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THIN FILM RESISTORS OF THE TANTALUM-RHENIUM SYSTEM

by

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SUMMARY

Alloy films of several tantalum-rhenium compositions have been prepared by vacuum deposition, and the electrical properties of these films have been compared with films of the pure metals. This survey study indicates that alloy films containing 50% or more tantalum may find application in the fabrication of hybrid integrated circuits or discrete resistors. The tantalum-rhenium system is of particular interest because the vapor pressures of these two metals are nearly identical in the range for thin film deposition. This indicates that a film stoichiometry could be held nearly constant over a long period of time while evaporating from a single source.

KEY WORDS

thin films, resistor films, tantalum films, rhenium films

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THIN FILM RESISTORS OF THE TANTALUM-RHENIUM SYSTEM

J. R. Rairden

I. INTRODUCTION

Vacuum-deposited films are being used increasingly for the fabrication of resistors for microelectronic applications and for discrete devices. A useful film material must possess a number of properties and characteristics including: (a) a high specific resistivity; (b) a low-temperature coefficient of resistance (TCR) in the operating temperature range; (c) environmental stability under processing and operating conditions; and (d) reproducibly obtainable properties under easily controlled processing conditions. In general, films consisting of two or more elements or compounds are used to achieve a high resistivity, low TCR material.

Perhaps the most widely used film resistor system is an alloy of nickel and chromium, usually in a ratio of 80:20. For vacuum deposition, a difficulty is encountered in controlling the stoichiometry of nickel/chromium films deposited from a single source because the two elements have significantly different vapor pressure characteristics, as can be seen in Fig. 1. Various film deposition techniques have been used to obtain reproducible films as has been reviewed by Siddall.(1)

A two-component system with elements having nearly identical vapor pressures in the range normally used for vacuum deposition is tantalum/rhenium as shown in Fig. 1. This indicates that films could be deposited for long periods of time using a single source--a factor of vital importance for low-cost production of resistor films. The purpose of this study was to evaluate the resistor characteristics of several Ta:Re compositions: 75:25; 50:50; and 25:75. The properties of these alloy films will be compared with pure films of Re and Ta.

Previously, both tantalum and rhenium films have been reported for resistor applications. Feldman and Culver(2) and Hemmer *et al.*(3) described the electrical properties of rhenium films. These investigators pointed out that the film properties changed unless packaged in a water-free environment. Tantalum resistor films have been reported by Maissel(4) who found that heat treatment of the films in air leads to large increases in resistance. This is attributed to grain boundary oxidation in the tantalum films.

[R.E. HONIG, RCA REVIEW, 23, 574 (1962)]

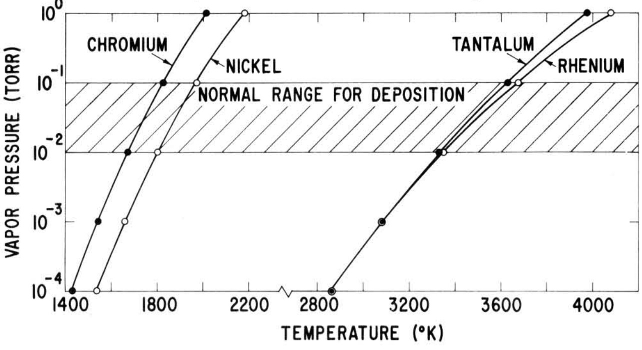

Fig. 1 Vapor pressure vs temperature.

II. EXPERIMENTAL PROCEDURE

A water-cooled 18- by 30-inch stainless steel vacuum chamber was used for film deposition. The pumping system included a liquid nitrogen-cooled, zeolite-filled, antimigration trap mounted between the chamber and a 6-inch oil diffusion pump.

The substrates used were glazed Alsimag 614 alumina (American Lava Co.). Prior to deposition they were thoroughly cleaned in a boiling detergent solution, rinsed in baths of deionized water, and dried in hot vapors of isopropyl alcohol. A tungsten-wire wound substrate heater was positioned behind the substrates, which were held in a stainless steel frame located 14 inches from the source. A platinum/platinum-rhodium thermocouple was pressed against the back of the substrate holder for a relative indication of temperature.

Evaporation was done from sources contained in water-cooled copper crucibles heated by a Temescal 10 kw electron beam gun. The sources were made from nominally high purity starting materials. The tantalum and rhenium sources were made from wrought metal; the alloy sources were made by mixing and pressing powder compacts of the proper proportions.

Four resistors were patterned on each 1- by 1-inch substrate using a precision-machined stainless steel stencil mask. The resistors were dumbbell-shaped with an active resistor of 1 by 10 mm (10 squares). After deposition, individual resistors were obtained for tests by scribing and breaking the substrates.

Resistor terminations were made just prior to electrical tests by scrubbing high-purity indium solder onto the "end areas" of the resistor films. Very thick (~1 mm) indium layers were used to minimize the contact resistance for two-probe testing. Previously, similar samples also were tested by the four-probe technique and it was found that the contact resistance was less than 0.1 ohm.

All electrical tests were made at room atmosphere. Temperatures above room temperature were achieved using a laboratory hot-plate. The temperature was indicated by a thermocouple mounted in a copper block located on the hot-plate surface next to the resistor. Heat treatment of resistor samples was done by placing them on a hot-plate in room environment.

Thermal aging tests were conducted by holding the samples in an oven at 125°C in room atmosphere. Periodically the samples were removed from the oven and their resistance was measured at 25° and 125°C.

Resistance measurements were made using a Fairchild Model 7000 digital multimeter. Temperature coefficient of resistance (TCR) data were calculated from resistance measurements made at room temperature and at 125°C. Resistivity data were calculated based upon film thickness measurements made using a Sloan Model M-100 angstrometer.

X-ray diffraction analyses were made by the Debye-Scherrer technique using powder obtained by scraping the films from the substrates.

III. RESULTS AND DISCUSSION

A. Pure Tantalum Source

These films were deposited 200Å to 300Å thick at deposition rates from 30 to 2000Å/min on substrates from room temperature to 375°C. TCR values were generally -125 to -300 ppm/°C with resistivities from 125 to 450 μΩ-cm. On 125°C aging tests, as-deposited films showed a resistance increase of 5% to 10% during >1000 hours. However, films heat treated at 250°C showed resistance drifts of ~0.4% to 0.8% during >1000 hours. The principal disadvantage of these films is their relatively large TCR values.

B. Pure Rhenium Source

Films 100Å to 300Å thick were deposited at 20 to 40Å/min on substrates from room temperature to 300°C. Typically, the high-temperature films were very metallike as evidenced by a high positive TCR of ~1000 ppm/°C. The resistivity of these films was ~100 μΩ-cm. Comparable values for room-temperature films were: TCR ≅ -100 ppm/°C; ρ ≅ 300 μΩ-cm. A near zero TCR film was measured on a substrate heated to ~100°C.

The resistance values of all samples drifted severely during aging tests at 125°C. A number of samples were heat treated at 250° and 350°C to attempt to stabilize the films by forming a protective oxide on their surface. The heat-treated films also showed large resistance drifts during aging. These films would not be useful in room atmosphere environment.

C. 75% Ta-25% Re Source

These films were deposited 300Å to 1100Å thick at rates from 50 to 100Å/min on substrates from room temperature to 300°C. The electrical values for films on substrates heated to ~200°C or less had rather highly negative TCR values (-150 to -300 ppm/°C) and high resistivities (300 to 800 μΩ-cm).

However, the films deposited on the 300°C substrate showed good characteristics after 400°C heat treatment. The properties of the as-deposited film were: R ≅ 40 Ω/□; TCR ≅ + 400 ppm/°C; and ρ ≅ 110 μΩ-cm. The properties of the films as a function of heat treatment are shown in Fig. 2. All of these films had a resistance increase of about 0.3% during 125°C aging for 1200 hours.

D. 50% Ta-50% Re Source

This composition was explored most thoroughly because of encouraging initial results. One of the two 50:50 sources made for this study was used for 22 evaporation runs. The initial weight of this source was ~30 grams and the final weight was ~20 grams. Films deposited in early runs had nearly the same properties as comparable films deposited in late runs indicating that a tantalum-rhenium source can be expected to have a long useful lifetime.

The effects of substrate temperature on the properties of films were evaluated using films ~300Å thick. Data are shown in Table I for ~100 Ω/□ films deposited at ~75Å/min. It can be seen that except for the room-temperature substrate, the films all have as-deposited TCR values less than -100 ppm/°C. Based upon this limited data, a substrate temperature of 200°C is preferred to minimize resistor drift due to 125°C aging.

Other tests made on films deposited from a 50:50 source indicate that stable resistors ranging from ~25 to ~100 Ω/□ can be fabricated. The TCR values of these films are less than 100 ppm/°C.

E. 25% Ta-75% Re Source

Resistor films of less than 100 Ω/□ were deposited

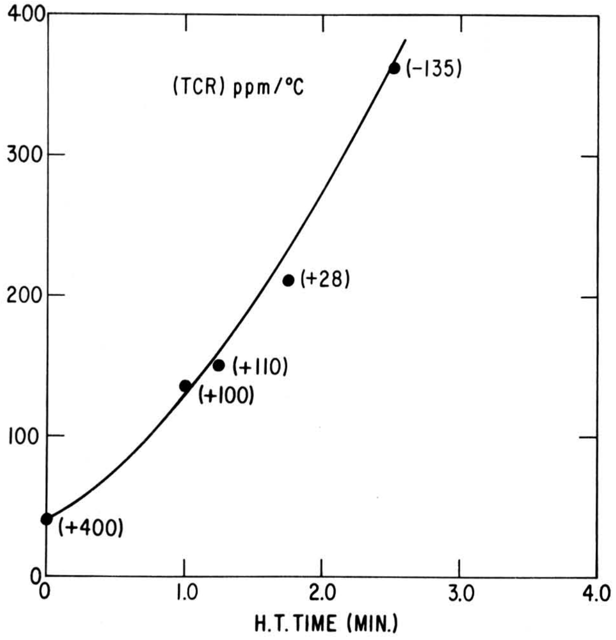

Fig. 2 Effect of heat treatment at 400°C on the resistancy of 75 Re/25 Ta films.

TABLE I

50% Ta-50% Re FILMS
EFFECTS OF SUBSTRATE TEMPERATURE

Deposition Conditions:

Power - 8 KV; 550 ma
Film Thickness - ~300Å
Deposition Rate - ~75Å/min

Substrate Temperature (°C)	As Deposited			After heat-treatment for 30 min @300°C			After aging for 1300 hours @ 125°C		
	$R_{25°C}$ (Ω/□)	TCR (ppm/°C)	ρ μΩ-cm	$R_{25°C}$ (Ω/□)	TCR (ppm/°C)	$\frac{R_{25°C} \text{ after H. T.}}{R_{25°C} \text{ as deposited}}$ (%)	$R_{25°C}$ (Ω/□)	TCR (ppm/°C)	$\frac{R_{25°C} \text{ after aging}}{R_{25°C} \text{ after H. T.}}$ (%)
Room Temp.	97.27	-153	353	114.0	-140	+17.2	115.5	-147	+ 1.31
120	93.31	- 81	289	125.7	-103	+34.7	127.1	-110	+ 1.11
200	87.34	- 82	245	113.6	- 96	+30.1	114.1	-105	+ .44
300	99.3	- 70	212	134.4	- 97	+35.3	186.6	-128	+38.8
400	98.6	- 60	234	157.6	-107	+59.8	179.5	-122	+13.9

from this source on substrates from room temperature to ~400°C. These films all were very unstable during 125°C aging tests, with or without a stabilizing heat treatment.

F. X-ray Diffraction Analyses

Several films deposited in this study were examined by x-ray diffraction analysis. For both the 75% Ta-25% Re and 50% Ta-50% Re films, only Ta-Re solid solutions were found. This would be reasonable based upon phase diagram studies which show a Ta-Re solid solution region up to ~49 w/o Ta.(5) The grain sizes of these films were very small as indicated by broad, diffuse lines in the diffraction patterns.

IV. CONCLUSIONS

Potentially useful resistor films consisting of tantalum and rhenium have been shown by this survey study. Present indications are that the film composition should contain 50% or more tantalum. Alloys in this compositional range have far superior thermal aging characteristics than pure rhenium films, and nearer zero TCR properties than pure tantalum films.

ACKNOWLEDGMENT

J. T. Furey performed much of the experimental work reported in this study.

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