual deposition process begins, the laminates are baked under vacuum in the deposition chamber during Soak 1 and Soak 2 until the temperature in the chamber reaches 150°C, and then are cooled in vacuum to 75°C prior to coating.

Residual Gas Analysis

Deposition characteristics are dependent on the vacuum environment;¹ consequently, the vacuum deposition environment was monitored with a quadrupole mass spectrometer. After monitoring

Figure 3. Planetary System, Showing Rotational Axis and Vapor Source Location

several deposition runs to verify the consistency of results, typical gas spectra and species were documented. Table 1 represents intensities of gas species for a typical deposition run. Figure 5 shows the relative intensities for baseline vacuums prior to and during deposition. The resulting spectra before bake-out and deposition indicated hydrogen, nitrogen, water, carbon monoxide, oxygen, and carbon dioxide. All of these species are expected during thermal treatment of the laminated polyimide substrates in a high vacuum environment. After high vacuum bake-out, the water and oxygen peaks are reduced substantially. During deposition, the major constituents are hydrogen, nitrogen, water and carbon monoxide. The increased hydrogen is due to the dissociation of the water by the reactive aluminum and the formation of aluminum oxide and hydrogen.

Figure 4. Typical Deposition Process

Table 1. Process Description-Quadrupole Mass Spectrometric Analyses

Process Stage*	Time Into Process (min)	Deposition Power (%)	Substrate Temperature (°C)	Chamber Pressure (mPa)	Quadrupole Mass Spectrometric Analyses** Relative Intensities (Arbitrary Units)					
					Atomic Mass Units					
					2	14	16,17,18	28	32	44
Soak 1										
Hi-Vac Chamber Bake	45	15	25	0.35	0.1	0.06	1.35	1.29	0.25	---
2 min	47	15	45	1.20	0.38	0.14	7.9	1.89	0.45	0.07
8 min	53	15	65	1.20	0.85	---	10.19	1.93	0.5	0.1
16 min	61	15	120	1.00	0.65	0.15	7.25	1.95	0.42	0.1
27 min	72	15	150	0.80	0.48	0.14	5.1	1.90	0.35	0.1
Chamber Cooldown	82	15	70	0.75	0.20	0.06	1.85	1.15	0.12	---
Soak 2	92	35	70	0.43	0.18	0.10	2.85	1.4	0.35	---
Preheat	96	95	65	0.48	0.85	0.15	1.55	2.15	0.05	0.1
Deposition Start	97	90	75	0.78	7.92	0.28	0.60	6.85	0.08	0.05
Deposits										
2 μm	104	90	150	1.56	6.45	0.50	0.55	9.63	0.1	0.15
3 μm	107	81	175	1.70	6.38	0.50	0.50	9.63	0.08	0.18
5 μm	113	81	190	1.70	6.55	0.50	0.50	9.63	0.08	0.18
6 μm	116	81	202	1.70	6.55	0.50	0.55	9.63	0.08	0.20
8 μm	123	81	205	1.80	7.05	0.50	0.50	9.58	0.07	0.25
10 μm	130	81	205	1.80	6.95	0.45	0.55	9.60	0.08	0.25
11 μm	133	81	213	1.60	7.05	0.40	0.45	9.63	---	0.15

Table 1 Continued. Process Description-Quadrupole Mass Spectrometric Analyses

Process Stage*	Time Into Process (min)	Deposition Power (%)	Substrate Temperature (°C)	Chamber Pressure (mPa)	Quadrupole Mass Spectrometric Analyses** Relative Intensities (Arbitrary Units)					
					Atomic Mass Units					
					2	14	16,17,18	28	32	44
End, 11.2 μm	135	15	200	0.52	4.08	0.07	0.30	1.5	---	0.07
Cooldown 1 hr	195	15	95	0.23	0.50	0.07	0.20	1.28	---	---

*Process Stage. Refer to Figure 4. Process modified slightly to allow for analysis; required longer processing time.

**Run 598 at 5 nm/s.

Figure 5. Quadrapole Mass Spectrometric Analyses

The increased carbon monoxide and nitrogen intensity (28 amu) during deposition comes primarily from the boron nitride-titanium-diboride composite crucible in the vapor source. Deposition runs were made without the laminated substrates and showed a similar increase in the 28-amu peak, to confirm that BN-TiB₂ crucibles in the vapor source constitute the main contributor to this observed 28-amu peak. The observed intensities of oxygen and water during deposition represent partial pressure to deposition rate (5 nm/s) ratios of 0.18 nPa·s/nm (1.4×10^{-11} torr·s/Å) for oxygen and 8.1 μPa·s/nm (6.2×10^{-9} torr·s/Å) for water. These partial pressure values for oxygen and water are below the critical points expressed by F. d'Heurle.² A mass spectra scan above 50 amu during deposition at a rate of 5 nm/s did not reveal any other significant amount of evaporated species in that range.

Vapor Source Position and Deposition Rate Effects

To evaluate the effects of the vapor source position on the uniformity of the deposit, several deposition runs were made at different positions relative to position zero (Figure 3) with a constant 7.5 nm/s deposition rate. Depositions were made at -1, -1/2, zero, and +1 vapor source positions, where the -1 position corresponded to a vapor source position 25.4 mm below the circle that represented the diameter of the curved laminates being plated. Similarly, the -1/2 position corresponded to 12.7 mm below the zero position and the +1/2 position to 12.7 mm above the zero position. The results of these tests are shown in Figure 6. It will be noted that the zero position gives the lowest uniformity value; that is, the least variation in thickness.

The effect of the deposition rate was also analyzed and is demonstrated in Figure 7. Results indicate a very slight tendency for the uniformity to decrease with deposition rate from 5 to 10 nm/s. Consequently, the deposition rate was maintained between the limits of 5 to 10 nm/s for this study.

Material Distribution from RF Vapor Source

The aluminum-copper alloy vapor distribution from the rf induction source is highly dependent on several features. These include the power level being applied to the crucible, temperature of the crucible, material composition of the crucible, geometry of the crucible, position of the crucible relative to the induction coil, purity and composition of the evaporated aluminum-0.3 percent weight copper source material, and the vacuum in the chamber during deposition. Consequently, steps were taken to control these effects to assure consistent deposits.

Figure 6. Thickness Uniformity Versus Vapor Source Position

Figure 7. Thickness Uniformity Versus Deposition Rate

The rf source utilized for this activity was shown previously in Figure 2. After evaluating these different features, the following parameters were established to evaluate the vapor distribution. The power level applied to the BN-TiB₂ crucible and the resulting temperature of the crucible were maintained to achieve the desired deposition rates of 5 and 10 nm/s. The standard BN-TiB₂ composite crucible was designed with a 15-degree internal taper. This crucible was positioned constantly at the standard zero position. The material of evaporation was the aluminum-0.3 weight percent copper alloy. The vacuum during deposition was standardized at 1.3 to 2.6 mPa (1 to 2 x 10⁻⁵ torr). Planets containing the curved substrates were located in the normal positions. Two deposition runs were made at 5 and 10 nm/s without rotating the planets. Data were collected (Table 2), using beta backscatter to establish the thickness/rate values which include physical measurements of the vapor angles and the associated source-to-substrate distances.

Table 2. Relationship of Vapor Angle and Source Substrate Distance to Deposition Rate

Target Rate = 5 nm/s

Vapor Angle (θ_v)	R(S)	Rate (nm/s)
0.00	37.13	6.00
12.20	36.32	5.65
30.00	32.28	5.00
41.00	30.23	4.55
44.00	26.03	4.35
47.00	25.30	4.10

Target Rate = 10 nm/s

Vapor Angle (θ_v)	R(S)	Rate (nm/s)
0.00	37.34	12.2
12.20	36.45	11.5
30.00	32.28	9.9
41.00	30.23	8.63
44.50	26.03	7.9
47.00	25.30	7.3

A deposition rate gradient for each of the two target deposition rates is generated (Figures 8 and 9), using these data (Table 2) in the cosine distribution for a surface source, as described by Behrndt.^{2,3} To acquire uniform deposit thicknesses over the entire surface of the curved substrate, the substrates are rotated to take advantage of the rate gradient. The observed deviation in the region of the 45-degree vapor angle for both is believed to be caused by the crucible dimensions which inhibit an ideal surface source condition.

Deposition Temperature

The maximum temperature experienced during deposition for the previously thermal-treated laminated substrates is a very important parameter in the deposition process. It can affect the

Figure 8. Deposition Rate Gradient (Target Deposition Rate, 5 nm/s)

Figure 9. Deposition Rate Gradient (Target Deposition Rate, 10 nm/s)

appearance of the final aluminum-copper deposits, primarily by the amount and type of outgassing species from the heated substrates that occur during the deposition. An alumel-chromel thermocouple, placed off the surface of the substrate, serves as a stationary reference thermocouple (Figure 10) to provide deposition temperature data on a run-to-run basis.

To correlate this stationary reference temperature with actual substrate temperatures that occur during deposition, a special polyimide laminate was prepared, using several thermocouples bonded to laminate layers and other positions in the chamber (Figure 10). Typical temperatures of laminate substrates measured with this setup during deposition are shown in Figure 11. The 5-nm/s run resulted in a maximum substrate temperature of 190°C, and the 10-nm/s run resulted in 225°C maximum. Because the temperature of the initially positioned stationary reference thermocouple normally trailed the substrate temperature by 40°C, it was repositioned until its temperature correlated with the maximum substrate temperature for each deposition rate.

The individual layers in the laminate showed slight temperature differentials during deposition. The coolest area was the aluminum foil backing, which was expected. The hottest area was between the two Kapton layers, which indicates that if the laminate separates it will probably begin in the Pyralux adhesive layer bonding the two Kapton layers.

Thickness and Uniformity of the Deposition

The thickness and uniformity were established, using the beta backscatter technique,⁴ by measuring 30 points on the surface of the coated laminates. The average total thickness and uniformity were then established by taking the coefficient of variation (standard deviation divided by average thickness).

Data from a typical laminate (Part L-0824) are given in Tables 3 and 4. The average total thickness of L-0824 was 11.17 μm with a standard deviation of 0.13 μm . This is equivalent to a thickness uniformity number, or coefficient of variation, of 1.14 percent.

Table 4 includes additional information on uniformity. The matrix average gives the thickness deviations for concentric contour rings at various angles from the pole of the laminate and deviations in thickness of wedge sections of the part from center to outside. Each contour ring is represented by five beta backscatter measurements at some angle from the pole of the laminate. For instance, at 4 degrees, contour ring, the five measurements show a thickness average of 11.33 μm with a deviation of 0.0736 μm , while at an angle of 36 degrees the five measurements

10A. THERMOCOUPLE LOCATIONS ON A BONDED LAMINATE SUBSTRATE

10B. LAMINATE-THERMOCOUPLE POSITIONING

Figure 10. Stationary Reference Thermocouple and Special Laminate Bonded With Thermocouples for Monitoring Deposition Temperature

Figure 11. Laminate Substrate Temperature Versus Deposition Time at 5 nm/s Deposition Rate

Table 3. Beta Backscatter Thickness Measurements

Data Point	Counts	Thickness (μm)
1	174730	11.3347
2	174256	11.23
3	174664	11.32
4	175136	11.4367
5	174774	11.3444
6	174685	11.3247
7	173702	11.109
8	174264	11.2318
9	174494	11.2824
10	174502	11.2842
11	174284	11.2362
12	174060	11.1871
13	173989	11.1715
14	174081	11.1917
15	174668	11.3209
16	173879	11.1475
17	173437	11.0516
18	174092	11.1941
19	173994	11.1726
20	173770	11.1238
21	173277	11.0171
22	173767	11.1232
23	173034	10.9517
24	173622	10.994
25	173329	11.0283
26	173171	10.9943
27	173062	10.9709
28	173548	11.0756
29	173496	11.0644
30	173995	11.1729
Part	L-0824	
Tester	100	
Date	9/28/76	
Measurements With Vacuum and Mask		
Thickness Average	11.1696 μm	
Standard Deviation	0.1276 μm	
Uniformity	1.14 Percent	

Table 4. Matrix Average for Beta Backscatter Thickness Measurements

Part	Angle From Pole of Laminate (Degrees)						Wedge Sections	
	4	12	20	24	31	36	Average	Deviation
	Thickness (μm)						(μm)	(μm)
Contour Ring	11.3347	11.3247	11.2362	11.1475	11.0171	10.9943	11.1758	0.1483
	11.23	11.109	11.1871	11.0516	11.1232	10.9709	11.112	0.093
	11.32	11.2318	11.1715	11.1941	10.9517	11.0756	11.1575	0.1285
	11.4367	11.2824	11.1917	11.1726	10.994	11.0644	11.1903	0.1574
	11.3444	11.2842	11.3209	11.1238	11.0283	11.1729	11.2124	0.1246
	Average							
	11.3332	11.2464	11.2215	11.1379	11.0229	11.0556		
	Deviation							
	0.0736	0.0836	0.0606	0.055	0.0633	0.0793		

show a thickness average of 11.056 μm and a deviation of 0.0793 μm . The six contour rings all recorded very consistent deviations with slightly decreasing thicknesses, from 11.33 μm in the central area to 11.06 μm near the outside of the curved laminate. The wedge sections represent six beta backscatter measurements taken from 4 degrees to 36 degrees from the pole of the laminate. Five equally spaced wedge sections were measured, and the thickness average was found to be very consistent.

The average uniformity number for 100 similarly curved laminates that were plated to 11.2 μm total thickness in the standard zero position is 1.21 percent, as determined for a full 30 beta backscatter measurements performed on each sample.

Thickness Repeatability

After establishing the optimum source position and deposition rate, the capability to deposit a film repeatably on a substrate was addressed. Among the most important features of this effort were the selection of a good thickness monitor and a reliable, repeatable thickness measurement technique. When the vapor source provides a repeatable vapor flux density, these items represent the primary control features for repeatable thickness values.

The thickness repeatability (yield) is defined as the number of parts within $\pm 0.5 \mu\text{m}$ divided by the total number of parts plated (illustrated in Figure 12 for the period from May through December, 1976). Improvements in the thickness monitoring device and the vapor source have contributed to the higher yield. The 72-percent yield for November, however, was below the normal for that time period. This lower yield corresponds to the period when only the older monitor was used, because the primary thickness monitor was returned to the factory for repair. Improvements in the beta backscatter technique⁵ and the establishment of experimental uncertainty also have contributed to these increased yields in meeting desired total thicknesses.

In general, the average yield for this rf vapor source system for a particular-sized substrate is approximately 90 percent. Variations encountered in the BN-TiB₂ crucible materials, their thermal behavior and vapor source position, substrate temperature and surface roughness, deposition vacuum, and planetary fixtures make a full 100-percent yield difficult to attain with respect to a desired total thickness.

Figure 12. Yield of Acceptable Parts Meeting Values for Total Thickness

Thickness Standards

The developed PVD process was used to prepare thickness standards of aluminum-copper and copper for polyimide (Kapton) substrates in preparation for the beta backscatter measurement study of these coated polyimide laminates.

Two fully-curved laminate substrates in planets were used in preparing the thickness standards, in addition to a third planet containing a fixture holding three 25.4-mm-diameter parallel optical flats and three 25.4-mm-diameter polyimide laminated aluminum buttons, all rotated to obtain acceptable primary uniformity. Thickness data for the standards are acquired by contact interferometer from the optical flats.

Thickness standards made to date (Table 5) provide aluminum-0.12 weight percent copper films with thicknesses from 9 to 13.4 μm and pure copper films ranging from 4.1 to 11.9 μm . These beta backscatter thickness standards permit high reliability in measuring of the aluminum-copper and pure copper deposits on polyimide. Copper standards are summarized in Table 6.

Weight gain, contact interferometry, and beta backscatter methods⁵ were used in the development of these thickness standards. A set, composed of 13 primary standards, was verified, certified, and put into service by the Metrology Engineering Department at Bendix. These standards were used to certify reference units (working standards) which are used by inspection and test engineering departments to inspect these products. All working standards are put on a periodic recall, at which time they are inspected for damage and compared to a set of primary standards. As a result of this procedure, the working standard is either recertified or replaced with a certified standard prior to its usage⁵.

The effectiveness of the primary standards was evaluated by a series of statistical tests, which were performed on each set of calibration data to provide an effective means to gauge the merit of the calibration data. From the data it was found that 90 percent of the count/second data points projected to thickness will be within $\pm 0.5 \mu\text{m}$ 90 percent of the time. This statement does not include variations that might exist in the acquisition of the count/second data on the reference units or any bias that might exist in the standard set as a whole.

Table 5. Summary of Aluminum-
0.12 Weight Percent
Copper Thickness
Standards

Deposited 11-75 through 3-76		
Deposition Run	Optical Flat	Thickness (μm)
1	1	11.18
1	3	11.20
2	5	10.03
2	6	9.98
2	7	10.01
2		9.96
4	13	12.57
4	14	12.60
4	16	12.52
4		12.42
5	5	12.62
5	6	12.60
5	9	12.60
		12.57
6	2	
6	10	10.08
6	12	10.08
6		10.06
7	17	13.39
7	18	13.36
7	19	13.41
		13.41
9	25	11.61
9	26	11.66
9	27	11.61
10	28	11.43
10	29	11.43
10	30	11.46
11	4	9.09
11	8	9.17
11	31	9.27

Table 5 Continued. Summary of
Aluminum-
0.12 Weight
Percent
Copper
Thickness
Standards

Deposited 2-77

Deposition Run	Optical Flat	Thickness (μm)
1	12	12.19
	15	12.24
	20	12.32
2	21	10.52
	22	10.57
	30	10.64

Deposit Characterization

Microstructural Analysis of Deposit

Typical surfaces and a fractured cross section of the aluminum-copper alloy deposits are shown in Figure 13. The periodic hillocks (white abnormalities in Figures 13B and 13C) on the film's surface are localized nucleation sites which are typical of thick PVD aluminum deposits.^{2,6,7} The addition of the 0.3 weight percent copper in the aluminum significantly reduced the initial number of these hillocks observed for pure aluminum deposits.^{6,8,9}

Defects typically originate from fingerprints, dirt, lint, and fibers that collect on the polyimide surface prior to loading a substrate into the vacuum chamber. The initial surface conditions of the polyimide substrate prior to deposition definitely determine the density of these defects on the deposit. Typical particulate defects are illustrated in Figure 14. Figure 14A shows a fibrous defect resulting from lint fibers present on the polyimide before deposition. It was found that lint and dirt particles are strongly attracted to the polyimide by electrostatic charges, and special handling and preparation techniques must be followed to reduce these defect sources. When these particulate-coated substrates are vapor deposited, defects such as those shown in both Figures 14A and 14B are present. In Figure 14B,

Table 6. Summary of Pure Copper
Thickness Standards

Deposited 1-77		
Deposition Run	Optical Flat	Thickness (μm)
1	4	7.21
	5	---
	8	---
2	9	9.83
	19	9.83
	23	9.88
3	16	10.44
	25	10.44
	54	10.49
4	55	11.76
	56	11.86
	57	11.81
5	4	10.64
	19	10.64
	23	10.61
6	16	10.41
	34	10.41
	31	10.41
7	32	4.06
	41	4.18
	42	4.19
8	45	10.36
	24	10.41
	52	10.34
9	9	9.78
	53	9.78
	56	9.80
10	10	8.79
	4	8.81
	23	8.74
11	16	11.02
	56	11.09
	9	11.05

13A. FRACTURED CROSS SECTION

PART NUMBER	L-0325
DEPOSIT THICKNESS	11.5 μm (115 $\text{\AA}$)
SUBSTRATE TEMPERATURE	180°C
VAPOR SOURCE MATERIAL	Al-0.3 WEIGHT PERCENT Cu
DEPOSITION RATE	5 nm/s (50 $\text{\AA}$ /s)

13B. SURFACE (45 DEGREES TILT)

13C. SURFACE (45 DEGREES TILT)

Figure 13. Fractured Cross Section and Surface of Aluminum-0.12 Weight Percent Copper Film (SEM Photomicrographs)

14A. LINT DEFECT, AFTER DEPOSITION

14B. DIRT DEFECT, ORIGINATING ON SUBSTRATE

14C. GENERAL DEFECT, ORIGINATING ON SUBSTRATE

14D. CROSS SECTION OF AFFECTED DEPOSIT

Figure 14. Observed Defects in Deposited Aluminum-Copper Films (SEM Photomicrographs)

the smaller whitish protrusions are the hillock-type defects mentioned. Other defects shown in Figure 14C typify those that originate from contaminated or deformed polyimide surfaces. A cross section (Figure 14D) through a defect (Figure 14C) illustrates its origin in the polyimide surface.

Copper Content Distribution

Thin films deposited from a liquid alloy source will normally have compositions different from their source.¹⁰ The degree of difference was investigated for the aluminum-copper alloy, using the electron microprobe and atomic absorption analytical techniques. The electron microprobe was used to evaluate the copper distribution in the deposit on a domed polyimide laminate. An elemental distribution analysis was performed with an electron beam size of approximately 1 μm being scanned across seven regions of a metallurgically prepared deposit. Ten data points for each of these seven locations were used to determine an average aluminum and copper content. Quantitative contents, as determined with a computer reduction program indicated that the copper contents were 0.12 weight percent within ± 0.02 weight percent and homogeneously distributed across the aluminum-copper films.

Consecutive runs using the identical aluminum-0.3 weight percent copper starting material were expected to result in a copper enrichment of the vapor load and ultimately the deposit. The trend was evaluated, using 91 aluminum-copper deposits from seven different crucibles. The atomic absorption technique was used to determine the average copper content of coated polyimide coupons that had been placed in the vacuum system for each consecutive run. Atomic absorption results indicated that the average increase in weight percent copper per consecutive deposition was approximately 0.003 percent. In addition, the atomic absorption data of these 91 coupons show the average copper content to be 0.12 ± 0.04 weight percent, which agrees with the electron microprobe data from the domed substrate referred to above.

Resistivity

The resistivity of the vacuum deposited aluminum-0.12 weight percent copper alloy on polyimide laminates was measured, using a four-point in-line probe system. This resistance and the film thickness measured with beta backscatter were used to calculate the resistivity of the deposited film.

The theoretical room temperature resistivity of pure aluminum thicker than 1 μm is $2.71 \mu\Omega\text{-cm}$.^{11,14} This reported value is dependent upon other effects, such as grain size, deposition rates, and substrate conditions, as well as the inherent errors in the resistance and thickness measurements.

Three polyimide laminates with aluminum-0.12 weight percent copper deposits were evaluated, using the four-point probe system. Thirty points per part were measured. The resulting average resistivity for each of the parts was 2.92, 3.01, and 3.03 $\mu\Omega\text{-cm}$ (Table 7).

These resistivities appear relatively constant for the aluminum-copper deposited films from the rf induction source and do not vary from pole to skirt. As mentioned, grain boundaries and interstitial contaminants introduced by the high temperature exposure of the laminated substrates during deposition affect resistivity. However, the observed resistivity values for the different parts from different deposition runs do not reveal these effects. As previously discussed, mass spectrometric analysis data suggest another contaminant source because of the species outgassed from the laminate that could be co-deposited with the aluminum-copper film. However, organic contamination from the substrate was not observed to any degree from the gas analysis test performed during deposition. It appears, however, that the major contaminating contributor was coming from the BN-TiB₂ crucible, as detected by mass spectrometric analysis during deposition. Another secondary contributor to the increased resistivity is the 0.12 weight percent copper content which is likely to be located in the grain boundaries of the aluminum and could act as an impurity.¹¹ However, transmission electron microscopic data are not available at this time to confirm this preferred grain boundary location.

Density

Density of the aluminum-0.12 weight percent copper films was examined, using Trioptic Measuring Machine, light section microscope, contact interferometer, and an analytical balance. Optical flats were weighed and then deposited with the aluminum-copper alloy. The average density of the deposits was found to be 2.68 g/cm³ with an uncertainty of ± 4 percent. The theoretical density of aluminum-0.12 weight percent copper is 2.7 g/cm³ at 20°C.¹² These data indicated that the thick aluminum-copper films are within 1 percent of theoretical density. This difference in density between theoretical and the PVD material could have easily resulted from the measurement uncertainties of the technique and the equipment used. A measurement with higher precision and accuracy is required to determine whether or not the deposits are 100-percent dense.

Adhesion

Adhesion testing necessarily involves testing the coatings to failure; consequently, an exact prediction of the strength of the

Table 7. Four-Point Probe Resistivity

Part	Resistivity ($\mu\Omega$ -cm)		
	R-866	R-923	R-924
Pole 1	2.9068	3.0088	3.0312
Ring 2	2.9168	2.9924	3.0070
3	2.8992	2.9967	3.0132
4	2.9402	3.0284	3.0338
5	2.9346	3.0149	3.0455
Skirt 6	2.9457	3.0322	3.0747
Average ($\mu\Omega$ -cm)	2.9239	3.0122	3.0342
Standard Deviation ($\mu\Omega$ -cm)	0.0190	0.0162	0.0243
Coefficient of Variation (Percent)	0.65	0.54	0.80

aluminum-copper film and polyimide interfacial adhesion is impossible. Experimental data are available which relate the effects of the pretreatment of a polyimide surface to acquire higher joint and peel strengths.¹³ However, the predeposit cleaning and vacuum thermal treating procedures used throughout this activity on the laminated samples and the deposition process have provided aluminum-copper films with good adhesion. Consequently, adhesion has not been a problem, but it is a film property which should be characterized.

To establish a semiquantitative measure of adhesion of the aluminum-copper deposit on polyimide, a direct pull test was developed which used cyano-acrylic adhesive-coated aluminum cylinders (64.5 mm² pull face) to sandwich the aluminum-copper deposited polyimide specimens. The specimens are pulled and identified as to fracture strength and location.

The results of the adhesion testing did not indicate any particular relationship between the deposit's vapor source composition,

thickness, substrate temperature, and the achieved adhesion values. The pull strength values were not indicative of the aluminum-copper deposit/polyimide interfacial adhesion. The failure regions in all cases were in the adhesive zone either between the aluminum plug and the polyimide substrate or the aluminum-copper deposit and the aluminum plug. The highest pull strength value achieved before the adhesive failed was 17.13 MPa. In general, the results of this pull test showed the adhesion of the deposited films to be above that of the adhesive used to bond the coated samples to the aluminum plugs. In addition, deposit/polyimide adhesion is more than satisfactory to withstand subsequent processing and handling.

ACCOMPLISHMENTS

A physical vapor deposition process with an rf induction vapor source has been developed to deposit 11.2- μm aluminum-copper films on polyimide laminate substrates. The total thickness values can be repeated within a $\pm 0.5\text{-}\mu\text{m}$ range with a 90-percent yield. The average thickness uniformity (standard deviation divided by average thickness) of the deposit acquired by beta backscatter for 100 substrates was 1.2 percent. The aluminum-0.3 weight percent copper vapor charge results in a copper content in the deposit of approximately 0.12 weight percent. The copper shows no preferential deposition pattern, as related to the surface of the laminate and its dispersion throughout the deposit.

The maximum laminated substrate temperatures are 190 and 225°C, respectively, for 5 nm/s and 10 nm/s deposition rates. The resistivity of the aluminum-copper deposits is 3 $\mu\Omega\text{-cm}$ and is stable from one deposition run to another. Also, the resistivity did not appear to be related to the geometry of the domed substrate. The density of the 11.2- μm aluminum-copper film was within 1 percent of theoretical, and its adhesion to the polyimide exceeds that required for postdeposition operations and handling. Physical vapor-deposited aluminum-copper films for beta backscatter thickness standards were provided and were instrumental in achieving the reported uniformity values and determining the beta backscatter measurement uncertainty.

FUTURE WORK

Future activity associated with the rf induction source system includes the installation of a new single-planet fixture (Figure 15) for deposition of larger domed-shaped polyimide laminate substrates. The new fixture has the capability for both two-axis and single-axis rotation. Also, a new lower frequency

Figure 15. Large Planetary Fixture

rf induction source (In Source) will be examined. This unit will have an autoloop control for maintaining the deposition rate. These two new equipment items are expected to improve deposit uniformity of flat laminates and permit examination of their deposition requirements.

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deposit of approximately 0.12 weight percent. The copper shows no preferential deposition pattern, as related to the surface of the laminate and its dispersion throughout the deposit. The resistivity of the aluminum-copper deposits is $3 \mu\Omega\text{-cm}$ and is stable from one deposition run to another. Also, the resistivity did not appear to be related to the geometry of the domed substrate. The density of the $11.2\text{-}\mu\text{m}$ -thick aluminum-copper film was within 1 percent of theoretical, and its adhesion to the polyimide exceeds that required for postdeposition operations and handling. Physical vapor-deposited aluminum-copper films for beta backscatter thickness standards were provided and were instrumental in achieving the reported uniformity values and determining the beta backscatter measurement uncertainty.

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