

- [54] **TANTALUM THIN FILM RESISTORS BY REACTIVE EVAPORATION**
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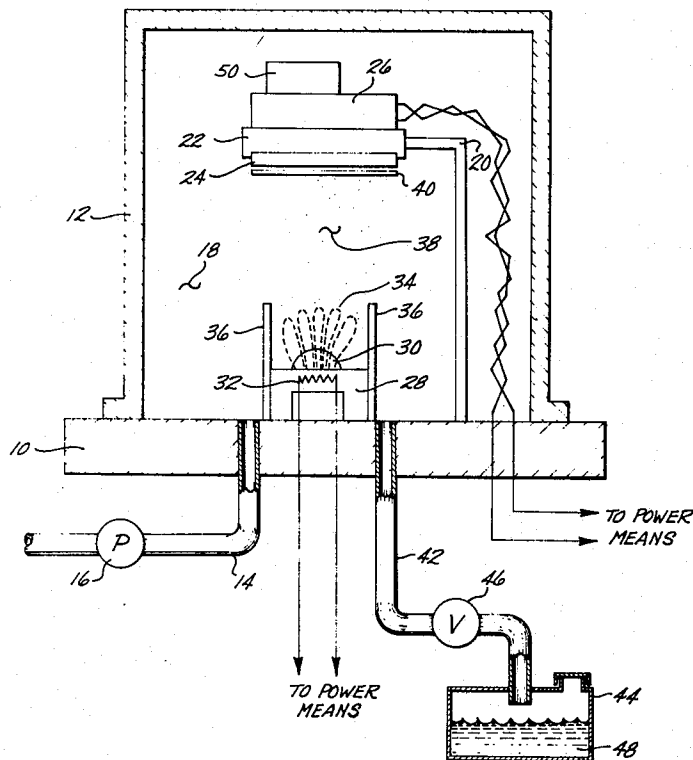
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- [58] Field of Search 252/63.5, 512; 117/201, 117/107, 227, 119; 204/192; 338/308

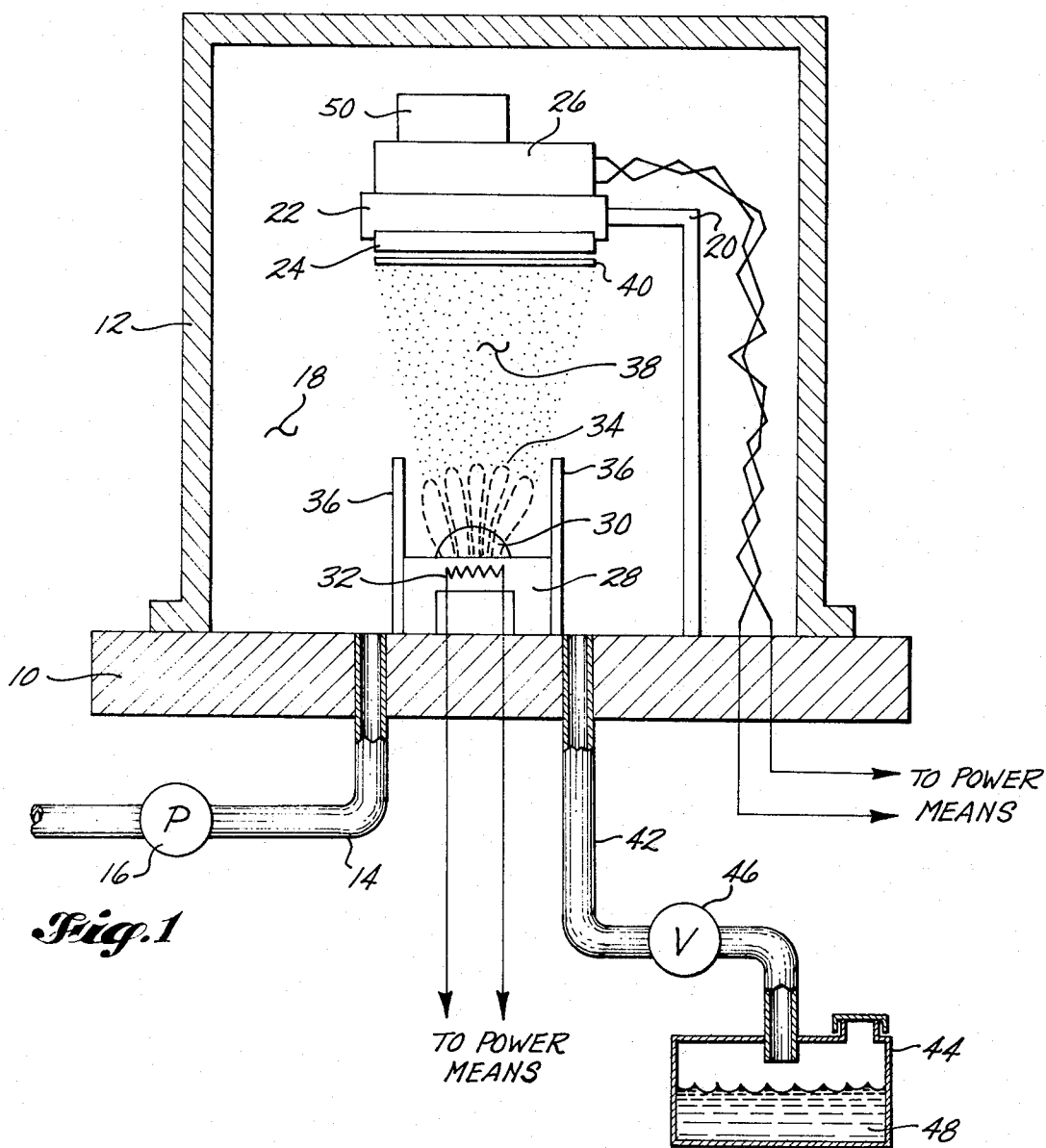
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[57] ABSTRACT

In a process of forming tantalum thin film resistors on a substrate by reactive evaporation, the present invention discloses a method for controlling product repeatability to close tolerances by controlling the substrate temperature and substituting a high water partial pressure for standard inert and reactive gaseous environments.

1 Claim, 5 Drawing Figures





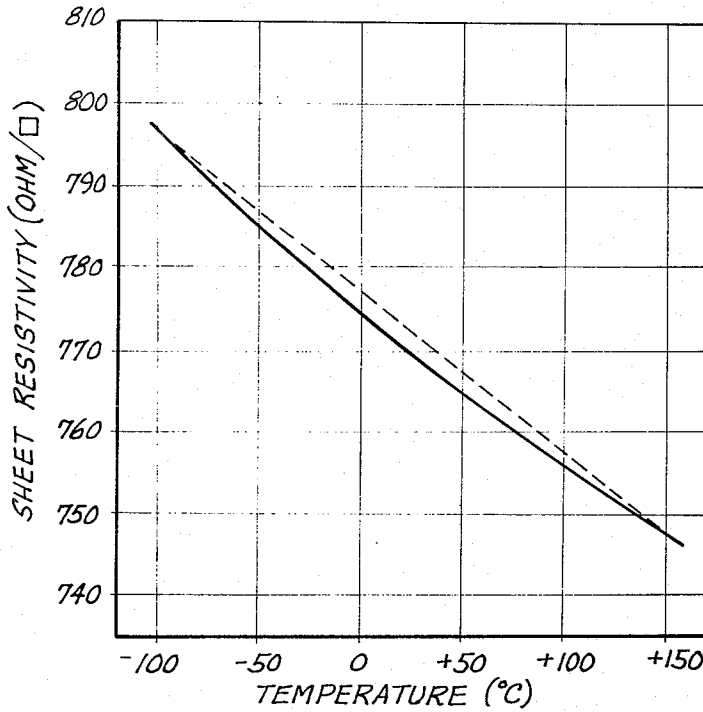


Fig. 2 SHEET RESISTIVITY VS. TEMPERATURE FOR A TYPICAL STABILIZED TANTALUM RESISTOR

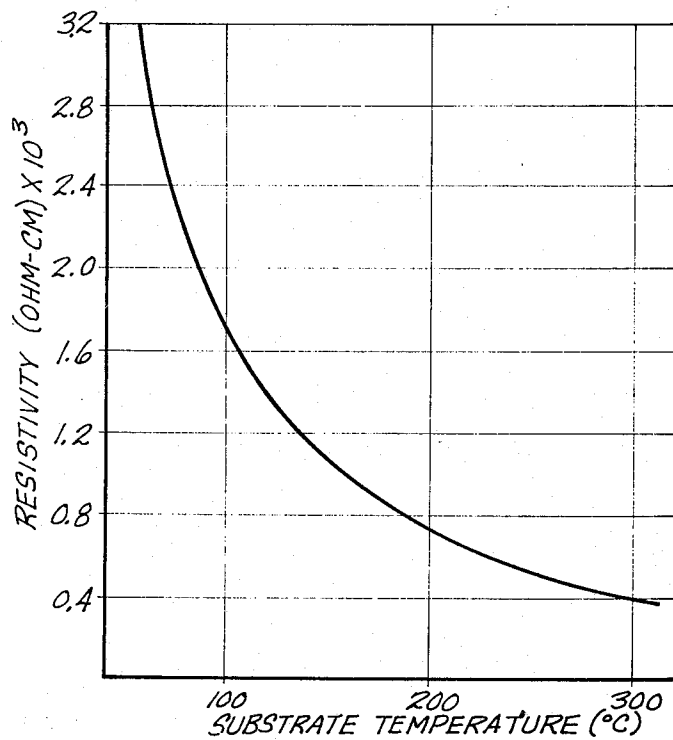
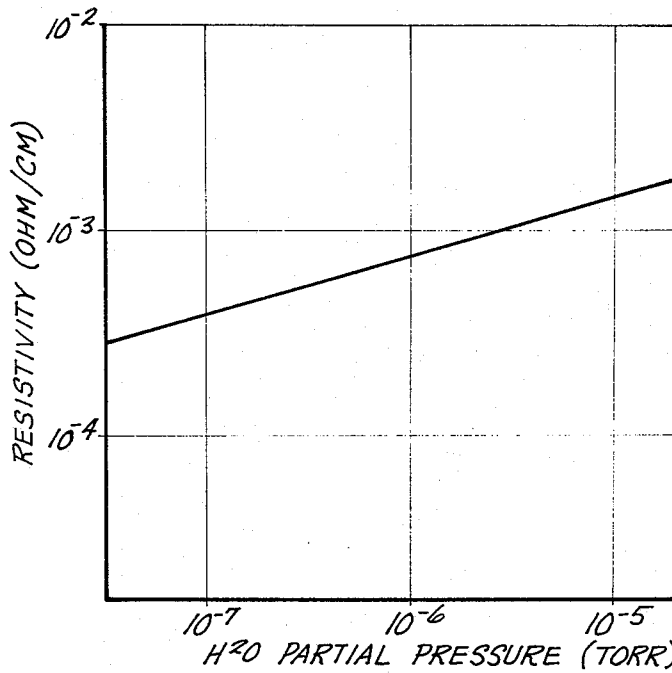
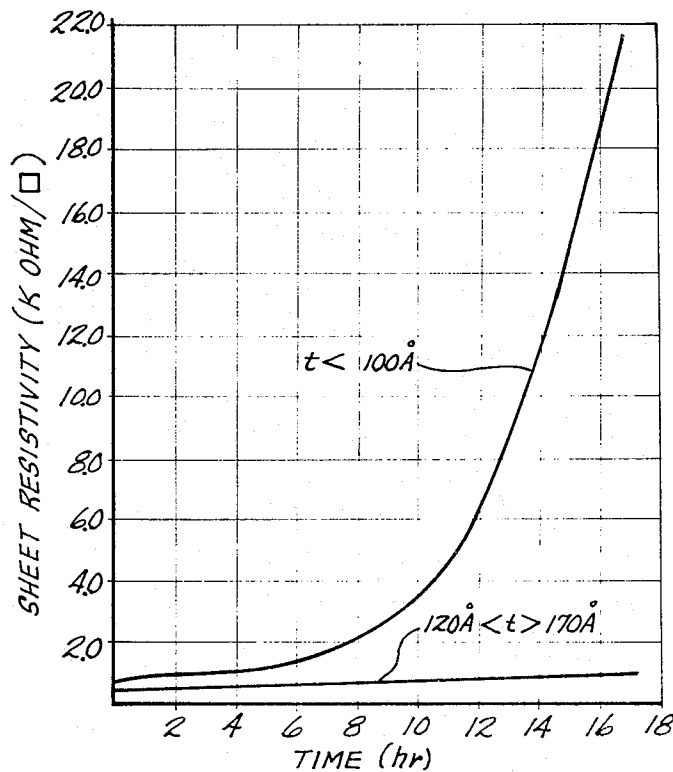


Fig. 3 AS DEPOSITED RESISTIVITY VS. SUBSTRATE TEMPERATURE AT 1×10^{-5} TORR H₂O FOR TANTALUM



AS DEPOSITED RESISTIVITY VS. WATER PARTIAL PRESSURE WITH A 100°C SUBSTRATE TEMPERATURE FOR TANTALUM

Fig. 4



SHEET RESISTIVITY VS. TIME IN AIR AT 300°C FOR TANTALUM RESISTORS

Fig. 5

TANTALUM THIN FILM RESISTORS BY REACTIVE EVAPORATION

BACKGROUND OF THE INVENTION

1. Field of the Invention

Relates to formation of thin film electronic components on substrates and more particularly to the formation of tantalum thin film resistors by reactive evaporation.

2. Description of the Prior Art

Thin film resistors based upon tantalum and tantalum compounds have been extensively applied in the fabrication of high quality thin film hybrid circuits because of their stability, relatively wide sheet resistivity range, and low temperature coefficient of resistance (TCR). These resistors have been formed by both vacuum deposition and sputtering techniques but sputtering in either an inert or reactive gas environment has been by far the more common process. Following deposition, the resistors have been formed using the subtractive or photolithographic process and then passivated by anodization. Passivation by anodization, however, negates the possibility of trimming with an arc erosion process. The formation of resistors by an additive or thru-mask vacuum evaporation process coupled with subsequent arc erosion trimming techniques produces a product with less finishing work on the film coated substrate than does the subtractive process. Moreover, where thru-mask deposition is required, it must be accomplished by evaporation.

It is, therefore, a primary object of the present invention to provide a deposition and stabilization process for tantalum thin-film resistors which is compatible with thru-mask vacuum evaporation and arc erosion trimming techniques.

It is a further object of the present invention to provide a deposition and stabilization process for tantalum thin-film resistors which allows a high degree of repeatability in the performance characteristics of the resistors thus formed.

It is yet another object of the present invention to provide a deposition and stabilization process for tantalum thin-film resistors in which the control variables are the largest and most easily controlled influences on the electrical properties of the evaporated tantalum films.

It is a final object of the present invention to provide a deposition and stabilization process for tantalum thin-film resistors wherein a thermally grown oxide layer can be used to provide adequate resistor stability while still allowing the resistors to be trimmed by arc erosion.

DESCRIPTION OF THE DRAWINGS

FIG. 1 is a simplified drawing of the equipment used in the process which is the subject of the present invention.

FIG. 2 is a graph depicting sheet resistivity vs temperature for a typical stabilized tantalum resistor.

FIG. 3 is a graph depicting as deposited resistivity vs substrate temperature at 1×10^{-5} torr H_2O .

FIG. 4 is a graph depicting as deposited resistivity vs water partial pressure with a $100^\circ C$ substrate temperature.

FIG. 5 is a graph depicting sheet resistivity vs time in air at $300^\circ C$ for tantalum resistors.

DESCRIPTION AND OPERATION OF THE INVENTION

In typical apparatus for evaporative deposition of tantalum there is a base 10 and a cover 12 capable of an airtight seal between the base 10 and the cover 12 when the cover 12 is placed in position on the base 10. The base 10 is provided with an outlet pipe 14 attached to pump means 16 whereby a vacuum can be drawn in the enclosure 18 formed by base 10 and cover 12.

Within the enclosure 18 is provided a support 20 and holding means 22. A substrate 24 upon which the thin film is to be deposited is held in the proper operative position by the holding means 22 and heated by heating means 26. Also within the enclosure 18 is a crucible 28 into which a tantalum source 30 is placed. In operation, filament 32 is operatively connected to an electrical power means (not shown) which causes electron stream 34 to be emitted from the filament 32. Electron stream 34 is deflected by magnets 36 onto the tantalum source 30 causing the tantalum source 30 to heat to evaporation. The evaporated tantalum vapor 38 from the above described electron gun moves to the surface of the substrate 24 and is deposited on the surface of substrate 24 in those areas not blocked by masking means 40.

In such apparatus to practice the present invention is added an inlet pipe 42 connected to an air tight enclosure 44 through valve means 46. In prior art teaching inlet pipe 42 is connected to a source of reactive gas. In the present invention, enclosure 44 contains water 48. Valve means 46 allows control of the water partial pressure within enclosure 18 while heating control means 50 which is operatively connected to heating means 26 allows control of the temperature of substrate 24.

In tests of the present invention, examples of which follow hereinafter, it was found that the parameters controlling the resultant resistive coating should be selected and controlled according to the formula:

$$(2,120/T_s) + \log (P_{H_2O}/1.1r^*) = B$$

where:

T_s = substrate temperature ($^\circ K$)

P_{H_2O} = water partial pressure (torr)

r^* = tantalum deposition rate (A/sec)

and where:

$-1.5 \leq B \leq -1.1$

$298^\circ K \leq T_s \leq 673^\circ K$

$1 \times 10^{-8} \text{ torr} \leq P_{H_2O} \leq 1 \times 10^{-4} \text{ torr}$

$0.1 \text{ A/sec.} \leq r^* \leq 10 \text{ A/sec.}$

EXAMPLE 1

The first example shows variations and findings without control of water partial pressure in the vacuum enclosure and provides a base for comparison of later controlled results employing the present invention.

The tantalum resistors were deposited from an electron gun vaporization source contained in a liquid nitrogen trapped, oil diffusion pumped vacuum system. The approximate film deposition rate and thickness were controlled and monitored with a water cooled piezoelectric quartz crystal microbalance. The deposition of the tantalum film was terminated after a given sheet resistivity of either 250 or 300 ohm per square had been achieved by an in situ resistance monitor. The substrates were heated with quartz iodine lamps and

temperature controlled by a thermocouple bonded to the back surface of the substrate. Total chamber pressure was determined with a nude ionization gauge mounted on the bell-jar. Later examples employed a residual gas analyzer (RGA) to monitor the various partial pressures in the m/e range from 0 to 50 and to adjust the water leak rate with a needle valve.

Ninety-five tantalum resistors were simultaneously deposited through metal masks onto 5 inch $\times$ 5 inch Corning 7059 glass substrates. Immediately prior to the tantalum deposition, 2,000 Å of S_iO_2 was deposited onto the substrates as an undercoat layer. Aluminum contact films of approximately 800 Å were deposited in the same pump-down cycle to complete the resistors. After deposition and removal from the chamber, the resistance of each device was measured. The substrate was then baked at an elevated temperature to stabilize and passivate the resistors. Following stabilization and return to room temperature, the resistance of each device was again measured. The resistance values were then analyzed by computer. The program calculated the substrate mean, variance, and normalized standard deviation for the resistance values before and after baking and for the drift rates which occurred during baking.

Initially, the tantalum resistors were deposited at a rate of 0.9 Å/sec. onto substrates heated to 100°C. No attempt was made with the deposits to adjust or control the total chamber pressure which always remained in the 5×10^{-6} to 1×10^{-5} torr range during the tantalum evaporation. Considerable variability in the tantalum resistivity was evident in the depositions. To achieve a deposited sheet resistivity of 300 ohms per square, the film thickness varied from less than 100 Å to about 170 Å. The effect of this uncontrolled thickness variation became very evident after the stabilization bake at high temperatures. The resistance increase attendant with the bake was found to be a strong function of the deposited film thickness. For example, after a one-hour air bake at 300°C, the thinner films, i.e., those less than 100 Å were either electrically open or very high valued while films thicker than 120 Å increased only 50 to 100% in value.

Upon investigation of the factor attendant to the production of the above resistors, it was discovered that: (1) the major chamber residual during a typical tantalum deposit was hydrogen but, this residual had little effect on the film electrical properties; and, (2) the water partial pressure and not O_2 or CO_2 was responsible for the variation in the deposited film resistivity.

EXAMPLE 2

As a result of the discovery of the effect of water partial pressure on film electrical properties, a water leak was added to the resistor deposition chamber and the water partial pressure controlled during the tantalum deposition using the RGA to monitor the m/e 18 peak. In an effort to avoid annealing resistance changes during the stabilization bake from complicating the experimental results, the deposition substrate temperature for the water residual studies was increased to 300°C. At this temperature and a deposition rate of 0.9 Å/sec., it was discovered that a water partial pressure of 1×10^{-5} torr resulted in tantalum films of the thickness and resistivity range required for satisfactory baking-stabilization, i.e., 120 – 170 Å for as-deposited films of 300 ohms per square.

The resistivity of films deposited using these deposition parameters was found to be much improved in reproducibility and predictability. Approximately 900 resistors formed on 11 different substrates averaged 680 ohms per square after a stabilization air bake. The mean normalized standard deviation for resistors on a substrate was 0.13 while the same parameter for the 11 different substrates was 0.15. Thus, the spread in resistance over the large substrate area was only slightly less than the spread between substrates. When extreme care was taken to avoid partial shadowing of the vapor beam during deposition the normalized standard deviation for a substrate dropped to the 0.07 to 0.10 range.

The TCR of these resistors was measured over the temperature range of -100°C to $+150^\circ\text{C}$. The temperature sensitivity of a typical film is shown in FIG. 2. It can be seen that a linear temperature dependence is obtained only in the high temperature region ($+50^\circ$ to $+150^\circ\text{C}$). At the lower temperatures the resistance change with temperature becomes greater than a linear rate. However, since the deviation from a linear dependence was small, the TCR's were calculated assuming a straight line curve between the two temperature extremes, i.e., the TCR was assumed to be the broken line displayed in FIG. 2. Under this assumption, the average TCR of resistors deposited onto ten different substrates was $-240 \text{ ppm}/^\circ\text{C}$ with a mean standard deviation of $5.7 \text{ ppm}/^\circ\text{C}$ on the individual substrates and $19.2 \text{ ppm}/^\circ\text{C}$ for the ten substrates.

EXAMPLE 3

After the above study of the characteristics of tantalum film deposited at 300°C, the substrate temperature was lowered to see the effect. It was then discovered that the tantalum film resistivity for a fixed water partial pressure of 1×10^{-5} torr and a deposition rate of 0.9 Å/sec. varied greatly with substrate temperature as shown in FIG. 3. As can be seen, the resistivity at a 100°C substrate temperature was about four times that at 300°C and the corresponding TCR was over 600 ppm/°C. The films depicted in FIG. 3 were deposited to a constant sheet resistivity of 300 ohms per square while the film thickness obtained from the quartz crystal microbalance was used to compute the resistivity in ohm-cm.

EXAMPLE 4

The specific dependence of resistivity upon the water residual was then determined by depositing films at a fixed substrate temperature of 100°C, deposition rate of 0.9 Å/sec., and sheet resistivity of 250 ohms per square while varying the water partial pressure as monitored with the RGA. The film thickness necessary to obtain the 250 ohms per square value was again obtained from the microbalance and was then used to calculate the resistivity. FIG. 4 shows the results of these tests and discloses a log-log relationship between the water partial pressure and the resistivity. It can be seen from FIG. 4 that to achieve the same resistivity at a 100°C substrate temperature as at 300°C, the water residual must be reduced by two decades; i.e., from 1×10^{-5} torr to 1×10^{-7} torr. However, since this pressure was much less than the total chamber pressure during a typical tantalum deposition, the use of the RGA was essential for deposition control at the lower substrate temperature.

The resistors deposited using this method were found to be identical to their high substrate temperature - high water partial pressure counterparts.

EXAMPLE 5

Because of the trimming constraints, the resistors produced in the previously described examples were stabilized by thermally growing a very thin oxide passivation film. Typically, this was accomplished with a three-hour air bake at 300°C and resulted in a resistance increase of approximately 100%. The resistance increase was initially very rapid, but after one hour, the resistance increased approximately linearly with time. During the "linear" region, the tantalum oxide grew at the expense of the underlying metallic resistor film. The behavior of the tantalum resistors relative to the stabilization treatment is shown in FIG. 5. Two distinct types of stabilization characteristics are shown. The one is for resistors which were deposited using deposition parameters resulting in film thicknesses in the 120 A to 170 A range while the other typifies resistors fabricated such that the film thickness was less than 100 A. As can be seen, the thin resistors display a greater initial increase in resistance, a higher rate of increase in the linear region, and a runaway characteristic as the oxidation process nears completion. Since all of these traits are undesirable in resistors for precision circuitry, controlling the deposition parameters is seen to be of critical importance.

SUMMARY

In a process for forming tantalum thin-film resistors on glass substrates by thru-mask, vacuum evaporation techniques with a fixed film deposition rate, the most critical parameters are substrate temperature and

water partial pressure during the tantalum deposition. Resistors thus formed have reproducible and predictable electrical properties when these parameters are rigidly controlled. An increase in the substrate temperature causes the tantalum film resistivity to decrease while the water partial pressure has the opposite effect. A high substrate temperature with a high water residual will be equivalent to a low substrate temperature with a low water residual in terms of electrical properties of tantalum film deposited within that environment.

Having thus described the present invention, what is claimed is:

1. In a process for the deposition of tantalum on a substrate by an evaporative process within a substantially evacuated chamber for the purpose of forming resistive elements of electronic circuits upon the substrate, a method of controlling the resistive value and the repeatability of the resistive value of the resistive elements thus formed by controlling the substrate temperature and the residual water partial pressure within the substantially evacuated chamber according to the relationship:

$$(2,120/T_s) + \log (P_{H_2O}/1.1r^*) = B$$

where:

T_s = substrate temperature (°K),
 P_{H_2O} = water partial pressure (torr),
 r^* = tantalum deposition rate (A/sec.), and

$$-1.5 \leq B \leq -1.1$$

and wherein:

- a. $298^\circ\text{K} \leq T_s \leq 673^\circ\text{K}$,
- b. $1 \times 10^{-8} \text{ torr} \leq P_{H_2O} \leq 1 \times 10^{-4} \text{ torr}$, and
- c. $0.1 \text{ A/sec.} \leq r^* \leq 10 \text{ A/sec.}$

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