Final Report
for

Apollo Communcation Systems Support

Task 8b-RF Sputtering System
Task 8c-High Ohms/Square Resistive Films

NAS 9-6636
W.E.C. GOHS-51431

Westinghouse

ELECTRIC CORPORATION

CR 108610

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TASK 8b

Introduction

The task objective was the development and delivery of an RF sputtering system to be used in conjunction with the CVC High Vacuum Evaporator and the RF power supply at NASA-MSC. The system was shipped from Westinghouse-Baltimore to NASA-Houston on 15 June 1970.

Procedure

The following items should be available at NASA-Houston for use with the RF sputtering system when it is set up by Westinghouse personnel:

(1) An RF power supply operating at 13.56 MHz, preferably crystal controlled (although this is not necessary), and operating with a 50 ohm output impedance (Type N connection).

(2) An argon gas supply. Oxygen and nitrogen for reactive sputtering should be available for future use. A gas mixing and flow measuring system is preferred (Figure 1). This arrangement mixes the gases, if required, and exhausts the argon or mixture to air, with a portion of the flow being diverted through a needle valve into the bell jar. By permitting the gas to flow briefly before opening this valve, any air which may have leaked into the mixing system is exhausted. Fluctuations in the flow rate into the bell jar are also minimized by this system.

(3) A water supply with about 50 PSIG pressure capable of two separate valvings, one for the water hoist required to lift the top plate of the sputtering system and one for the water cooling of the targets and substrate tables.

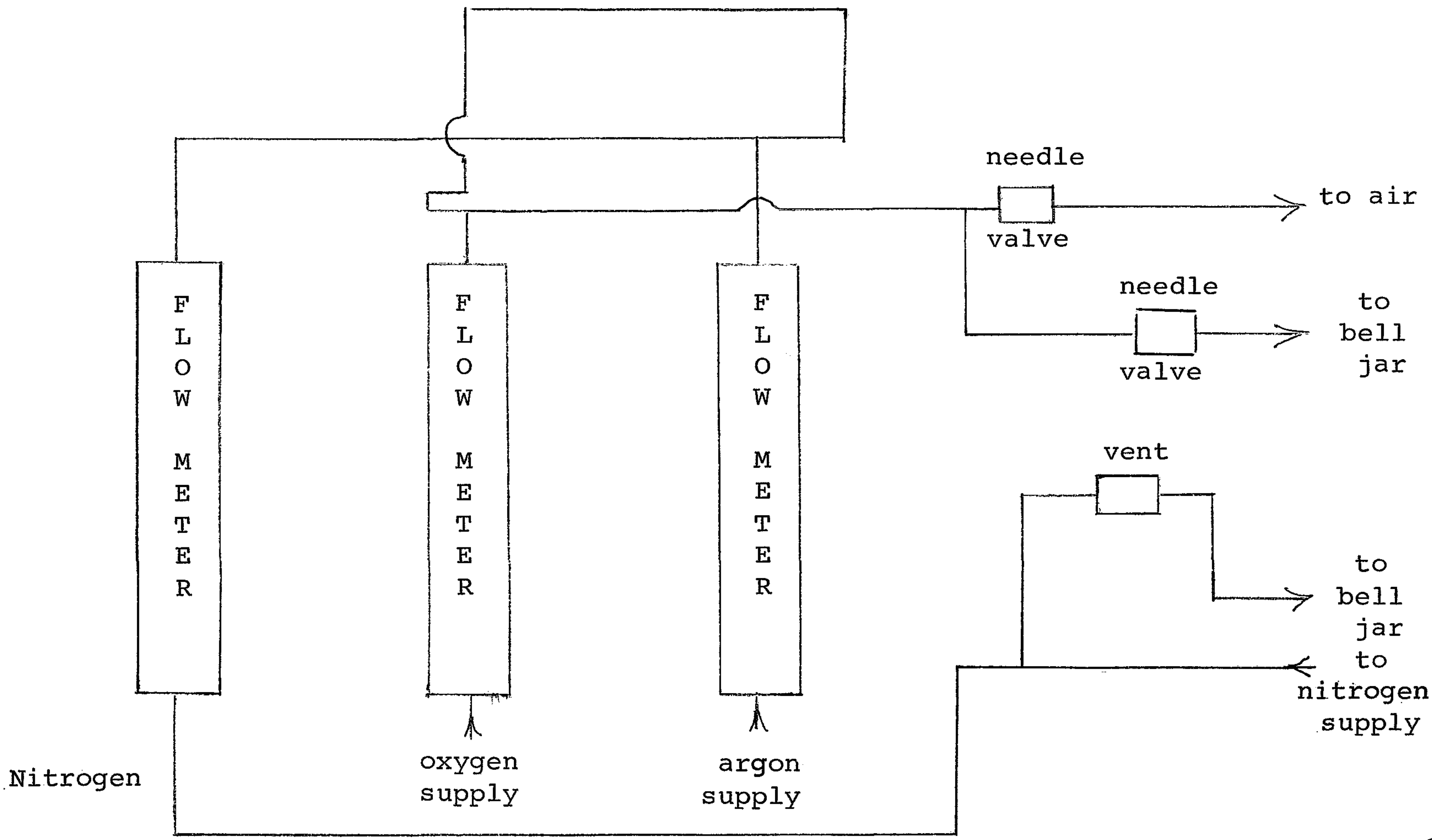

Figure 1
Task 8b

It is also suggested that a Bird Wattmeter be purchased for use with the RF system. The unit is a "Thruline" Wattmeter Model 43 (50 ohm impedance) manufactured by Bird Electronic Corporation of Cleveland, Ohio. The insert required is 500 watt, 2-30 MHz range, type 500H.

Upon receipt of the system it should be unpacked and checked for damage. It should then be mounted on the appropriate vacuum station and the tie downs, which are found attached to the feedthrough ring, should be bolted to the baseplate of the vacuum station. The holes through the tie downs are used for alignment and holes (1/4-20) one-half inch deep should be drilled and tapped in the baseplate. These are required to prevent tipping of the system when the top plate is in the raised position. The water and gas should be connected to the appropriate points and the vacuum capacitors installed. A Pirani gauge, four five-inch diameter aluminum substrate platens, two targets and an instruction manual are provided with the system. Once the system is ready for operation a Westinghouse engineer will demonstrate the operation of the system.

The following procedure should be followed when operating the sputtering unit:

(1) Plug in fan.

(2) Plug in Pirani Gauge.

(3) Turn on all waters.

(4) Turn on power supply.

(5) Turn Pirani Gauge to the 100 scale.

(6) Align substrate and shield to proper target.

(7) Turn on proper gas.

(8) Turn on exhaust.

(9) Turn off ion gauge.

(10) Set gas flow to proper setting.

(11) Let gas flow for 1 minute.

(12) Open bleed.

(13) Set power control to 40-45.

(14) Close high vacuum valve until a glow discharge is established, then open valve until desired pressure is read on Pirani Gauge.

(15) Maximize glow color with load tuning.

(16) Turn power control to proper forward power setting - will be given in watts.

(17) Minimize reflective power (closest to zero) with load tuning.

(18) Set timer.

(19) Maintain forward power, reflective power and pressure at correct values during run.

(20) At end of run, turn down power to zero.

(21) Move substrate and shield and switch to other target.

(22) Make sure proper alignment is maintained.

(23) Set scale on Pirani Gauge to 100.

(24) Set power control to 40-45.

(25) Follow steps 14-20.

(26) Turn off main switch on power control unit.

(27) Turn scale on Pirani Gauge back to 100.

(28) Turn off Pirani Gauge.

(29) Close bleed and gas supply.

(30) Turn off water.

(31) Open gate valve.

This procedure has been used for all Westinghouse RF sputtering trials.

TASK 8c

Introduction

The task objective was to develop a process for sputtering high ohms/square resistive films to form thin film resistors. Thin film resistors are preferable to diffused resistors in integrated circuits because of their lower temperature coefficient of resistance, lower parasitic capacitance, and greater isolation from the rest of the circuit. These characteristics become especially important in an ionizing atmosphere where diffused resistors may be effectively shorted due to photocurrents.[1] As the resistance of thin film resistors depends on their length/width ratio, the high ohms/square films yield larger value resistors in a smaller area as compared to lower sheet resistance films. Sample chromium disilicide resistors with aluminum contacts were fabricated and tested as described below.

Procedure

The RF sputtering system was used according to the instructions in the final report for Task # 8b. Nominal

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[1] Stafford, K. R. and Oberlin, D. W., Solid State Technology, May 1970, p. 68.

sputtering conditions were as follows:

Micrometer leak valve setting	19.5
Argon flow	10 cfh
Pressure	8 microns
Power	100 watts

All runs included a one-hour pump-down and five minutes of pre-sputtering with the substrates shielded. Both materials were deposited in the same cycle. A deposition time of 30 seconds for chromium disilicide yielded films with sheet resistances of 1000 ± 50 ohms/square. This time is critical and a few seconds more or less will result in far different resistance values. Aluminum must be sputtered for 30 minutes to obtain a 5000 Å film. The power may be increased to 200 watts and the time reduced to 15 minutes with approximately the same results. Aluminum films sputtered at the lower power were shinier in appearance.

Oxidized silicon wafers were used as substrates for the resistors. They were cleaned prior to deposition in hot sulfuric acid, hot nitric acid, and hot deionized water. After depositing $CrSi_2$ and aluminum, the contact and resistor patterns were photoengraved. The $CrSi_2$ was etched for 30 seconds in the following solution:

1 part 95-5 (95% HNO_3, 5% HF)

3 parts acetic acid

The etch designated in the literature[2] for $CrSi_2$ is a mixture of concentrated phosphoric, nitric and hydrofluoric acids (60-5-1 by volume). However, when this solution was used on the $CrSi_2$ deposited on glass substrates it proved to be slower than stated even when heated and had a tendency to etch the glass. Unbuffered HNO_3 - HF in a 95-5 combination (by volume) etched the $CrSi_2$ at the rate of 250 Å/second at room temperature which is too rapid for the thin ($\sim$200 Å) films. The addition of acetic acid slowed this rate down to about 6 Å/second.

Following resistance and temperature coefficient of resistance measurements, the wafers were annealed in nitrogen for 10 minutes at 500°C and the TCR measurements were repeated.

Results

Sintering before photoengraving resulted in chromium disilicide films which were not electrically continuous. It is postulated that the aluminum alloyed with the silicon in

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[2] Waits, R. K., Trans. Met. Soc., AIME, 242, March, 1968, p. 491.

$CrSi_2$ and that the alloy was soluble in aluminum etch. Other investigators have observed reactions between the resistor film and aluminum contacts during annealing which produced a low resistivity phase along the resistor, although this effect was not observed with films containing more than twenty-five atomic per cent chromium.[3]

Attempts to deposit the chromium disilicide film over a photoengraved aluminum pattern were unsuccessful, the $CrSi_2$ being too thin to cover the steps in the aluminum. Lack of conduction could have been caused by the formation of an aluminum oxide layer on the surface of the aluminum. However, 50 Å of aluminum oxide would have broken down under the applied 100 volts, so it is more probable that there was no continuity over the aluminum steps.

Four wafers were processed, the sheet resistances measured with a four point probe, and resistor measurements made by using an oscilloscope. The sheet resistances show a 10% variation over the surface of each wafer. Talysurf measurements on the 1000 ohms/square films indicate thicknesses in the 200 Å range. The test pattern chip

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[3] Waits, R. K., The Journal of Vacuum Science and Technology, 6, No. 2, pp. 309-310.

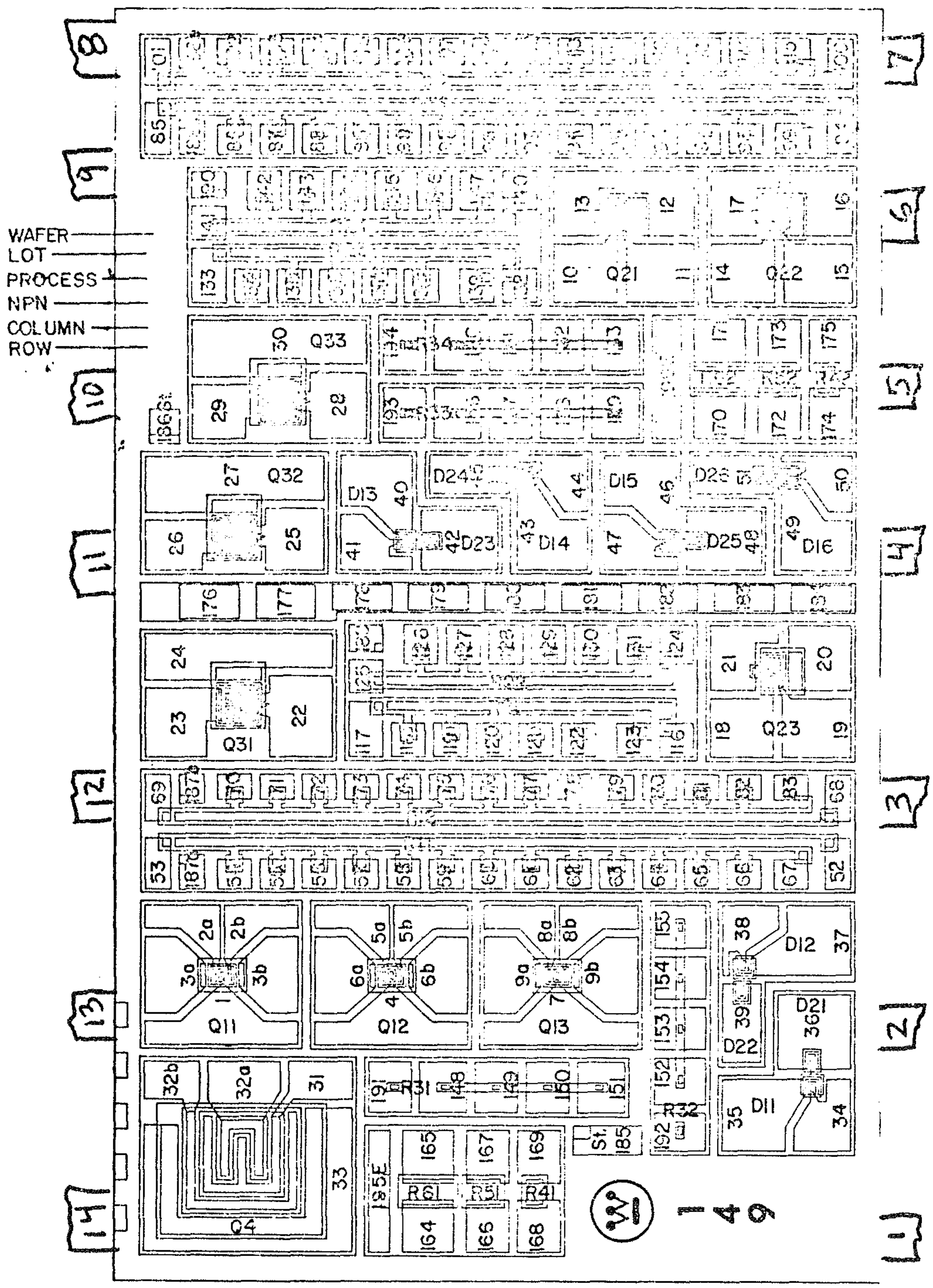

Figure 1
Task 8c

Table 1

Squares	R_{11}	R_{12}	R_{13}	R_{14}
152	52-53	68-69	84-85	100-101
15	53-54	69-70	85-86	101-102
25	53-55	69-71	85-87	101-103
35	53-56	69-72	85-88	101-104
45	53-57	69-73	85-89	101-105
55	53-58	69-74	85-90	101-106
65	53-59	69-75	85-91	101-107
75	53-60	69-76	85-92	101-108
85	53-61	69-77	85-93	101-109
95	53-62	69-78	85-94	101-110
105	53-63	69-79	85-95	101-111
115	53-64	69-80	85-96	101-112
125	53-65	69-81	85-97	101-113
135	53-66	69-82	85-98	101-114
148	53-67	69-83	85-99	101-115
10	54-55	70-71	86-87	102-103
13	66-67	82-83	98-99	114-115
300	52-67	68-83	84-99	100-115

Squares	R_{21}	R_{22}	R_{23}	R_{24}
74	116-117	124-125	132-133	140-141
6	117-118	125-126	133-134	141-142
16	117-119	125-127	133-135	141-143
26	117-120	125-128	133-136	141-144
36	117-121	125-129	133-137	141-145
46	117-122	125-130	133-138	141-146
61	117-123	125-131	133-139	141-147
11	118-119	126-127	134-135	142-143
15	122-123	130-131	138-139	146-147
135	116-123	124-131	132-139	140-147

Squares	R_{31}	R_{32}	R_{33}	R_{34}
15	148-151	152-155	156-159	160-163
5	148-149	152-153	156-157	160-161
5	149-150	153-154	157-158	161-162
5	150-151	154-155	158-159	162-163

1	R_{41}	168-169	R_{42}	174-175
1/2	R_{51}	166-167	R_{52}	172-173
1/3	R_{61}	164-165	R_{62}	170-171

selected for the resistor testing is shown in Figure 1. The number of squares between specific pads can be obtained from Table 1. Data from these samples appears below.

Table 2
Resistance Measurements

Wafer No.	Sheet Res. (ohms/square)	Resistor No.	No. Squares	Resistance (kilohms)	Experimental Sheet Res. (ohms/sq.)
1	704	R_{11}	25	20.0	800
			95	71.5	753
			10	8.6	860
		R_{13}	25	20.0	800
			10	8.6	860
		R_{51}	1/2	.360	720
2	733	R_{11}	25	20.0	800
			95	71.5	753
			10	8.35	835
		R_{13}	25	18.5	743
			10	7.9	794
		R_{51}	1/2	.37	740
3	740	R_{11}	25	18.6	744
			95	67.6	712
			10	8.0	800
		R_{13}	25	18.0	721
			10	8.0	800
		R_{51}	1/2	.375	750
4	775	R_{11}	25	18.5	740
			95	70.0	738
			10	8.0	800
		R_{13}	25	17.8	715
			10	7.5	750
		R_{51}	1/2	.376	750

The temperature coefficient of resistance (ppm/°C) is equal to $10^4 \, [(R_2/R_1) - 1]$ where R_1 is the resistor value at 50°C and R_2 is the resistor value at 150°C. Diode sputtered chromium disilicide reportedly has a positive temperature coefficient (2500 - 2900 ppm/°C) below 350°C and is negative at higher temperatures.[4] Other sources list RF sputtered films 500 Å thick with sheet resistances of 300 ohms/square and temperature coefficients of -275 ppm/°C.[5] The temperature coefficients of the films sputtered were predominantly negative prior to annealing.

Table 3
TCR Measurements

Wafer No.	Sheet Res. (ohms/square)	No. Squares	R_1 (kilohms)	R_2 (kilohms)	R_2/R_1	(ppm/°C)
1	704	25	16.7	16.7	1.00	0
		95	62.5	60.0	.960	-400
		1/2	.291	.281	.965	-350
2	733	25	18.0	18.0	1.00	0
		95	66.7	66.7	1.00	0
		1/2	.322	.312	.968	-320
3	740	25	18.4	16.7	.908	-920
		95	66.6	64.0	.960	-400
		1/2	.368	.352	.956	-440
4	775	25	18.0	17.6	.977	-230
		95	50.0	64.0	1.280	+2800
		1/2	.357	.347	.973	-270

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4 Waits, R. K., <u>The Journal of Vacuum Science and Technology</u>, 6, No. 2, pp. 309-310.

5 L. E. Terry, Final Report, AD 827212 (February 1968), p.36.

Measurements on the same resistors after a ten minute sintering at 500°C indicated an increase in the resistor values rather than the expected decrease due to aluminum migration. Large widely varying positive values appear for the TCR. The increased resistivity could be explained by the formation of larger, more widely separated, islands in the thin film as a result of annealing. This would increase the distance electrons must tunnel in order to move from conglomerate to conglomerate. This configuration, however, usually exhibits a negative temperature coefficient.

Table 4
TCR Measurements After Annealing

Wafer No.	Sheet Res. (ohms/square)	No. Squares	R_1 (kilohms)	R_2 (kilohms)	R_2/R_1	(ppm/°C)
1	704	25	27.2	28.0	1.032	+320
		95	100.0	100.0	1.00	0
		1/2	.416	.800	1.92	+9200
2	733	25	32.0	33.3	1.040	+400
		95	116.5	120.0	1.028	+280
		1/2	.562	.666	1.185	+1850
3	740	25	33.3	35.0	1.050	+500
		95	120.0	150.0	1.25	+2500
		1/2	.281	.875	3.12	21,200
4	775	25	37.0	50.0	1.350	+3500
		95	120.0	134.0	1.12	+1200
		1/2	.240	.626	2.6	+16,000

Conclusions:

Chromium disilicide resistors having sheet resistances as high as 1000 ohm/square can be deposited by RF sputtering in argon. Devices annealed at 500°C in nitrogen exhibit an approximate doubling of resistance and a transition from a negative to a positive temperature coefficient of resistance.

Several areas of further study suggest themselves. The effects of sputtering power and pressure, contact metal and annealing temperature on the film resistivity and temperature coefficient should be investigated in order to explain and minimize the variations in film properties as a result of annealing. Life testing of the chromium disilicide resistors should also be undertaken before they are incorporated into a working system.