CONF-770339--2

BDX-613-1801

DEPOSITION OF UNIFORM ALUMINUM FILMS ON KAPTON LAMINATES BY ELECTRON BEAM EVAPORATION

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SUMMARY

Aluminum films 10 μm thick with a thickness uniformity in the one percent range have been obtained on the Kapton surface of a laminated substrate consisting of Kapton/Kapton/aluminum foil bonded with a thermosetting adhesive. The processing of the substrates before deposition necessary to obtain reasonable deposition cycle times and a minimal amount of deposition system contamination was developed. 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). The deposited films exhibited a high specular reflectance and were thus mirror-like in appearance. Film resistivity was within 10 percent of that of bulk aluminum, and the films displayed a strong (111) fiber texture.

Running Title: DEPOSITION OF ALUMINUM ON KAPTON

DISCLAIMER

DEPOSITION OF UNIFORM ALUMINUM FILMS ON KAPTON LAMINATES BY ELECTRON BEAM EVAPORATION

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INTRODUCTION

Vacuum-deposited aluminum is the most frequent choice for conductor metallization of integrated circuits[1,2] and for coatings of front-surface mirrors.[3] Aluminum thin films to serve as conductor lines for semiconductor devices are typically in the 1 μm thickness range. Silicon wafers are oftentimes elevated to temperatures near 250°C before aluminum deposition to obtain desired film properties. Aluminum films for mirror applications are typically 0.1 μm thick, and the substrates are not pre-heated before deposition. The substrate materials in both the semiconductor and mirror applications are compatible with high vacuum; i.e., they are thermally stable at temperatures normally encountered in vacuum deposition and have minimal outgassing.

This paper reports on a process for the vacuum-deposition of high quality aluminum films 10 μm thick on the interior surface of domed three-layer laminated substrates consisting of Kapton/Kapton/aluminum foil bonded with a thermosetting 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. A three-substrate planetary arrangement was used to attain thickness uniformities of nearly ±1 percent across a 20-cm-diameter substrate. The required film properties were, in addition to the

stringent thickness uniformity requirement, adequate adhesion, a near mirror-like surface, and a resistivity approaching that of pure bulk aluminum.

EXPERIMENTAL

A. Substrates

The substrates consisted of two layers of the polyimide Kapton H (Du Pont) backed by a layer of aluminum foil, as shown in Figure 1. Each layer was 0.125 mm thick and was pressure formed to the 15-cm radius of curvature before bonding with a thermosetting sheet adhesive (Pyralux, Du Pont). The inside Kapton layer had a planar brim outside the active 20-cm (chord length) substrate to facilitate handling and fixturing.

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 loss was noted through 275°C. Subsequent mass spectrometric analysis indicated only a loss of moisture from the Kapton up to 275°C. Mass spectrometric analysis of the full Kapton/Kapton/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. Although overall outgassing was minimized by vacuum prebaking at 0.1 mPa (1×10^{-6} torr), an atmospheric bakeout at 150°C for two hours was found to be sufficient to result in minimal outgassing of n-butanol in subsequent vacuum exposures to 130°C.

B. Deposition System

The deposition system was a vertically-split stainless steel bell jar chamber pumped by a liquid nitrogen-trapped oil diffusion pump. Chamber pressure before deposition was typically in the low 10 μPa (10^{-7} torr) range. The deposition source was a commercial 5-cm-diameter copper hearth electron beam source 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 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. 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.

C. Residual Gas Analysis and Deposition Procedure

Deposition chamber residual gases were monitored with a quadrupole mass spectrometer (UTI 100C, Uthe Technology, Inc.). The outgassing characteristics of the substrate laminate were investigated further and a procedure was developed which permitted the deposition of aluminum films at a pressure of 0.1 mPa (1×10^{-6} torr). A one-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 quadrupole mass spectrometer--about 1 nPa (10^{-11} torr)) at temperatures below 130°C. A low power (250 watt) vacuum bake in the deposition chamber with the quartz radiant heaters for thirty minutes elevated the substrate temperature to 125°C. This resulted in sufficient degassing that, after a short cooling period, deposition could be performed at a system pressure of 0.1 mPa (1 X 10^{-6} torr) or lower.

The evolved sequence for deposition was the following.

1. Bake cleaned substrates (mounted in deposition fixture to facilitate Step 2) at atmosphere for a minimum of one hour at 150°C.
2. Rapidly transfer substrates to deposition system. A room temperature air exposure in excess of five minutes resulted in substantial moisture absorption and excessively long pump-down time. Pump chamber into 0.1 mPa (10^{-6} torr) range.
3. Thirty minute low-power bakeout of substrates to 125°C.
4. Substrate cooldown. This step varied from a minimum of about one minute to approximately eight hours, the time necessary to cool to room temperature.
5. Aluminum was deposited at 6 nm/s (60 Å/s) to a nominal thickness of 10 μm. Total deposition time was thus approximately thirty 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.

The chamber pressure rose to about 0.1 mPa (1 X 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 2. 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 H_2, CH_4, CO, and CO_2. 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 gun 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 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 μPa (10^{-7} torr) range by adding a processing step such as electron bombardment degassing or oxygen glow discharge cleaning of the chamber prior to deposition.

RESULTS AND DISCUSSION

A. Thickness Uniformity

The thickness of the aluminum films was determined by a beta backscatter technique developed at the Kansas City Division of Bendix[4] in which nominally 10 μm thick aluminum on Kapton was measured with a precision of ±1 percent. Each part was measured at thirty points distributed across the dome. The uniformity was expressed as the standard deviation of the thirty 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 3. 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° from vertical for all experiments. No systematic variation in film thickness across a dome, such as radial thinning as was seen on non-uniform parts, could be inferred from the thickness measurements when optimum geometrical parameters were chosen. The bar at the left of Figure 3 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 one percent.

B. Reflectance

The film surface reflectance was measured by a laser reflectometer technique reported elsewhere.[5] The total reflectance with a 632.8 nm wavelength source ranged from 81 to 91 percent with the majority of the films at 89 $\pm$1 percent. These results are comparable to those reported for freshly evaporated[3,6,7] and sputter-deposited[8,9] films in the 0.1 μm (1000 Å) thickness range. The amount of reflected light that was specularly reflected was generally in the 85 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 4; there are only a small number of surface roughening features such as hillocks[10,11,12] 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 deposition pressure. 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 2. When air was leaked into the chamber to maintain a pressure of 1 mPa (1×10^{-5} torr) during deposition, the total reflectance decreased only a few percent but the specular reflectance decreased to the 25 percent range (see Ref. 5).

C. 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 all films, including those deposited at 1 mPa (1×10^{-5} torr), was within 10 percent of that of bulk aluminum (i.e., less than 3 $\mu\Omega$-cm at 20°C). This result is consistent with previous work[1] in which the critical ratio of total system pressure to deposition rate at which film resistivity increased was 4 mPa s/nm (3×10^{-6} torr s/Å). The ratio for the high pressure deposit in this study is 0.2 mPa s/nm (2×10^{-7} torr s/Å). This tolerance of resistivity to higher pressure is also in agreement with previous investigations[10,13,14] in which the effects of high partial pressures of H_2O, O_2, and N_2 during deposition were considered. It should be noted that, in the case of the air leak, the oxygen partial pressure as measured by the mass spectrometer mounted

on the deposition chamber was reduced by a factor of 20 during aluminum deposition.

The sheet resistance of one dome was measured at the 30 beta backscatter thickness determination positions; all values were ±2 percent of the average, and, expressed in the same manner as the beta backscatter results (standard deviation of 30 values divided by average sheet resistance), the uniformity of sheet resistance was 0.8 percent. The thickness uniformity of this film was measured to be 0.9 percent.

D. Fiber Texture

Several segments of deposits, taken both near the dome center and toward the outer skirt, were analyzed with a Norelco pole figure device using Cu K_α radiation. The crystallographic orientation of all deposits showed a very strong fiber texture with (111) parallel to the substrate as illustrated in Figure 5. Vacuum deposited face-centered cubic (FCC) metals often exhibit a (111) fiber texture.[15] Figure 6 is a plot of X-ray intensity vs 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° between (111) planes in FCC metals.

SUMMARY

A process has been developed for the vacuum-deposition of 10-μm-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, and a resistivity near that of bulk aluminum were obtained. In addition, no films subjected to

snap-pull tape adhesion testing failed. 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 H_2O, O_2, and possibly N_2 along with relatively low substrate temperatures contribute to this effect. The films exhibited a high degree of preferred orientation with (111) parallel to the substrate.

ACKNOWLEDGEMENTS

The author would like to thank P. E. Smith for his experimental assistance, C. J. Cook for TGA and MS analysis of substrate materials, R. V. Heckman and R. J. Russell for film thickness measurements, L. D. Wonnell for setting up the laser reflectometer, E. L. Hollingsworth for the scanning-electron micrograph, J. B. Mustapha for four-point probe measurements, and D. E. Lacey and D. M. Jarboe for X-ray analysis.

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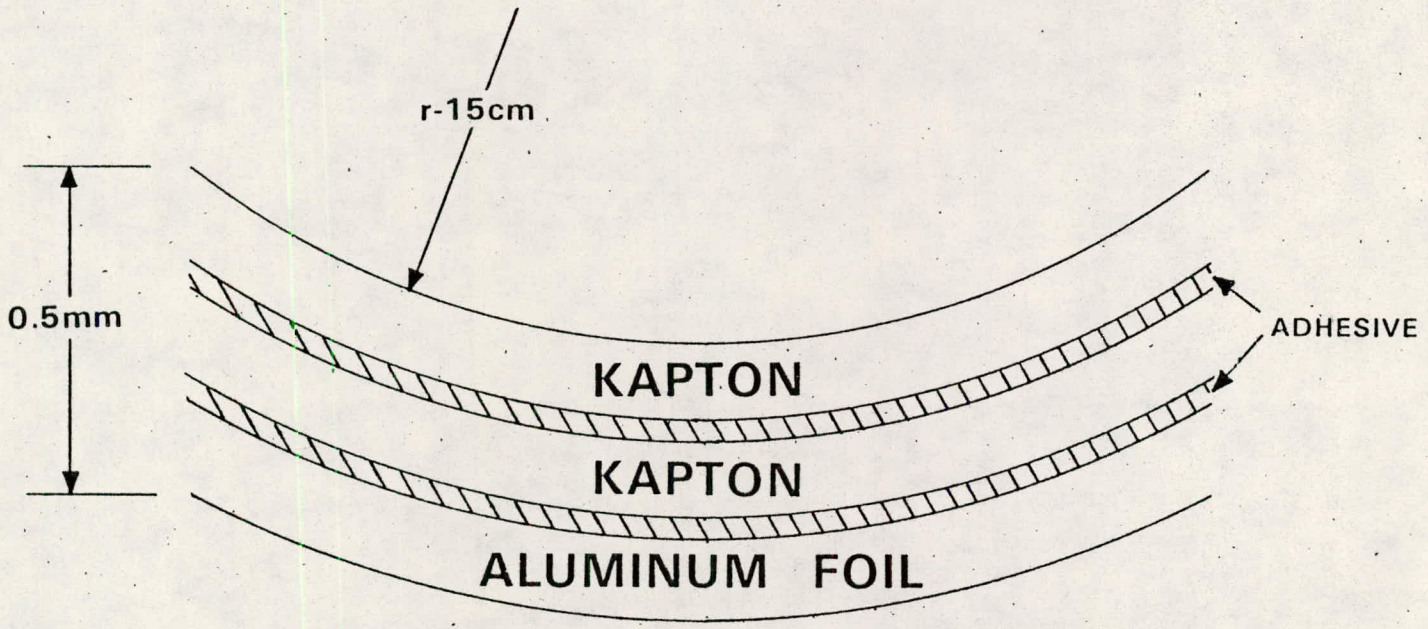
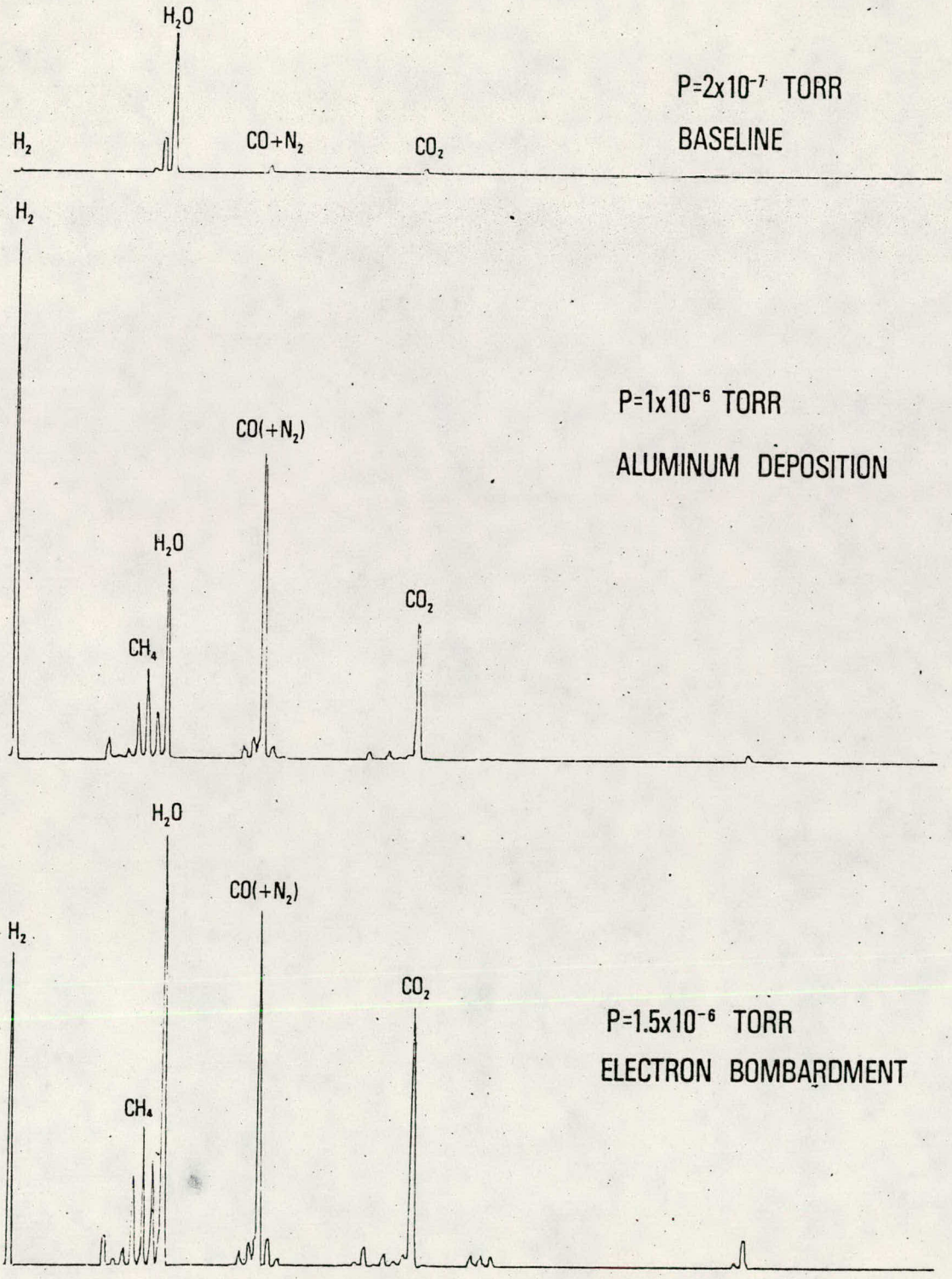
FIGURE CAPTIONS

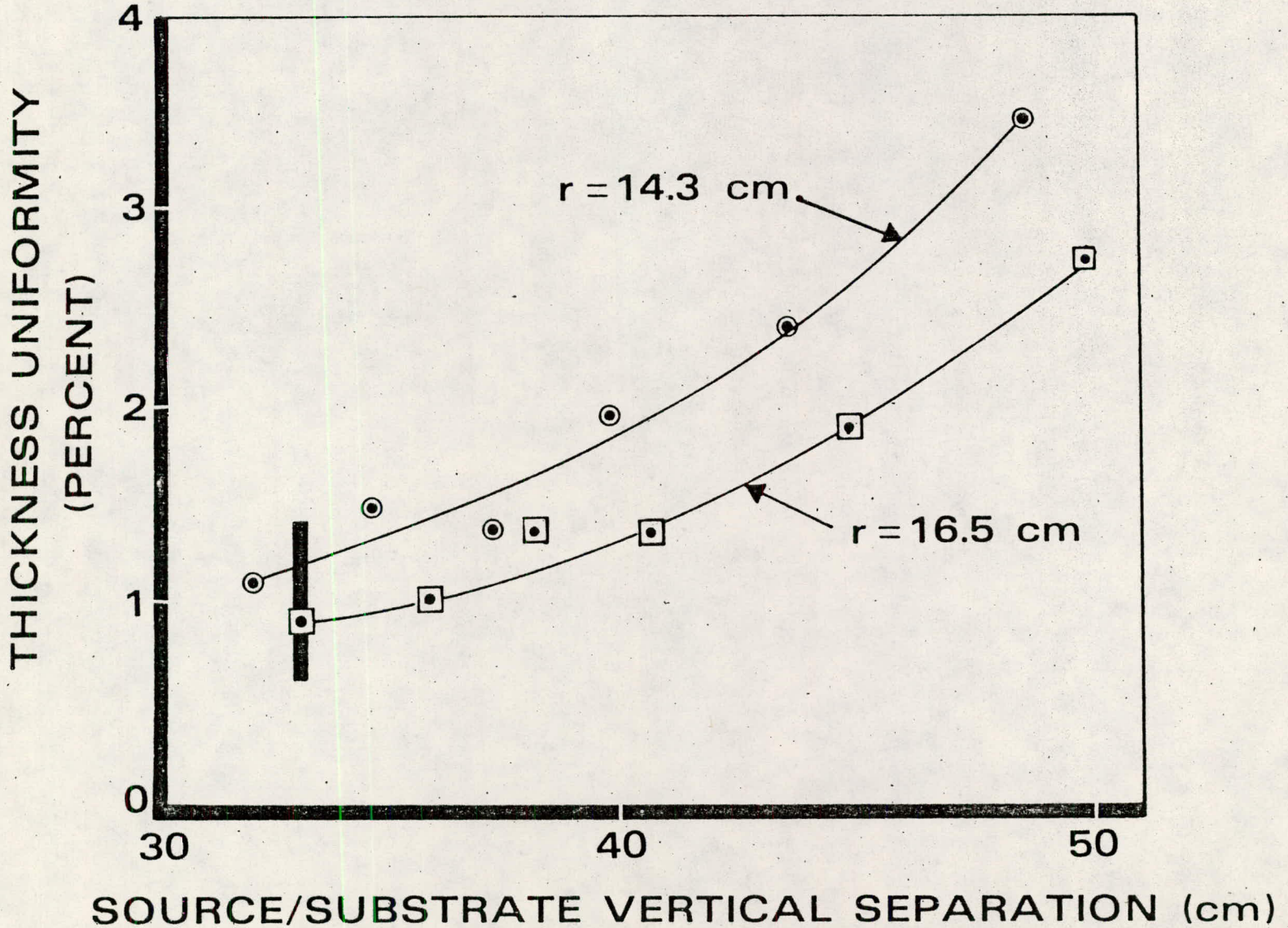
1. Schematic cross section of substrate laminate.
2. Residual gas spectra of deposition chamber after substrate bakeout and before deposition (top), during aluminum deposition onto Kapton laminates at 6 nm/s (center), and during electron bombardment of chamber walls and fixturing by auxiliary filament with evaporator electron emitter off (bottom). 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) and some of the other smaller peaks are attributed to backstreamed pump fluids. All spectra were taken at the same mass spectrometer sensitivity.
3. Film thickness uniformity vs 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 75 domed parts deposited at that position.
4. SEM micrograph of the surface of a typical 10 μm thick deposit.
5. (1$\bar{1}$1) X-ray pole figure of typical 10 μm thick aluminum film on Kapton. The shaded areas denote those regions where the X-ray intensity was above background.
6. (1$\bar{1}$1) X-ray intensity vs angle from the sample normal for a typical 10 μm thick aluminum film on Kapton. This corresponds to a plot of intensity vs radial distance in the pole figure of Figure 5.

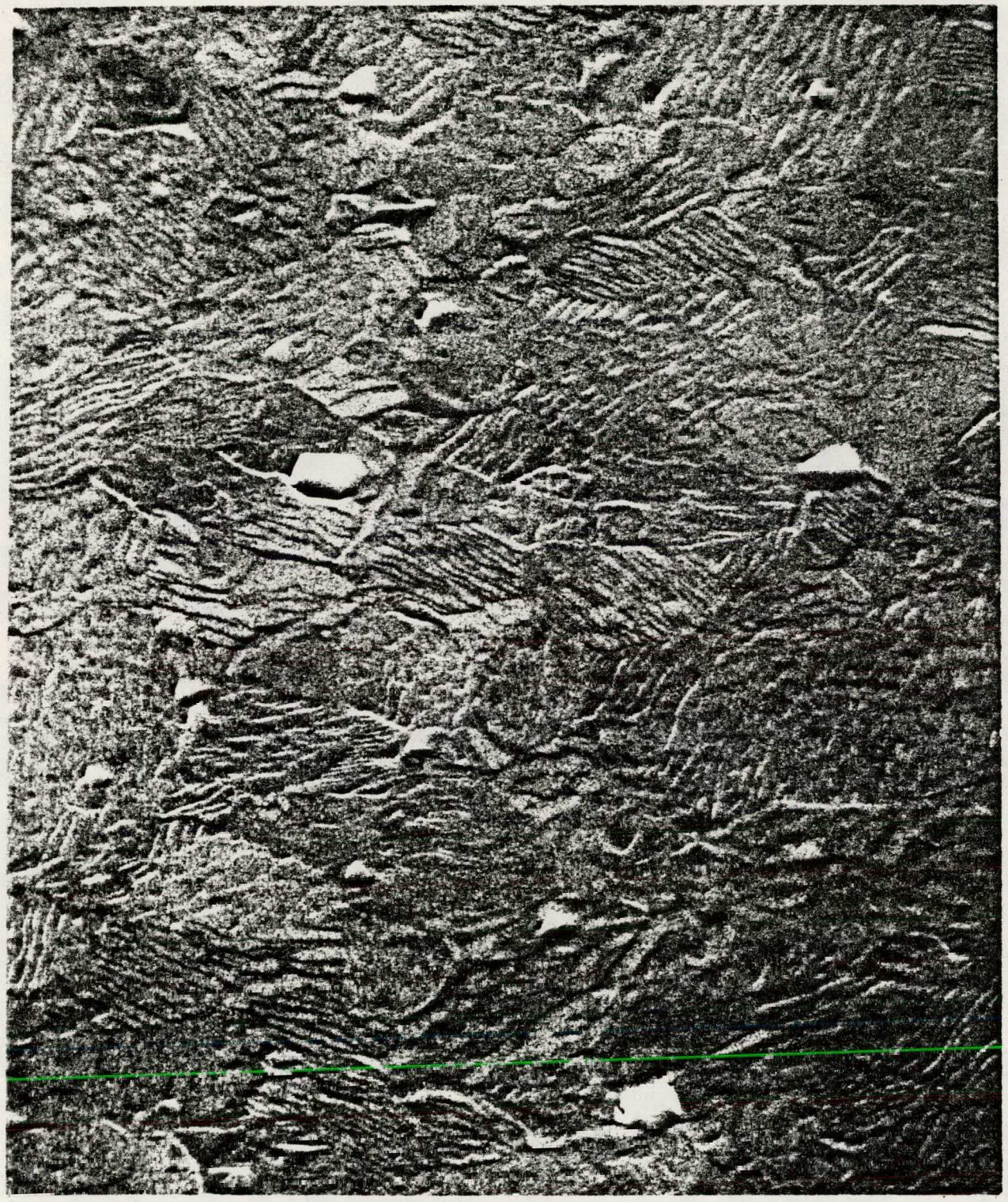
SUBSTRATE CROSS SECTION




H_2O
H_2
$CO+N_2$
CO_2
P=2x10⁻⁷ TORR
BASELINE
H_2
$CO(+N_2)$
H_2O
CO_2
CH_4
P=1x10⁻⁶ TORR
ALUMINUM DEPOSITION
H_2O
$CO(+N_2)$
H_2
CO_2
CH_4
P=1.5x10⁻⁶ TORR
ELECTRON BOMBARDMENT

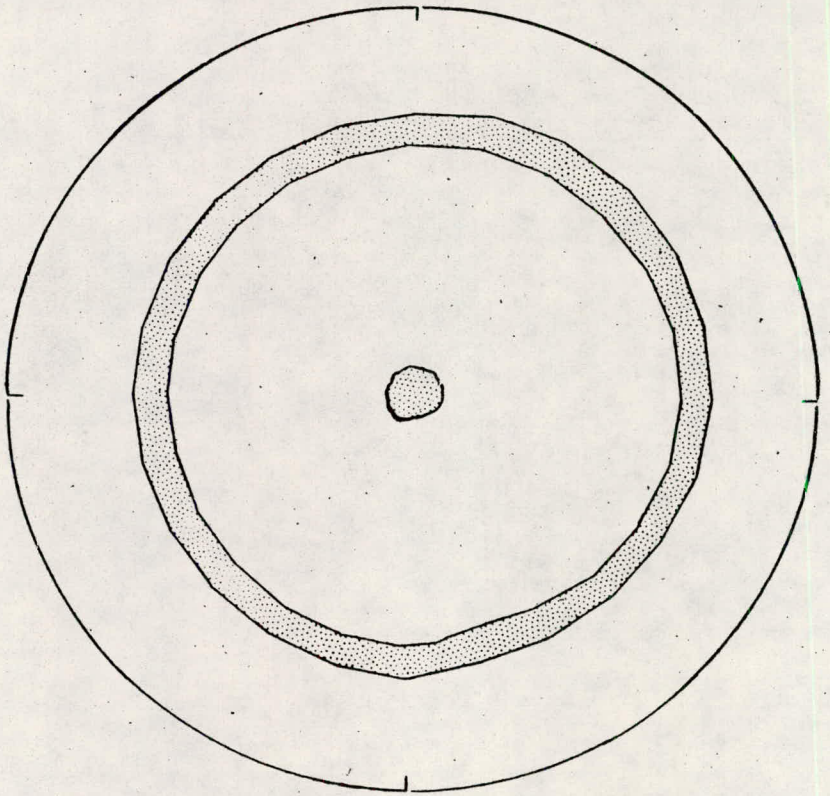
ALUMINUM THICKNESS UNIFORMITY VS SOURCE/SUBSTRATE GEOMETRY





10 μm

(111) X-RAY POLE FIGURE
ALUMINUM ON KAPTON



(111) X-RAY INTENSITY VS. ANGLE ALUMINUM ON KAPTON