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MASTER

A CONDUCTANCE DEVICE FOR MONITORING
THIN FILM GROWTH

by

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CONTRACT AT(29-1)-1106

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ABSTRACT

This report describes a device which can be used to measure the very low resistances (0.02 ohms to 10,000 ohms) of evaporated metal films. This information will be of use to persons involved in coating processes where the thickness of the metal being deposited must be controlled while the substrate is in the vacuum chamber. Accuracies in the range of ± 2 percent are obtained on aluminum in the 200 to 3000 Å thickness range.

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INTRODUCTION

A device capable of measuring the thickness of metal coatings on various substrates was needed. Most devices used for this type of measurement require that the vacuum be broken before the substrate can be taken out of the chamber to measure the thickness of the coating. This process is time consuming if vacuum conditions have to be restored to apply additional film thickness on the substrate.

The device described in this report furnishes the operator with an instantaneous reading during the coating process. Film thickness is displayed in terms of resistance on a wide view meter. Deposition onto a substrate is controlled by a shutter and proceeds to a predetermined resistance value. The calibration curve for a given metal thickness versus resistance is plotted from multiple beam interferometry values.

SUMMARY AND CONCLUSIONS

1. This measuring device is very simple to fabricate and operates satisfactorily throughout the range of 200 Å to 3000 Å for aluminum. Since the rate of rise of resistance when the evaporation starts is extremely fast, the meter pointer cannot be followed accurately; hence measurements below 200 Å are not recommended with this instrument.
2. The detecting element is small enough so that it can be mounted in a vacuum chamber with little difficulty. It can also be positioned on a revolving platform with sliding contacts if a substrate is to be rotated.
3. The sensing unit can be used repeatedly with aluminum without detrimental effects or changes in its original condition. Cleaning of the unit requires about 2 minutes before it can be reinserted into the vacuum chamber for another measurement.
4. Outgassing of the internal components is negligible since most parts are metallic.

5. The rate of aluminum resistance change in the 200 Å to 3000 Å range is very slow. This allows an operator to stop an evaporation by activating a shutter between the source and the substrate without a large degree of thickness over-run.
6. The device may be used for most evaporated metals after plot of a suitable calibration curve under similar operating conditions. Changes of substrate to source distance, vapor incidence angle and substrate temperature should be avoided because all of these factors affect the film resistance.
7. Substitution of glass detection strips with evaporated copper contacts will allow bakeout of the detectors and reduces the step height at the contact ends of the monitor strips.
8. This device differs from a simple ohmmeter and derives its accuracy from the fact that the monitor strip is inserted in the circuit as a meter shunt rather than in series with the meter shunt as is done in an ohmmeter.

DESIGN AND CONSTRUCTION

Metering Circuit

The resistance measuring circuit (Figure 1) consists of a 100 microampere meter in series with a current-limiting resistor and a 3-volt dry cell. A meter with a large viewing area was chosen to facilitate its reading. The resistors comprising the resistivity range were selected so that full-scale reading would be obtained with the largest resistor in the circuit. As the reading in the viewing meter goes down, additional sensitivity can be obtained by turning the range knob to a higher range. It is important that switching to lower resistance values be made only during an actual film thickness measurement. Severe damage to the meter movement can result if a higher sensitivity setting is made without a load at the measuring terminals. The switch range is constructed so that operation will be made from a less sensitive to a more sensitive position. An Off position is also included so that the life of the battery may be prolonged

Figure 1. RESISTANCE MEASURING CIRCUIT

while the instrument is not in use.

The current drain in the battery is such that 100 microamperes would be drawn from the source if the instrument were to be left On when not in use. The least sensitive position (0-1000 ohms) would be the one most likely to be left On because the meter would read full-scale in this position. Absence of a load in all other positions would cause an out-of-scale deflection on the meter movement with possible damage to the meter. The life expectancy of the battery could well be considered shelf-life because the current drain is so small.

The voltage source consists of two 1-1/2 volt Type-D flashlight cells which can be mounted within the 6x5x4 in. Minibox* enclosing the meter and other parts. Heavy duty cells such as No. 6 ignition dry cells are more desirable for this application; however, external mounting must be provided because of their physical size. Resistor values for the range switch were chosen so that a one ohm span could be obtained for the most sensitive position. The other three positions were 10 ohms, 100 ohms, and 1000 ohms, respectively (see App. 1 for calculations pertaining to the selection of these resistor values). Two terminal posts (banana jacks) were mounted on the back panel of the Minibox. Connection of the resistance sensing unit, to be placed within the vacuum chamber, was made to the terminals mounted at the back plate.

* Trademark of Bud Radio Corporation.

Resistance Sensing Units

The resistance sensing units are cut from a sheet of non-sensitized printed circuit board 1/16 in. thick containing a thin layer of copper on one surface. A strip, 1-1/4 in. by 1/4 in., was etched clean for 3/4 in. in the center, leaving 1/4 in. copper contacts on each end. Etching was accomplished by dipping the ends in lacquer and then immersing the strip in diluted nitric acid solution. Two to 3 minutes are required to etch the exposed copper away, leaving only the circuit board surface between two conductive ends. The lacquer was then removed with acetone. Sensing strips used to monitor aluminum evaporation can be reused after cleaning with dilute NaOH solution. An example of the results obtained from a reused sensing unit is shown in Table I.

TABLE I
RESISTANCE MEASUREMENTS FOR 65 mg Al

Unit No.	Initial Run	Repeat with Recleaned Units
1	4.00	3.77
2	4.15	4.50
3	4.85	3.42
4	4.75	4.48
5	3.72	4.40
6	4.25	4.85
7	3.23	3.25
8	4.25	3.95
Avg.	4.15	4.08
Std. Dev.	.52	.57

The resistance units are held in position adjacent to a substrate by two alligator clips; however, other mounting methods are possible. Slip-in contacts may be preferable for some applications.

Frosted glass strips with evaporated copper ends may be substituted for Bakelite* sensing strips in instances where the measuring head is located in a bakeable system (Figure 2). Other non-conducting materials with low vapor pressures and low porosity may also be used.

* Trademark of Union Carbide Corporation

Figure 2. DETECTION ELEMENT

The sensing strips are connected to the metering device through any usual high-vacuum electrical feed-through. Lead wires within the system should be coated with Teflon* TFE to reduce outgassing. Ceramic coated wires may be used in bakeable systems. The lead wires should be as short as possible to reduce their resistance.

A precision resistor should be substituted for the sensing strips to check instrument operation prior to its use. This will insure the instrument is operating properly.

DISCUSSION

Instrument Operation

An ohmmeter suffers from inverse-square-law characteristics and loss of accuracy when measuring less than 50-ohms resistance. This instrument eliminates these drawbacks.

* Trademark of E. I. du Pont de Nemours & Co., Inc.

The resistance monitor described in this report has very good linearity for high resistance values (3 ohms or higher) and gradually deviates from linearity for resistance below 3 ohms. Additional linearity can be obtained using a meter movement with low internal resistance. The high sensitivity scale covers one ohm of resistance for full-scale deflection; therefore, increments as low as 0.02 ohms can easily be detected. Higher scales are convenient during the early part of a film deposition where the resistance varies from an infinite resistance to a relatively low value (10 ohms or less).

The resistance sensing unit, which is the key to this type of measurement, can be considered as a shunt for the meter movement. The resistance in this sensing unit gradually decreases as the metallic film is deposited on the non-conductive strip. Since this conductive film is essentially connected across the 100-microampere meter, a greater shunting effect will take place as the thickness of the coating increases. The readings obtained from the microammeter are in units of resistance and not of thickness; therefore, a conversion should be made to arrive at the desired answer.

Calibration

Calibration charts can be prepared by recording the resistance values obtained with this instrument and then measuring the coating thickness by some other method such as multiple beam interferometry (Table II and Figure 3). The sensing units are placed inside the vacuum chamber facing the aluminum source and adjacent to the substrate. Several sensing units are coated to different thicknesses--noting the final resistance reading on the meter. Film thicknesses are then measured by interferometry and the readings correlated. In this manner, sufficient data is obtained for a plot. Film thickness can then be made by reference to a desired resistance value. Thickness is controlled by use of a shutter between the evaporation source and substrate. Because of the difference in conductivity in various metals, calibration charts should be made up for each metal. Widely varying deposition rates for the same metal will also produce a change in the calibration curve because of a change in nucleation characteristics of the metal film.

TABLE II

RESISTANCE MONITOR PERFORMANCE
FOR EVAPORATED ALUMINUM

Thickness in Angstroms*	Measured Res. in Ohms
100	425
146	120
229	62
288	29
364	19.7
510	10
809	5.72
930	4.78
1774	1.98
2139	1.60
2542	1.36
3097	1.20

*Values obtained by interferometry

Figure 3. RELATIONSHIP BETWEEN ALUMINUM FILM THICKNESS AND MONITOR RESISTANCE.

Instrument Modifications

Automation of this system is accomplished by relay contact meters which energize a solenoid when the correct resistance value is reached. Activation of the solenoid, in turn, activates a shutter mechanism to stop the metal evaporation.

It is usually desirable to monitor the film coating at several locations. This requires additional resistance sensing units and a selector switch installed in the metering box. This selector switch connects the various sensing units to the readout device and thus one can determine whether a uniform film coating has been made.

APPENDIX 1

CALCULATIONS FOR SELECTION OF RESISTOR VALUES

Calculation should be started on the least sensitive range (0-1000 ohms).

For full scale deflection on the microammeter, the current drawn from the source should be 100 microamps; therefore, the limiting resistor R should be:

$$R = E/I \quad \text{Where: } E = \text{source voltage} \\ I = \text{microamps}$$

$$R = 3/100 \times 10^{-6} \\ R = 3 \times 10^4 \text{ ohms}$$

The internal resistance of the meter is 1050 ohms, thus the exact value for limiting resistor R should be 3×10^4 minus 1050, or 28950 ohms. If a resistor with a 1 percent tolerance were to be used in this application, a 29K ohm resistor would be used with negligible error introduced.

Calculations for the other limiting resistors follow a similar pattern; however, since the resistance ranges vary in factors of ten, the limiting resistors will vary in the same fashion, or:

$$R_2 = 3K \text{ ohms when } I = 1.0 \text{ ma} \\ R_3 = 300 \text{ ohms when } I = 10 \text{ ma} \\ R_4 = 30 \text{ ohms when } I = 100 \text{ ma}$$

The schematic shown in Figure 1 does not include the modified values for the limiting resistors since this instrument was only a prototype model; however, for accurate results, the exact resistor values should be used.