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DEPOSITION AND CHARACTERIZATION OF VACUUM
DEPOSITED ALUMINUM FILMS ON KAPTON LAMINATES

By D. M. Sherman
Department 814

Published July 1978

Topical Report
G. J. Hale, Project Engineer

Prepared for the United States Department of Energy
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DEPOSITION AND CHARACTERIZATION OF VACUUM DEPOSITED ALUMINUM FILMS ON KAPTON LAMINATES

BDX-613-1873 (Rev.), UNCLASSIFIED, Topical Report, Published July 1978

Prepared by D. M. Sherman, D/814

A process has been developed for the vacuum deposition by electron beam evaporation of high quality aluminum films 10 μ m thick on domed three-layer laminated substrates consisting of Kapton/Pyralux/Kapton/Pyralux/Aluminum (Du Pont Corporation). Thermogravimetric analysis and mass spectrometry of the substrate materials and in-process residual gas analysis were used to determine the outgassing characteristics of the substrate laminate and to aid in the development of suitable thermal processing. The laminated substrates required bakeouts both at atmosphere and in high vacuum prior to deposition to permit evaporation at a pressure of 0.1 mPa (1×10^{-6} torr). Film properties that were obtained were a thickness uniformity across a 200 mm diameter part in the 1 percent range, a resistivity near that of bulk pure aluminum, a smooth and shiny film surface, and adequate film adhesion.

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CONTENTS

Section	Page
SUMMARY.	5
DISCUSSION	6
SCOPE AND PURPOSE.	6
PRIOR WORK	6
ACTIVITY	6
<u>Substrates</u>	6
<u>Deposition System.</u>	7
<u>Deposition Procedure</u>	11
<u>Residual Gas Analysis.</u>	12
<u>Film Thickness Uniformity.</u>	12
<u>Film Surface Reflectance</u>	15
<u>Electrical Resistivity</u>	20
<u>Microstructure and Preferred Orientation</u>	23
ACCOMPLISHMENTS.	25
FUTURE WORK.	26
REFERENCES	27
DISTRIBUTION	29

ILLUSTRATIONS

Figure		Page
1	Schematic Cross Section of Substrate Laminate.	7
2	Electron Beam Source Deposition System (P-95453)	8
3	Interior View of Deposition Chamber (P-95458)	9
4	Dome-Shaped Substrate Mounted in Planetary Fixture (P-96583, P-96582).	10
5	Residual Gas Spectra of Deposition Chamber. .	13
6	Beta Backscatter Film Thickness Measurement Assembly (P-94066).	14
7	Film Thickness Uniformity Versus Source/Sub- strate Vertical Separation and Substrate Radial Offset in the Horizontal Plane From the Electron Beam Evaporator Hearth.	15
8	Laser Reflectometer Schematic	17
9	SEM Micrographs of the Surfaces of 10- μ m-Thick Aluminum Films (P-98345).	19
10	SEM Micrographs of Four Copper-Doped Aluminum Films of Varying Optical Appearance (P-98316)	21
11	Aluminum Resistivity Versus Relative Rates of Impingement of Reactive Residual Gases and Aluminum on the Surface	22
12	(111) X-Ray Pole Figure of Typical 10- μ m-Thick Aluminum Film on Kapton . . .	24
13	(111) X-Ray Intensity Versus Angle From the Sample Normal for a Typical 10- μ m-Thick Aluminum Film on Kapton	25

SUMMARY

Aluminum is extensively deposited onto plastics by vacuum evaporation^{1,2} for both functional and decorative applications. It is well known that polymeric materials may contain volatile constituents in the form of moisture, solvents, or plasticizers. These components may be liberated in vacuum and result in deposited film deficiencies such as poor adhesion, low reflectance, or high resistivity. Satisfactory results for some applications can be obtained² by lacquering surfaces, especially of decorative parts, before vacuum processing or by using vacuum systems with exceptionally high pumping speeds. Some compromise is generally reached, however, between process simplicity and deposited film properties.

A process has been developed for the vacuum-deposition by electron-beam evaporation of high quality aluminum films 10 μm thick on domed three-layer laminated substrates consisting of Kapton/Kapton/aluminum foil (Du Pont Corporation) bonded with Pyralux (Du Pont), a modified acrylic thermoplastic adhesive. Thermogravimetric analysis and mass spectrometry of the substrate materials and in-process residual gas analysis were used to determine the outgassing characteristics of the substrate laminate and to aid in the development of suitable thermal processing. The laminated substrates required bakeouts both at atmosphere and in high vacuum prior to deposition to permit evaporation at a pressure of 0.1 mPa (1×10^{-6} torr).

Required film properties were a thickness uniformity across a 200 mm diameter part in the 1 percent range, a resistivity near that of bulk pure aluminum, a smooth and shiny film surface, and adequate adhesion. A beta backscatter technique was used for film thickness determination; uniformity in the 1 percent range was attained. An integrating sphere reflectometer was used for surface appearance measurements; the process that was developed yielded films whose surfaces had a total reflectance of 90 percent with 80 to 90 percent of the reflected light specularly reflected. Film resistivity was found to be an indication of the relative rates of impingement of reactive residual gases and aluminum on the surface during deposition. The resistivity was determined both with a four-point probe and with photoprocessed resistor patterns; the evolved deposition process resulted in an aluminum film resistivity within 5 percent of that of bulk pure aluminum. Film adhesion was sufficient to withstand adhesive tape snap-pull testing. X-ray pole figure analysis showed that the deposited films exhibited a strong (111) preferred orientation.

DISCUSSION

SCOPE AND PURPOSE

The goal of this activity was twofold; first, to develop a process for the deposition of high quality pure aluminum films on dome-shaped Kapton laminate substrates that met stringent film thickness uniformity requirements, and secondly, to develop suitable techniques for the characterization of the deposited films. The deposition technique chosen was thermal evaporation from a commercial electron beam source. The vacuum system used was a newly-acquired stainless steel bell jar system evacuated by an oil diffusion pump. Film characteristics of interest were, in addition to film thickness, surface appearance, electrical resistivity, mass density, adhesion, and film microstructure.

PRIOR WORK

Vapor deposition of aluminum onto laminated Kapton substrates was initiated at Bendix Kansas City in July, 1974. Initial efforts of this development activity were to modify an existing ion-plating vacuum system (Endurex) to include an rf vapor source with appropriate planetary fixturing, to establish an aluminum deposition process with this system to meet test part thickness and uniformity requirements, and to procure a new vacuum system (Davis-Wilder) featuring a commercial electron beam source. During this initial phase, the surfaces of the aluminum films deposited in the rf vapor source system (Endurex) were not as shiny as desired. By adopting a copper-doped aluminum source, the optical appearance of the resultant copper-doped aluminum films was noticeably enhanced; however, the addition of the copper and the resultant potential problems of long-term degradation from a multicomponent materials system were undesirable. Also, while establishing a viable deposition process to provide functional test parts to Lawrence Livermore Laboratory, the characterization of the structure and properties of these initial films was limited. After receipt and installation of the new vacuum deposition system with the electron beam source (Davis-Wilder), this study was initiated and utilized procedures consistent with high vacuum practice for depositing aluminum.

ACTIVITY

Substrates

The substrates consisted of two layers of the polyimide Kapton H backed by a layer of aluminum foil (Figure 1). Each layer was 0.125 mm thick and was pressure formed to the radius of curvature before bonding with a thermosetting sheet adhesive (Pyralux, Du Pont). The inside Kapton layer had a planar brim outside the active substrate to facilitate handling and fixturing.

Figure 1. Schematic Cross Section of Substrate Laminate

Thermogravimetric analysis (TGA) of Kapton was performed to aid in the development of the thermal processing necessary to produce aluminum films with the desired properties. The Kapton displayed an immediate weight loss upon heating from room temperature, and by 140°C there was a weight loss of 1.5 percent. No further weight reduction was noted through 275°C. Subsequent mass spectrometric analysis indicated only a loss of moisture from the Kapton up to 275°C. This result is in basic agreement with the mass spectrometric work of Hait;³ the added outgassing products carbon monoxide and carbon dioxide observed by Hait at 300°C were not observed in this work for Kapton exposures to 275°C. Mass spectrometric analysis of the full Kapton/Pyrallux/Kapton/Pyrallux/Aluminum foil laminate indicated that n-butanol as well as water vapor were outgassed in substantial quantities at temperatures as low as 100°C, but that a bakeout prior to analysis was effective in reducing outgassing. A 1-hour atmospheric prebake at 150°C was found to be sufficient to limit the outgassing of the substrate to only water vapor (within the sensitivity limit of the mass spectrometer--about 1 nPa or 1×10^{-11} torr) at temperatures below 130°C.

Deposition System

The deposition system, manufactured by Davis and Wilder, was a vertically split stainless steel bell jar chamber pumped by a liquid nitrogen-trapped oil diffusion pump. The system and the interior of the deposition chamber are shown in Figures 2 and 3, respectively. Chamber pressure before deposition was typically in the low 10 μ Pa (1×10^{-7} torr) range. The deposition source was a 50-mm diameter copper hearth electron beam source (Airco Temescal Supersource) powered by a 10 kW supply and controlled by a quartz crystal oscillator controller. The source material used was nominal 99.999 percent pure aluminum. The chamber walls and

Text continued on page 11.

Figure 2. Electron Beam Source Deposition System

Figure 3. Interior View of Deposition Chamber

Figure 4. Dome-Shaped Substrate Mounted in Planetary Fixture
(The surface temperature thermometer is shown attached
to the aluminum back layer.)

all permanent chamber fixturing in line of sight with the source were covered with removable stainless steel shielding to minimize system downtime for cleaning and to minimize the level of particulates in the deposition chamber. Small bimetallic surface temperature thermometers were mounted on the back surface of substrates for direct temperature measurement (Figure 4). A free-standing thermocouple positioned adjacent to the substrates was correlated with actual substrate temperatures for use when surface temperature thermometers were not used. Quartz radiant heaters were used for chamber and substrate bakeout. Additional heat for system bakeout was furnished by resistive heaters on the main valve body and hot water in the chamber water jacket and electron beam source hearth. The deposition chamber was equipped with a quadrupole mass spectrometer (UTI 100C, Uthe Technology, Incorporated) for residual gas analysis. Mass spectra were recorded on an X-Y recorder or, when rapid recording was desired, by photographing an oscilloscope trace.

Deposition Procedure

The following sequence permitted aluminum deposition to be performed at a chamber pressure of 0.1 mPa (1×10^{-6} torr) or lower with a total vacuum system cycle time of approximately 2 hours.

- Bake cleaned substrates (mounted in deposition fixture as shown in Figure 4 to facilitate step 2) at atmosphere for a minimum of 1 hour at 150°C.

- Rapidly transfer substrates to deposition chamber. A room temperature air exposure in excess of 5 minutes resulted in substantial moisture absorption and excessively long pumpdown time. Pumpdown time to 0.1 mPa (1×10^{-6} torr) was extended from 40 minutes to 3 hours, for example, when air exposure was increased from 5 to 30 minutes (no vacuum bakeout in either case).

- Pump chamber into 0.1 mPa (1×10^{-6} torr) range.

- Initiate 30 minute low-power bakeout of substrates to 125°C.

- Substrate cooldown step was varied from a minimum of about 1 minute to approximately 8 hours, the time necessary to cool to room temperature, in order to vary the substrate temperature during deposition.

- Aluminum was deposited at 6 nm/s ($60 \text{ \AA/s}$) to a nominal thickness of 10 μm . Total deposition time was thus approximately 30 minutes. Substrate temperature rise during deposition was 40°C for substrates initially at room temperature and about 15°C for substrates at 100° to 110°C before deposition.

Residual Gas Analysis

The chamber pressure rose to about 0.1 mPa (1×10^{-6} torr) during deposition independent of the base pressure before power was applied to the electron beam gun. The source of the residual gas is seen from the mass spectra in Figure 5. The upper spectrum is a typical baseline spectrum taken before deposition. The center spectrum shows the chamber residual gases during aluminum deposition; the total pressure has increased by a factor of five, and there is a dramatic rise in the concentration of hydrogen, methane, carbon monoxide, and carbon dioxide. The lower spectrum was taken while the chamber and fixturing were subjected to electron bombardment from an auxiliary thermionic cathode located at the center of the chamber (deposition run off). A comparison of the center and lower spectra illustrate that the pressure rise during deposition was caused by the electron stimulated desorption of gases from the chamber walls and fixturing and was not, for example, due to the outgassing of the deposition source emitter assembly or the aluminum charge. The difference in the ratio of water vapor to hydrogen partial pressures between the center and lower spectra is due to the dissociation of water vapor by the reactive aluminum and the formation of aluminum oxide and hydrogen in the case where aluminum is being evaporated. Although this desorption result has not been pursued, it may be possible to deposit aluminum in the low 10 μ Pa range by adding a processing step such as electron bombardment degassing or oxygen glow discharge cleaning of the chamber prior to deposition.

Film Thickness Uniformity

The thickness of the aluminum films was determined by a beta backscatter technique developed at Bendix⁴ in which nominally 10- μ m-thick aluminum on Kapton was measured with a precision of ± 1 percent. Each part was measured at 30 points distributed across the dome (Figure 6). The uniformity was expressed as the standard deviation of the 30 measurements divided by the average thickness because of the statistical nature of the beta backscatter process. The dependency of aluminum thickness uniformity upon source/substrate geometry is given in Figure 7. Uniformity was sensitive to both vertical separation of source and substrate (abscissa) and radial offset of the substrates (r); the substrate planetary was centered over the evaporator hearth and the substrate "normal" or planet spindle was maintained 60 degrees from vertical for all experiments. No systematic variation in film thickness across a dome, such as radial thinning as was seen on nonuniform parts, could be inferred from the thickness measurements when optimum geometrical parameters were chosen. The bar at the left of Figure 7 denotes the range of uniformity values, 0.6 to 1.4 percent, obtained for 75 parts deposited at that position; the average uniformity for these parts was less than 1 percent.

Figure 5. Residual Gas Spectra of Deposition Chamber (The electron stimulated desorption was performed with 20 mA emission current at an accelerating potential of 1 kV. The heavier gases (AMU 50, 51, 52, 77, and 78) are attributed to backstreamed pump fluids.)

Figure 6. Beta Backscatter Film Thickness Measurement Assembly

Figure 7. Film Thickness Uniformity Versus Source/Substrate Vertical Separation and Substrate Radial Offset in the Horizontal Plane From the Electron Beam Evaporator Hearth (The vertical bar at the lower left indicates the range of thickness uniformity values obtained for the 75 domed parts deposited at that position.)

Long term monitoring of over 275 domed parts has resulted in some deviation in uniformity from the range shown in Figure 7. Although explicit experiments have not been performed to substantiate sensitivity of thickness uniformity to factors other than source/substrate geometry, it is expected that electron beam source characteristics (power or deposition rate, electron beam size, and molten pool height in crucible), planetary rotation speed, and shutter and shielding placement may very well affect deposited film uniformity.

Film Surface Reflectance

General Considerations

The first assessment of the quality of a vacuum deposited thin film is often how mirror-like or specular the film appears.

Processing variables such as elevated substrate temperatures,^{5,6} high residual gas pressures,^{5,6} impure source material,¹ and substrate stains often lead to cloudy-appearing films. The matte-like appearance of a film may be an indication of potential functional or reliability deficiencies. Film adhesion may be diminished if a contaminated substrate causes cloudiness. High residual gas pressure or the inclusion of impurities from an impure source result in an increase in the resistivity of a metal film.⁷ A smooth surface is one of the prime requirements of a reflective film for optical applications. A rough surface (dull surface) of a first level metal conductor of a multilevel integrated circuit can lead to interlevel shorts,^{8,9,10} and alignment for photolithography can be impaired.^{5,10}

A subjective evaluation of film surface shininess is usually sufficient; any substantial deviation from the norm is apparent. However, a quantitative measure is sometimes valuable for determining smaller variations in appearance than can be readily perceived visually, for providing a permanent record, and to permit a correlation between films that cannot be compared directly. Several test methods and instruments are available for measuring specific chromatic and geometric attributes of the appearance of metal surfaces.^{11,12,13,14} In this study an integrating sphere technique (after Barkmen¹⁴) was used for the measurement of the total and specular reflectance of the films.

Reflectometer Arrangement

The arrangement of the integrating sphere reflectometer is shown in Figure 8. The beam of the 1.5 mW HeNe laser ($\lambda = 632.8$ nm) passed through an aperture and then through two opposite ports of an integrating sphere ($r = 100$ mm). The sample was first positioned such that the specularly reflected beam exited the sphere and was imaged on the aperture. The aperture was dark colored so there was minimal reentry of the specular beam into the sphere. A nearly circularly-shaped specular beam without substantial streaking indicated that the total specular beam had exited the integrating sphere; the light intensity in the sphere as measured by the photodetector (I_D) was proportional to the amount of light diffusely scattered from the film. A second measurement was made in which the sample was tilted slightly to contain the specularly reflected beam inside the sphere; I_T was proportional to the total film reflectance. The fraction of the reflected light that was specularly reflected was thus $(I_T - I_D)/I_T$. Total reflectance values were obtained by replacing the sample by a mirror of known reflectance. A first surface dielectric mirror of 98.4 percent reflectance was used in this study.

Figure 8. Laser Reflectometer Schematic (A dome-shaped sample in position for the diffuse intensity (I_D) measurement. The sample is tilted slightly for the total reflectance measurement. The arrangement is suitable for both planar and dome-shaped surfaces.)

Reflectance Measurements

The total reflectance with the 632.8 nm wavelength source was 90 ± 1 percent. This value is comparable to that reported for freshly evaporated^{1, 15, 16} and sputter-deposited^{5, 17} films in the $0.1 \mu\text{m}$ (1000 Å) thickness range. The amount of reflected light that was specularly reflected was in the 80 to 90 percent range; this corresponds to an optical appearance of a mirror-like surface. The planar nature of a typical film surface is seen in Figure 9; there are only a small number of surface roughening features such as hillocks^{6, 18} to diffusely scatter incident light.

It was anticipated that specular reflectance would decrease with increasing substrate temperature during deposition, but there was no noted variation in the temperature range of 25° to 125°C. A strong dependency was found, however, between specular reflectance and chamber residual gases during deposition. All depositions yielding the reflectance values noted above were performed at a total pressure near 0.1 mPa (1×10^{-6} torr) with the residual gas composition as shown in the center spectrum of Figure 5. A controlled ambient during deposition containing oxygen at a partial pressure of 4 μ Pa (3×10^{-8} torr) or water vapor at a partial pressure of 40 μ Pa (3×10^{-7} torr) resulted in a decrease in specular reflectance to the 25 percent range while the total reflectance decreased only a few percent. Comparative SEM micrographs of the surfaces of a typical 10 μ m film and one deposited at an oxygen partial pressure of 4 μ Pa (3×10^{-8} torr) are given in Figure 9.

Alternative Techniques for Assessing Specular Reflectance

Two other techniques based upon the measurement of diffusely scattered light have been evaluated for the assessment of deposited film appearance. The first consists of measuring the intensity of diffused light reflected into an optical microscope. The arrangement that has been used is for a white light beam incident approximately 60 degrees from film normal to illuminate a film held in the flat position (film normal along microscope optical axis), and the amount of diffusely reflected light to be quantified with a photographic exposure meter. The exposure meter can replace one eyepiece of a binocular microscope or can be mounted in the camera tube of a microscope that is so equipped. An equivalent arrangement would consist of using coaxial bright field illumination to measure a film held at an orientation inclined from the optical axis.

The second technique is the direct application of a commercial photographic reflection densitometer (Macbeth RD-514, Killmorgen Corporation). This instrument directs a light beam onto the film surface at 90 degrees and measures the intensity of the diffusely reflected light at all azimuth angles with an elevation of 45 ± 5 degrees.

For this evaluation, four copper-doped aluminum films whose appearances ranged from quite cloudy to very shiny were selected and are shown (Figure 10). Total reflectance ranges from 80 to 89 percent, while the fraction of reflected light that is specularly reflected varies from 51 to 97 percent. The general surface topographies of the four films are similar (the "matrix") but the concentration of hillocks differs considerably; the higher concentration of hillocks corresponds to the lower reflectance values.

- A. DEPOSITED AT 0.1 mPa (1×10^{-6} torr)
WITH RESIDUAL GAS COMPOSITION AS SHOWN
IN THE CENTER SPECTRUM OF FIGURE 5

- B. OXYGEN PARTIAL PRESSURE MAINTAINED AT
 $4 \mu\text{Pa}$ (3×10^{-8} torr) DURING DEPOSITION

Figure 9. SEM Micrographs of the
Surfaces of 10- μm -Thick
Aluminum Films

The photographic densitometer and microscope exposure meter readings are given below the micrographs in Figure 10. The intensity of light scattered from the dullest film was assigned a value of 100 for both techniques. A high sensitivity exists for both methods in the appearance range that is commonly encountered for vacuum-deposited aluminum films. Furthermore, the correlation between microscope and densitometer readings suggests that the relative reflectivity is not very sensitive to the exact geometrical arrangement of sample, light source, and detector.

Although these techniques provide only relative film shininess information, they do offer ease of measurement and, in the case of the microscope, the capability of characterizing selected areas at high magnification.

Electrical Resistivity

Film sheet resistance was determined both by four-point probe measurements and with suitably photoprocessed resistor patterns. Film thicknesses were obtained by the beta backscatter technique and by interferometry. The resistivity of films deposited at 0.1 mPa (1×10^{-6} torr) was within 5 percent of that of bulk pure aluminum (less than $2.85 \mu\Omega\text{-cm}$ at 20°C). Several tests were performed to determine the tolerance of the resistivity to increased system pressure during deposition. Controlled ambients of air, oxygen, and water vapor were maintained during deposition. The results (Figure 11) were in close agreement with the work of d'Heurle⁶ and Dheer¹⁹ when all results were expressed⁹ in terms of resistivity as a function of relative impingement rates on the surface of the reactive residual gases and aluminum (the ratio of partial pressure to deposition rate).

Resistivities of film on fully domed parts have been determined and expressed by the following evolved procedure.

Film thickness values used were those obtained by the standard beta backscatter procedure; 30 points on six concentric rings were measured. These 30 values were reduced to six by obtaining the average thickness value for each ring.

Film sheet resistance was measured with a four-point probe and a precision microohmmeter; the same 30 points measured initially by the beta backscatter technique were measured. Sheet resistance was obtained by multiplying measured probe resistance by a probe-dependent constant (equal to 4.532 for straight line, equally spaced, probes on an infinite film²⁰). These 30 values were reduced to six by obtaining the average sheet resistance for each ring.

TOTAL REFLECTIVITY (PERCENT)	80
PERCENT SPECULAR	51
DENSITOMETER (RELATIVE)	100
EXPOSURE METER (RELATIVE)	100

TOTAL REFLECTIVITY (PERCENT)	82
PERCENT SPECULAR	74
DENSITOMETER (RELATIVE)	89
EXPOSURE METER (RELATIVE)	86

TOTAL REFLECTIVITY (PERCENT)	84
PERCENT SPECULAR	87
DENSITOMETER (RELATIVE)	54
EXPOSURE METER (RELATIVE)	56

TOTAL REFLECTIVITY (PERCENT)	89
PERCENT SPECULAR	97
DENSITOMETER (RELATIVE)	14
EXPOSURE METER (RELATIVE)	14

Figure 10. SEM Micrographs of Four Copper-Doped Aluminum Films of Varying Optical Appearance

Resistivity was determined for each ring by multiplying ring average thickness by ring average sheet resistance. Single-value resistivity of a part was obtained by averaging the values of the rings.

Figure 11. Aluminum Resistivity Versus Relative Rates of Impingement of Reactive Residual Gases and Aluminum on the Surface

The sheet resistance of one dome was expressed in the same manner as the beta backscatter results (standard deviation of

30 values divided by average sheet resistance); all values were ± 2 percent of average and the uniformity of sheet resistance was 0.8 percent. The thickness uniformity of this film was measured to be 0.9 percent.

Microstructure and Preferred Orientation

Several attempts were made to observe the microstructure of the deposited films. Metallurgical mounting, polishing, and observing optically and in the SEM resulted in some indications of an equiaxed grain structure but none of the etchants used yielded conclusive results. Films were torn at room temperature and at liquid nitrogen temperature; no crystallographic character was observed on the exposed edges. Film microstructural characterization could be accomplished in a straightforward manner by transmission electron microscopy; this has not been performed to date because of the inaccessibility at Bendix of a transmission microscope.

Several segments of deposits, taken both near the dome center and toward the outer skirt, were analyzed with a Norelco pole figure device using $\text{Cu K}\alpha$ radiation. The crystallographic orientation of all deposits showed a very strong fiber texture with (111) parallel to the substrate as illustrated in Figure 12. Vacuum deposited face-centered cubic (FCC) metals often exhibit a (111) fiber texture.²¹ Figure 13 is a plot of X-ray intensity versus angle from the sample normal where the degree of preferred orientation can be seen; the high angle maximum corresponds to the interplanar angle of 70.5 degrees between (111) planes in FCC metals.

Adhesion

Several films were subjected to adhesive tape (Scotch Magic Transparent Tape) snap-pull testing to obtain a qualitative assessment of adhesion. The tape was applied both to finished surfaces and to films scribed with a sharp blade; no interfacial failure occurred in any of the tests.

Film Density

The hygroscopic behavior of Kapton would not permit deposited film mass density measurements to be made on the film/overgrowth system aluminum/Kapton; a small weight variation in the Kapton substrate would cause a severe error in derived film density. Density determinations were made on films deposited on 25 mm diameter quartz optical flats. The flats were positioned behind a circular mask for deposition resulting in a 20 mm diameter circular aluminum deposit on the face of the flat. Film thicknesses were determined with a Link interferometer, a combination stylus/interferometric measurement instrument.

Figure 12. (111) X-Ray Pole Figure of Typical 10- μ m-Thick Aluminum Film on Kapton (The shaded areas denote those regions where X-ray intensity was above background.)

Initial measurements of film volume resulted in density values of 2.48 to 2.60 g/cm³ which is 92 to 96 percent of full aluminum density. An analysis of the volume measurement procedure was undertaken to determine if it could be conclusively stated that the vacuum-deposited films were less than 100 percent dense. A traversing stylus instrument (Dektak, Sloan Technology Corporation) was used to determine the taper of the film edge; a volume reduction and hence a density increase of approximately 3 percent was realized by accounting for the film tapered edge. When the roughness of the film surface is taken into account a further correction of 1 to 3 percent, again in the direction of increasing density, can be realized; the relatively large range of the correction is due to the variable surface roughness obtained by varying parameters such as residual gas pressure and substrate temperature during deposition.

Figure 13. (111) X-Ray Intensity Versus Angle From the Sample Normal for a Typical 10- μ m-Thick Aluminum Film on Kapton

The conclusion of this analysis is that it cannot be conclusively stated that the deposited aluminum films are less than full density; more precise measurement techniques and more sophisticated analytical techniques would be required. Transmission electron microscopy would yield straightforward, conclusive evidence of the presence or absence of microporosity in the vacuum-deposited films.

ACCOMPLISHMENTS

A process has been developed for the vacuum-deposition of 10- μ m-thick aluminum films on domed-shaped laminated Kapton substrates by electron beam evaporation. The desired film properties of thickness uniformity in the one percent range, a high specular reflectance, a resistivity near that of bulk aluminum, and adequate adhesion were obtained. The volatility of the adhesive and the hygroscopic nature of Kapton necessitated extensive bakeout, first at atmosphere and then in high vacuum, before aluminum deposition. The high specular reflectance of

such thick films was unexpected. It is thought that the low partial pressures of such active gases as oxygen and water vapor along with relatively low substrate temperatures contribute to this effect. The films exhibited a high degree of (111) preferred orientation.

Techniques for film characterization that had previously been developed were utilized for ascertaining the quality of the deposited films; these include the beta backscatter technique for film thickness determination and the X-ray pole figure device for preferred orientation determination. Techniques were developed for measuring the total and specular reflectance of the surface of the films, film electrical resistivity, and an improved determination of film mass density.

FUTURE WORK

Future activity should include microstructural characterization of the aluminum films by transmission electron microscopy. A substantial amount of activity is currently taking place in several laboratories to develop a technique for quantifying vacuum-deposited film adhesion onto flexible substrates; a method for measuring the adhesive strength of aluminum on Kapton would certainly be desirable.

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D. E. Lacey, D/816, SG-3	45
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D. M. Sherman, D/814, UNCLAS Topical, July
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