Deposition of Tantalum Films with an Open-Ended Vacuum System

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New devices using vacuum-deposited metal films require a high-speed, low-cost method of vacuum deposition. The capability of the open-ended multiple-chamber deposition equipment has been investigated to determine its suitability for depositing tantalum nitride thin films. This was accomplished by examining the measurable electrical properties of the deposited film and by determining the stability of resistors made from these films.

Tantalum films produced by the open-ended deposition system were found comparable to those produced by many bell-jar systems. It was possible to control the addition of nitrogen to the films, and tantalum nitride films of satisfactory stability were obtained. Because the open-ended deposition method can produce large quantities of suitable thin films, it is expected that this will be an important process in the manufacture of future products.

I. INTRODUCTION

Tantalum thin film circuit techniques developed at Bell Telephone Laboratories[1] can produce resistor and capacitor circuit elements and associated interconnections. Such tantalum film circuits have high stability and good reliability, superior to that of discrete components with their multiple interconnections.[2]

The Western Electric Company has developed a continuous open-ended vacuum system for deposition of these tantalum films. This system provides for the passage of substrates through a sequence of chambers which vary in pressure from atmospheric pressure to high vacuum and then back to atmospheric pressure. The design of this system and the details of its operation have been previously reported.[3]

This open-ended system has advantages for quantity deposition of thin films. All vacuum chambers remain at their operating pressures; no time is lost pumping down prior to deposition. Work chambers need not be exposed to room atmosphere and possible contamination. Degassing and preheating operations can be restricted to the substrates and associated carriers; repeated degassing of the system is unnecessary. Substrate motion is continuous through the system; no operator handling or manipulation is required.

The open-ended deposition process differs in a number of ways from earlier work with batch processes using bell-jar vacuum chambers. Chamber materials and hardware are very different from those developed for round bell-jar enclosures. Substrates move through the sputtering glow zone, continuously passing the cathode. This motion produces thermal gradients which result from the dynamic equilibrium conditions for a given substrate speed. Deposited films are the result of an integration of the effect of each part of the cathode, rather than the result of a static pattern of deposition. Film thickness can be controlled by the length of chamber and the speed of substrate motion as well as by deposition rate.

II. TEST PROCEDURE

To investigate the effects of these changed deposition conditions, the product of the open-ended machine was examined to ascertain whether the films have satisfactory properties, and also to determine that there was no adverse effect on the subsequent processing operations. The evaluation of the quality of film deposition in the open-ended system consisted of the following parts:

First, examination was made of the tantalum film deposited without any intentional nitrogen addition. The properties of tantalum film could be strongly altered by contaminant gases from atmospheric leaks or by outgassing of material in the sputtering chamber. Examination of this tantalum film quality should reveal any inadequate cleaning or adverse effect from the deposition method.

Second, the properties of the films were examined as a function of the amount of nitrogen added to the sputtering atmosphere. This establishes the ability to add sufficient nitrogen to produce useful resistor films, as demonstrated by stability, resistivity, and temperature coefficient measurements.

Third, the reproducibility and control of the tantalum nitride deposition process were examined by repeat depositions at the same operating point, and by the examination of many depositions which deviated only slightly from the operating point for most suitable film properties.

Fourth, an examination was made of uniformity of deposition over the width and length of the substrate.

III. MEASUREMENT PROCEDURE

Satisfactory film quality is judged initially by measuring three film properties: thickness (Å), specific resistivity (ρ), and the temperature coefficient of resistance (α). In order to insure that the variability of film properties is due to the machine processing system and not to errors in the *measurement* of the properties, the test details and procedures were evaluated.

A test pattern was developed to insure that all films would have their properties measured on the same effective area and at the same position on the substrate. The zigzag test pattern for a 1.5-inch by 3-inch substrate is shown in Fig. 1. It consists of 20 resistors with a nominal line width of 0.015 inch, each having a path length of 144 squares. The resistors are interconnected by a center stripe and have separate terminal tabs for each resistor. The test resistors are defined by using silk-screen techniques to apply a resist to a tantalum-coated substrate. The unwanted film is removed by etching.

3.1 *Film Thickness Measurement*

In preparing films for thickness measurements, hot sodium hydroxide is used to remove the unwanted tantalum film without appreciable etch

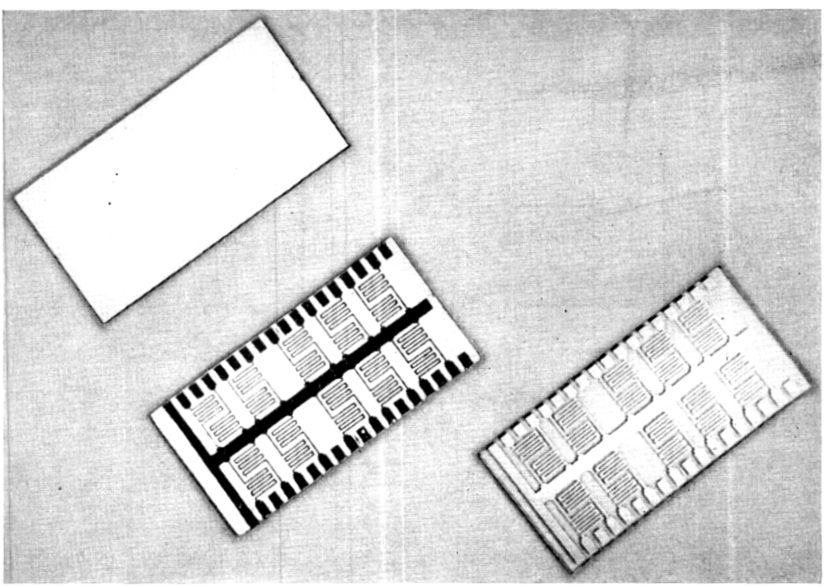

Fig. 1 — Resistor pattern for film property evaluation.

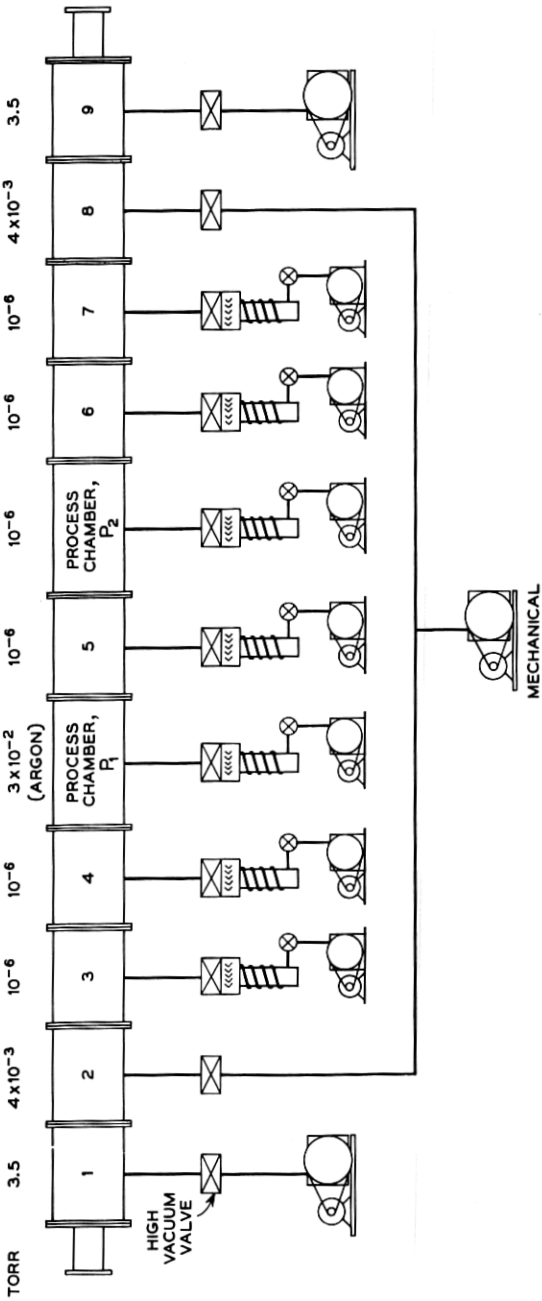

Fig. 2 — Open-ended vacuum system, dynamic operating conditions.

of the glass substrate surface beneath the deposited film. After the resist has been removed, the films are measured using a Talysurf instrument.[4] For thickness measurements of the 1200-Å films deposited in this open-ended vacuum system, the 1σ error of measurement is 56 Å.

3.2 *Specific Resistivity*

The specific resistivity is computed as follows

$$\rho = R_s \text{Å} \times 10^{-2} \text{ microohm-cm}$$

where R_s is sheet resistance in ohms per square and Å is thickness in angstroms.

The sheet resistance of an unetched film is determined by a four-point probe measurement in ohms per square. For convenience, these measurements are made using a simplified direct-reading meter of 1 per cent accuracy.

3.3 *Temperature Coefficient of Resistance Measurement*

After the test resistor pattern has been defined by etching, connections are made to the center stripe and the appropriate tab areas. The resistance is measured at 30°C and at 60°C. The temperature coefficient of resistance is then computed as follows:

$$\text{TCR}(\alpha) = \frac{R_{60} - R_{30}}{R_{30}\Delta T} \times 10^6 \text{ ppm/°C}$$

where R_{30} and R_{60} are in ohms and ΔT is in degrees centigrade. Error of measurement studies indicate a 1σ error of 3 ppm/°C.

IV. ANALYSIS OF UNDOPED TANTALUM FILM

In order to show that the machine process is reproducible at a useful quality level, a series of experiments were run. For this experimental work, one 1.5-inch by 3-inch coated lime glass slide was produced per minute. A carrier 5 inches in length was used to bring the substrate through the chambers. The chamber lengths were such that the carrier and substrate remained in the first four chambers for a total of 15 minutes of high temperature preheating at four decreasing pressure levels. The pressure levels used for this experiment are shown in Fig. 2. Table I gives the preheating power and the sputtering conditions used.

The results of these experiments, shown in Fig. 3, indicate that films deposited in this manner have a specific resistivity of 240 microohm-cm

TABLE I — EXPERIMENTAL OPERATING CONDITIONS

Preheat Stations	#1	#2	#3	#4
Preheat lamp input, watts	300	300	300	220
Sputtering potential, vdc	4500			
Sputtering current, ma	500			
Sputtering pressure, microns (gauge)	32			
Cathode-anode spacing, inches	2.0			
Experimental cathode area, in^2	158			
Deposition rate, Å/min	300			

and a temperature coefficient of resistivity of +56 ppm/°C at a nominal thickness of 1190 Å. The quality of these films is comparable to that obtained by batch processes using bell-jar systems.

4.1 *Process Controllability*

The process controllability for these films was estimated from control charts to have a standard deviation of 11 microohm-cm in specific resistivity and 27 ppm/°C in temperature coefficient of resistance. Film thickness was shown to be controllable, with a standard deviation of 50 Å about a mean of 1190 Å. Based on these results, the process was deemed to be controllable and reproducible for tantalum films.

V. NITROGEN DOPING

Tantalum films without intentional additives are used primarily to make capacitors. Work done by Gerstenberg and Mayer[5] has established that the *resistors* with the best stability were made when one to five per cent of nitrogen is added to the sputtering atmosphere, the amount depending on the pumping and geometry characteristics of the particular system. This nitrogen reacts with the tantalum, and the resulting film contains appreciable tantalum nitride. Having established that the open-ended vacuum deposition system could produce satisfactory tantalum films, it was next necessary to investigate the ability of the system to produce nitrided tantalum resistors with suitable component properties.

The properties of the films of tantalum nitride depend on the environment in the sputtering chamber. Geometry, voltage, current, pressure, gas composition, and gas thru-put all affect the film properties. Slight differences in chamber materials, glow region, gas flow paths, or thermal gradients can also have a major effect on the amount of nitrogen needed to produce film with satisfactory properties. It is customary, therefore, to investigate the relationships between film properties and nitrogen quantity in any new deposition system. This is done by experimentally

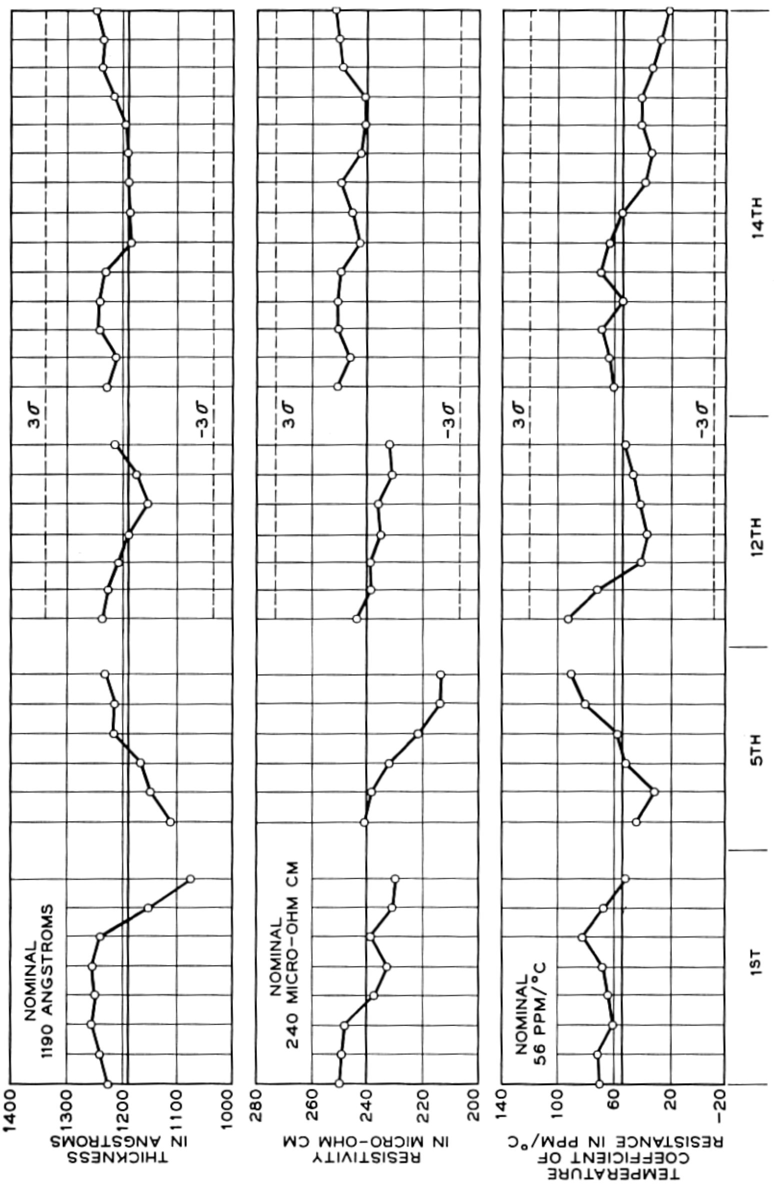

Fig. 3 — Characteristics of tantalum films deposited in the open-ended vacuum system.

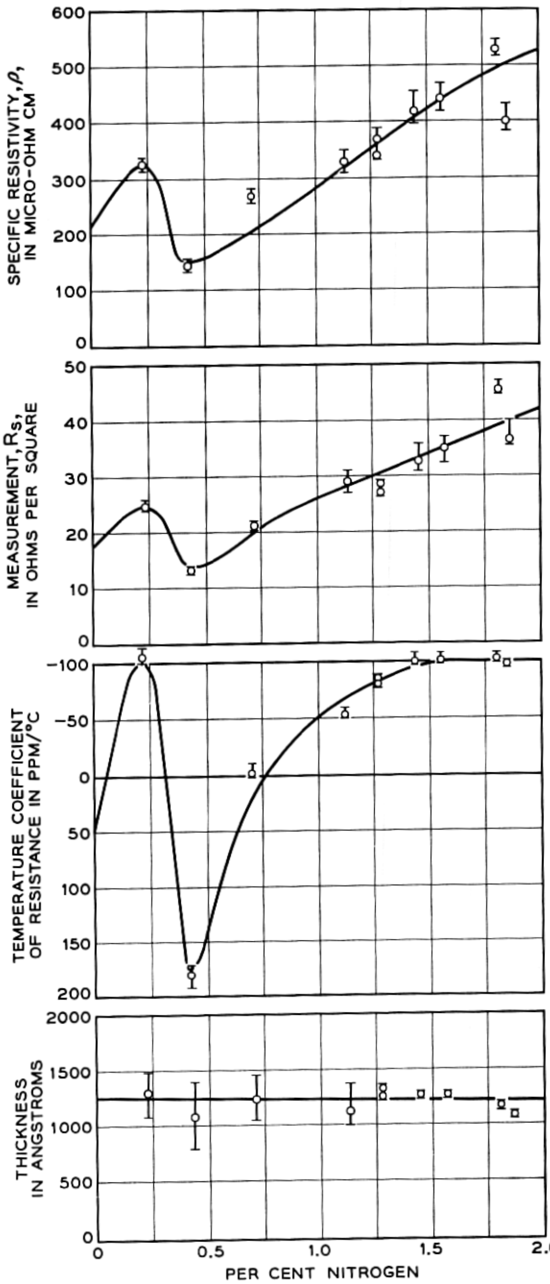

Fig. 4 — Nitrogen-doped product characteristics.

determining the characteristic curve for each of the important nitrogen-film property relationships. These characteristic curves must be determined for each vacuum system, and the proper operating point chosen for each. The influence of trace impurities of nitrogen in the open-ended vacuum system was therefore explored by a series of characterization experiments in the machine processing system. The experimental procedure did not materially differ from that used in the earlier undoped experiments. The operating conditions previously stated in Table I were again held in all cases. The only additive was the controlled flow of nitrogen gas, which was mixed with the argon prior to entering the sputtering chamber.

A single experiment, of the series used for this purpose, consisted of establishing an operating point by adjusting the flow of nitrogen gas until the sheet resistivity was some desired value, and holding it at that value to within ±1 ohm/square. Sample slides were sent through the machine at 10-minute intervals to determine that the sheet resistivity was in control, thus assuring that drifts were removed from the system. Then 20 consecutive slides were given a film deposition in the machine.

Each experimental lot was sampled as follows: four consecutive slides in the center of the lot were processed into resistors; four slides were used to determine the initial film characteristics; and four more were used to examine such physical properties as adhesion, visual defects, and the anodizability of these films. The remaining slides were held as spares for future exploratory studies.

5.1 *Nitrogen-Doped Film Characteristics*

The influence of nitrogen on the characteristics of these resistors after processing is shown by the curves in Fig. 4. The data presented here show that doped films from this machine processing system exhibit a characteristic form similar to that previously reported for tantalum nitride films produced in bell-jar systems.[6] Films with low resistivity and high positive temperature coefficient are formed in the vicinity of 0.30 to 0.40 per cent nitrogen.

5.2 *Accelerated Life Test Data*

The ultimate criteria for satisfactory films are the observed qualities of the circuit elements made from the films. Resistors made of tantalum and tantalum nitride should have a stability characteristic of less than 1 per cent drift in resistance in a 20-year lifetime. Accelerated aging tests, used by J. S. Fisher,[7] permit relative judgments to be made much earlier than 20 years — in fact, tests of standard pattern resistors at twice rated load can differentiate between performances of resistors in about 3 months.

The resistor pattern used for accelerated life testing consists of 24 resistors, each rated at 0.5 watt. This resistor pattern is shown in Fig. 5. Twelve resistors are arranged on each side of a common center strip on the 1.5-inch by 3-inch alkali-free glass substrate (Corning Code 7059). Each resistor is formed by a zig-zag pattern of lines 0.008 inch wide, containing 364 squares. The components are defined by using a conventional photo-resist (KMER)* and etched in a hydrofluoric-nitric acid mixture.

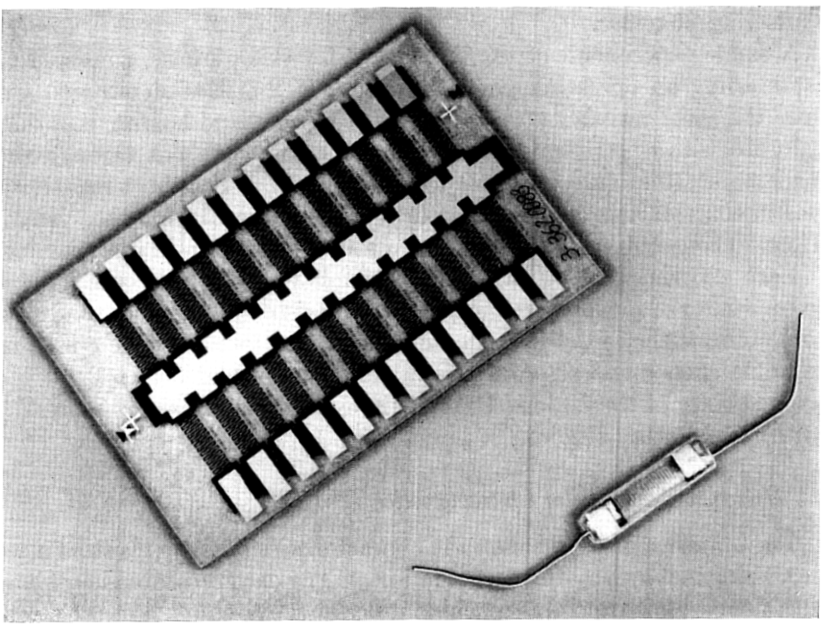

Fig. 5 — Product stability test pattern.

Nichrome and gold are evaporated in turn onto the terminal areas. The films are bath-anodized to 30 volts in citric acid.[8] Oven baking at 250°C in air for five hours is used to stabilize the films. Resistors are then separated into individual units and trim anodized to 15,000 ohms ±1 per cent wherever possible. For initial sheet resistance of greater than 40 ohms/square, it is necessary to trim anodize to a maximum of 20,000 ohms ±1 per cent.

The stability of resistors, for the range of nitrogen additive from 0.0 to 1.84 per cent, was studied by placing eight resistors under double-

* Kodak Metal Etch Resist, Eastman Kodak Company.

rated power life test, four from each of two slides in the center of the lot. This life test consists of a dc power load of one watt in ambient air at 30°C ± 5°C, and corresponds to 40 watts/in² of tantalum film.

The performance of these films under such conditions can be seen in Fig. 6. The stability characteristics change rapidly with slight varia-

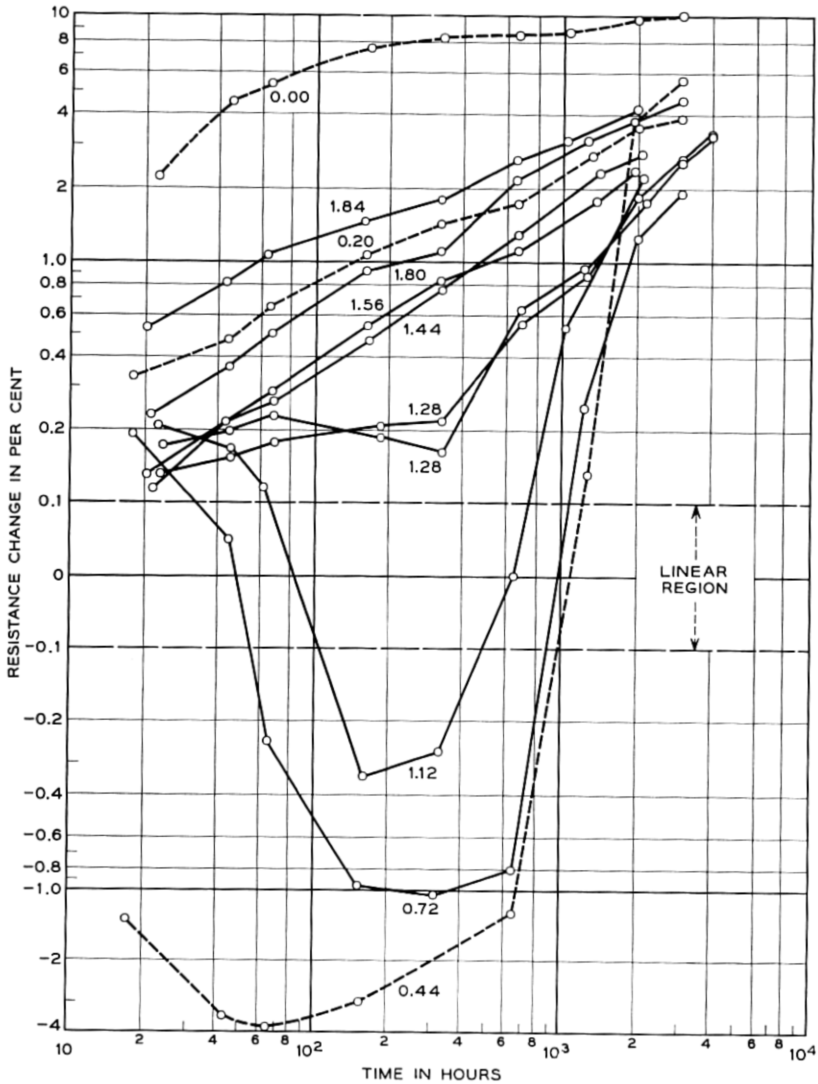

Fig. 6 — Accelerated life test of resistors with 0.0 to 1.84% nitrogen.

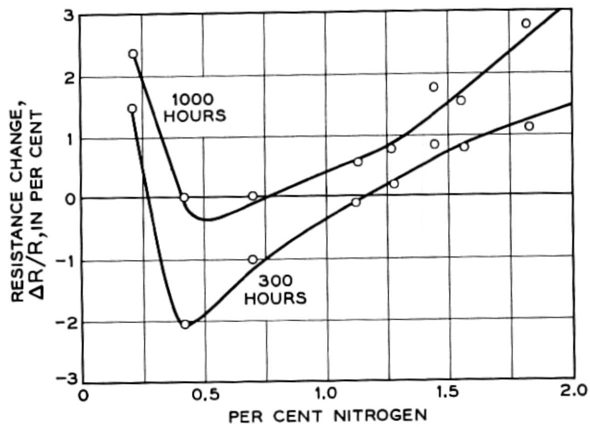

Fig. 7 — Life test time cross section.

tions in amounts of nitrogen doping. The data shown here for resistance change ($\Delta R/R$) were obtained on the same films whose nitrogen doped characteristics prior to life tests were shown in Fig. 4.

The 0.0 per cent nitrogen lot shows almost 9 per cent increase in 1000 hours. The 0.2 per cent nitrogen film appears to be more stable. The 0.44 per cent nitrogen film, at the bottom of the figure, exhibits a decrease in resistance in the first 1000 hours. However, as more nitrogen is added, the decrease in resistance is reduced until it has almost disappeared in the vicinity of 1.56 per cent nitrogen.

These data can be analyzed in a different manner by plotting time cross sections of the data against per cent nitrogen. Fig. 7 shows that this data-display technique produces a curve with the same characteristic form as the tantalum properties previously plotted. The dip in the curve occurs at the same per cent nitrogen for $\Delta R/R$ as it does for the other film properties. This minimum in each property has been previously observed in product produced in bell jars. It is believed that in the vicinity of the dip the product possessed greater metallic purity than at other nitrogen levels.

The films that were made with about 1 per cent nitrogen added to the sputtering atmosphere seem to provide the least total resistance change on this plot. Re-examination of Fig. 6 shows, however, that these films went through a large negative change in resistance before returning to original value. If films with consistent behavior are chosen instead, those with a nitrogen additive of about 1.48 per cent are to be preferred.

When changes in resistor films having 1.44 to 1.56 per cent nitrogen are examined on a log-log plot (as in Fig. 6), the drift behavior is found to be quite linear, with a trend line that can be defined by the equation:

$$\log_{10} \Delta R/R = -3.74 + 0.63 \log_{10} t.$$

This drift rate produces resistance changes at 1000 hours that are comparable to those reported from batch process bell-jar-deposited films.

Many research workers are expending considerable experimental work to establish equivalency of accelerated power aging rates to the aging rate of resistors when used at the more normal power dissipation of 20 watts/in^2. Such work indicates that the 1.48 per cent nitrogen resistors should have an average change of 0.4 per cent in 20 years under normal load. With allowance for the variability of film from run to run, this group of films should be processable into resistors with maximum aging change of less than 1 per cent. Of course, considerably more time must elapse and more correlations must be established before the exact equivalency of normal aging to such accelerated aging can be determined.

VI. NITROGEN DOPED FILM REPRODUCIBILITY

Since nitrogen doping adds a new and major variable to the operating conditions of the machine processing system, experimental runs were made to demonstrate the reproducibility of the doped film properties. Over a typical five-month period, for example, six runs were made at a particular nitrogen level of 1.28 per cent. The machine processing system was adjusted to the standard operating conditions previously mentioned. The average values of the three resistor characteristics α, ρ, and R_s for each run are shown in Table II.

6.1 *Reproducibility of Life Performance*

The stability of tantalum resistors was discussed previously in connection with the characterization curves of Fig. 6. To evaluate the ability

TABLE II — NITROGEN-DOPED FILM REPRODUCIBILITY

Sputtering Date	Temperature Coeff. of Resistance α ppm/°C	Specific Resistivity ρ $\mu\Omega$-cm	Sheet Resistance $R_s \Omega/\square$
10-2	−79	300	25.2
10-25	−81	375	26.6
11-1 A.M.	−82	334	26.5
11-1 P.M.	−87	374	27.9
1-23	−78	392	27.6
2-15	−73	318	28.1
Average	−80	349	27.0
Std. dev.	±5.5	±33	±1.1

(These standard deviations were estimated from the range of the data.)

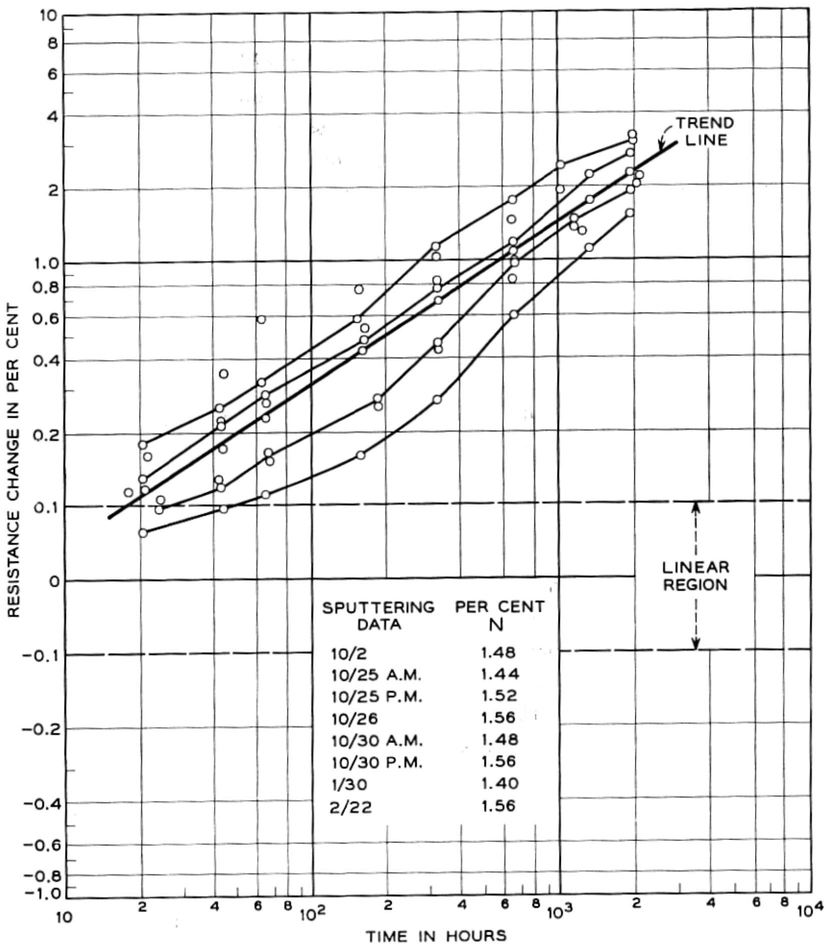

Fig. 8 — Accelerated life test of resistors with 1.40 to 1.56% nitrogen.

of this system to produce films of consistent stability, the aging characteristics of tantalum films with 1.48 ± 0.08 per cent nitrogen were examined. Resistors were processed from 8 separate runs of film having the previously mentioned nitrogen levels. The results of accelerated aging tests of these resistors are shown in Fig. 8. Sufficient power was applied to each resistor to produce a power dissipation of 40 watts per square inch of tantalum area. While there is some spread of resistance change due to the variation in nitrogen content, these resistors do consistently exhibit closely similar aging rates. The difference between films shows up as changes in resistance at the 20-hour measurement.

VII. FILM UNIFORMITY

Post-deposition processing of tantalum films requires that the resistor film be anodized to achieve stability and to adjust the resistance of the film to a required value.[8] Using etch techniques, multiple networks can be produced from a single substrate. Economical processing should be performed on the full substrate area, rather than on an individual resister or network. Economic production of large volumes of stable thin film circuits, then, requires not only that the deposition process produce a high output of film-coated substrates at a low cost, but also that the properties of the deposited films be uniform over the area of the substrate.

The resistance of the tantalum-nitride film produced in the open-ended system has a variation of 5 per cent over an effective length of 2.8 inches (see Fig. 9). This variation is comparable to that of bell-jar product, and makes possible production of resistor networks with a tolerance of ±3.0 per cent on the individual resistors. The resistance variation is not random, but has a definite pattern of higher resistance near the ends of the substrate. Since the substrate moves through the deposition zone at a constant speed, this suggests some effect of the substrate carrier on the film uniformity.

Typical tantalum-nitride film properties from a single open-ended system, under controlled production conditions, may vary 50 microohm-cm in resistivity, 100 Å in thickness, and 20 ppm/°C in temperature coefficient. This variability in film properties does not contribute significantly to the complexity of subsequent processes. However, if film deposi-

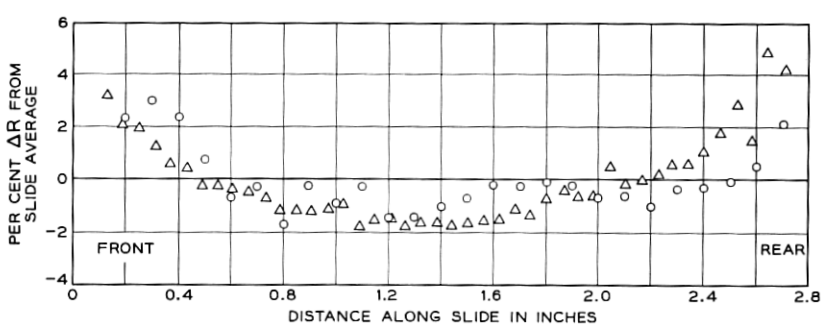

Fig. 9 — Resistance profile, two slides with 1.14% nitrogen.

tion is accomplished by using the larger number of bell-jar systems which would be required to meet the same production demand, the film properties would be influenced not only by the variability of a single chamber, but also by the chamber-to-chamber variability of the associated bell-jar systems. Compensation for this total variability will significantly influence the complexity and even the design of some of the subsequent process equipment and hence the over-all manufacturing cost of thin film resistor networks. The use of the open-end system to deposit tantalum should simplify quantity manufacture and reduce costs significantly.

VIII. CONCLUSION

At the present stage of the developmental work, it can be concluded that the open-ended in-line vacuum concept can be used to deposit large quantities of tantalum for thin film resistors. Each machine can coat two 5-inch by 5-inch substrates per minute. One such machine, on one-shift operation, can therefore produce approximately 4,000,000 square inches of metal film per year. Such films have exhibited the required stability, uniformity and reproducibility. Further work is in progress to optimize film characteristics. The work to date has established the feasibility of manufacturing production using this new deposition concept.

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