TI Report No. 03-65-112

October 1965

THIN FILMS FORMED BY ELECTROCHEMICAL REACTIONS

Quarterly Progress Report No. 7

1 June 1965, 31 August 1965

Contract No. DA 36-039 AMC-02324(E)

DA Project No. ICO-24401-A112-04

Prepared by

R. Scot Clark

Mike Gaze

Texas Instruments Incorporated

Semiconductor-Components Division

Post Office Box 5012

Dallas, Texas, 75222

For

U. S. Army Electronics Command

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ABSTRACT

Basic investigations in the residual gas contamination during sputtering and oxidation mechanism have yielded information that contributes to increased process control. Also, the oxidation experiments depict a structure that can be modified to aid in temperature coefficient of resistance control.

A process control investigation for fabricating 1000 Ω/□ resistors and > 0.35 pF/mil^2 capacitors on the same silicon substrate has been completed. The results show the yield for the 1000 Ω/□ ± 20% tolerance resistive film is 62% and for both the resistive and capacitive films, the yield is 38%.

An etch problem on the 455 KHz amplifier has delayed fabrication of this circuit. The problem has been solved by using a KMER mask to deposit the Ta_2O_5 dielectric through.

R. Scot Clark
Project Engineer

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SECTION I
INTRODUCTION

A. PURPOSE

The purpose of this investigation is the development of techniques for fabricating thin conductive, resistive and dielectric films, by complete or partial oxidation of a suitable metallic film deposited in predetermined patterns and sequences. The substrate of interest is primarily an oxidized silicon semiconductor slice containing diffused active elements.

B. OBJECTIVES

The objectives of this contract are:

1. Resistive Film
   1) Resistance up to 25,000 Ω/□
   2) TCR within ± 150 ppm over the temperature range -55°C to 125°C
   3) Power dissipation 2.5 ω/in^2
   4) < 1.0% change in resistance at 1000 hour on load life test
   5) Tolerance ± 2%

2. Capacitive Film
   1) Capacitance 2-5 pF/mil^2
   2) Dissipation factor < 1.0% at 1 KHz/sec
   3) Voltage breakdown > 75 V

4) TCC within ± 200 ppm/°C from -55°C to 125°C

5) Insulation resistance at 30 V and 25°C ≥ 1000 meg MFD or 10,000 MΩ and at 85°C ≥100 meg MFD or 1000 MΩ

3. Test Vehicle

1) Fabrication of a 455 KHz amplifier

2) Suitable life test of components of 455 KHz amplifier

SECTION II
EQUIPMENT DESIGN

A. CLEAN-DRY SPUTTERING PROCESS

1. General

A recent investigation in the control of the parameters of reactively sputtered resistors and capacitors has provoked the redesigning of sputtering equipment. The high incidence of dielectric failures due to dust particles is defined as one problem and lack of control of sputtering ambient gases contamination is defined as the second problem.

A clean-dry sputtering system is being designed and built expressly for thin-film sputtered resistors and capacitors. The design should considerably reduce these problems and increase the degree of control of the resistor and capacitor parameters. Experiments with a residual gas mass spectrometer outlined in the body of the report add considerably to the understanding of ambient gas condition that is required for good process control.

2. Residual Gas Analysis

The samples described in this work were prepared in conventional bell jar pumping systems with a glass bell jar 18 inches diameter by 30 inches high (volume 125 liters), an oil diffusion pump of nominal speed 1500 liters second, and a backing pump of nominal speed 12 cubic feet a minute; a liquid nitrogen cooled trap is used above the diffusion pump to prevent back streaming. System pressure is

regulated by means of a calibrated Alpert valve used as a leak and by throttling the diffusion pump using a gate valve. Initial adjustment is made to a chosen value of pressure as read by a thermocouple gauge, the glow discharge is then started and final adjustment made to particular values of current and voltage.

The major tool used for this investigation was an Aero Vac model AV1 vacuum analyzer. This instrument operates by ionizing the gas by a means similar to that used in an ionization gauge. The ionized gases are then deflected by a magnetic field according to their mass/charge ratio. Variation of the electrostatic field causes the different ion species to impinge on a collector for identification.

Unfortunately, this instrument only operates over the pressure range 10^{-4} torr to 10^{-9} torr, whereas the pressures used during sputtering are of the order 10^{-2}, 10^{-3} torr; therefore, direct analysis of the composition of the system gas during sputtering is not possible. Two possible methods of analysis are to take a sample of the system gas and reduce its pressure to a workable value by means of expanded volume techniques or to use differential pumping of the mass spectrometer lead to provide an operating pressure within the desired range. The first method is likely to give errors due to adsorption and desorption; the second is not convenient to use with this instrument, as it requires an appreciable amount of ancillary equipment and calibration and is liable to errors caused by differential pumping of the different mass species. In addition, both methods are rather tedious.

Therefore, the method adopted in practice was to make two separate series of mass scans over the pressure range 1×10^{-6} to 1×10^{-4} controlling the pressure in the first case by manipulation of the gate valve and in the second case by admitting a 50/50 mixture of Argon/oxygen. Typical results for the major constituents of a vacuum are shown in Figs. 1 to 7.

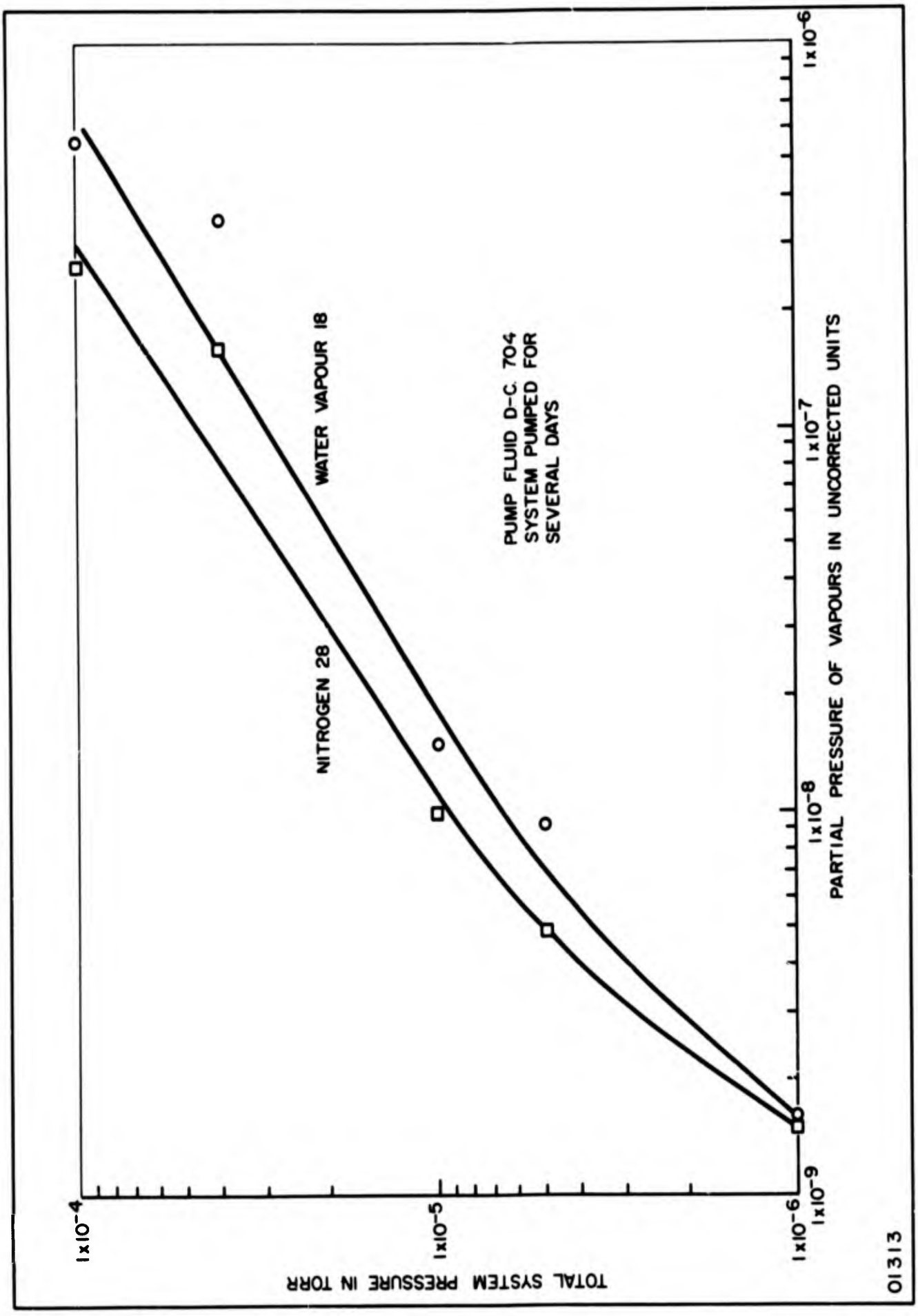


Fig. 1. System Total Pressure Adjusted by Pumping Speed

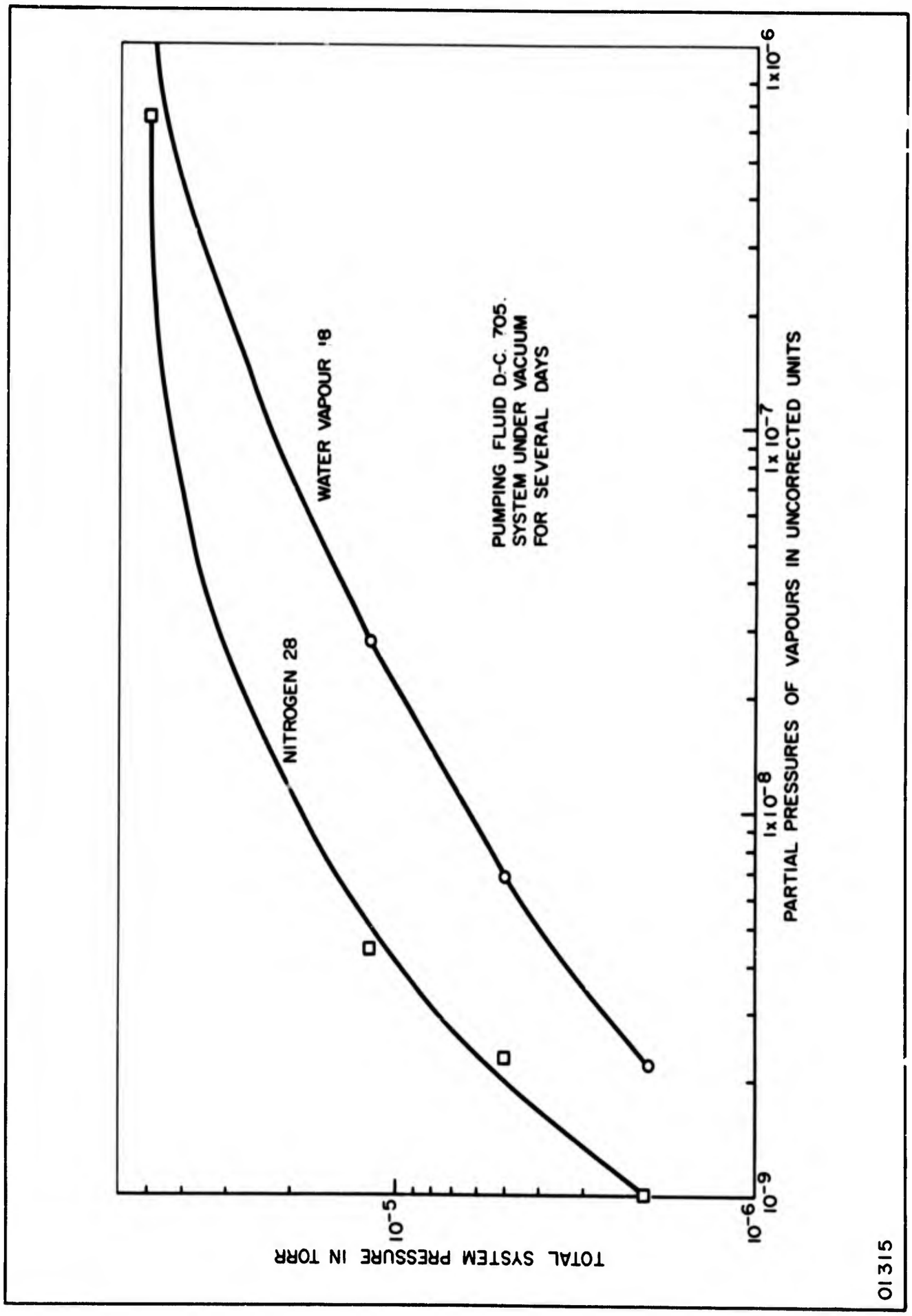
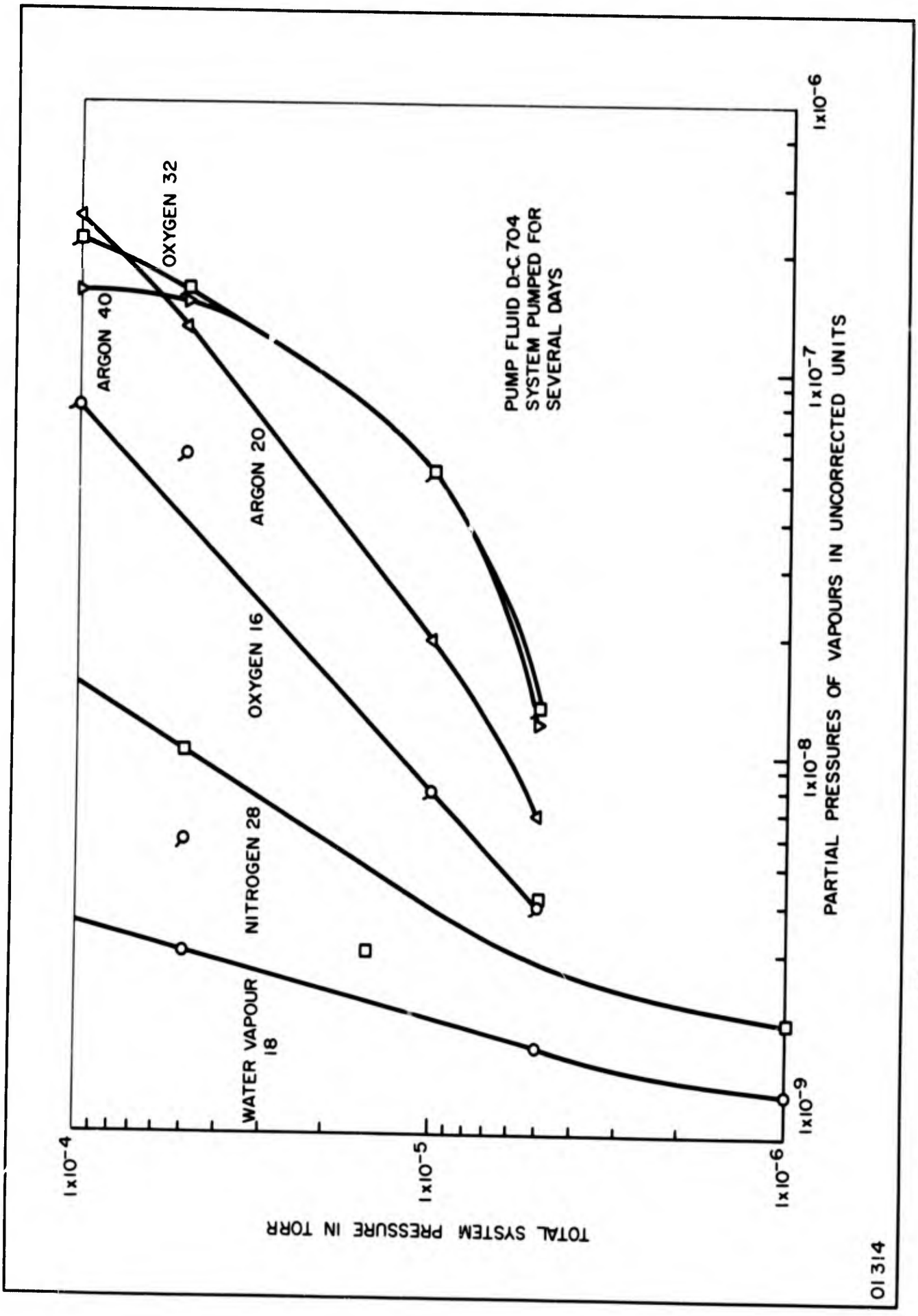


Fig. 2 — System Total Pressure Attainable During Pumping


TOTAL SYSTEM PRESSURE IN TORR
1x10⁻⁴
1x10⁻⁵
1x10⁻⁶
1x10⁻⁹
1x10⁻⁸
1x10⁻⁷
1x10⁻⁶
PARTIAL PRESSURES OF VAPOURS IN UNCORRECTED UNITS
WATER VAPOUR 18
NITROGEN 28
OXYGEN 16
ARGON 20
ARGON 40
OXYGEN 32
PUMP FLUID D-C 704
SYSTEM PUMPED FOR
SEVERAL DAYS
01 314

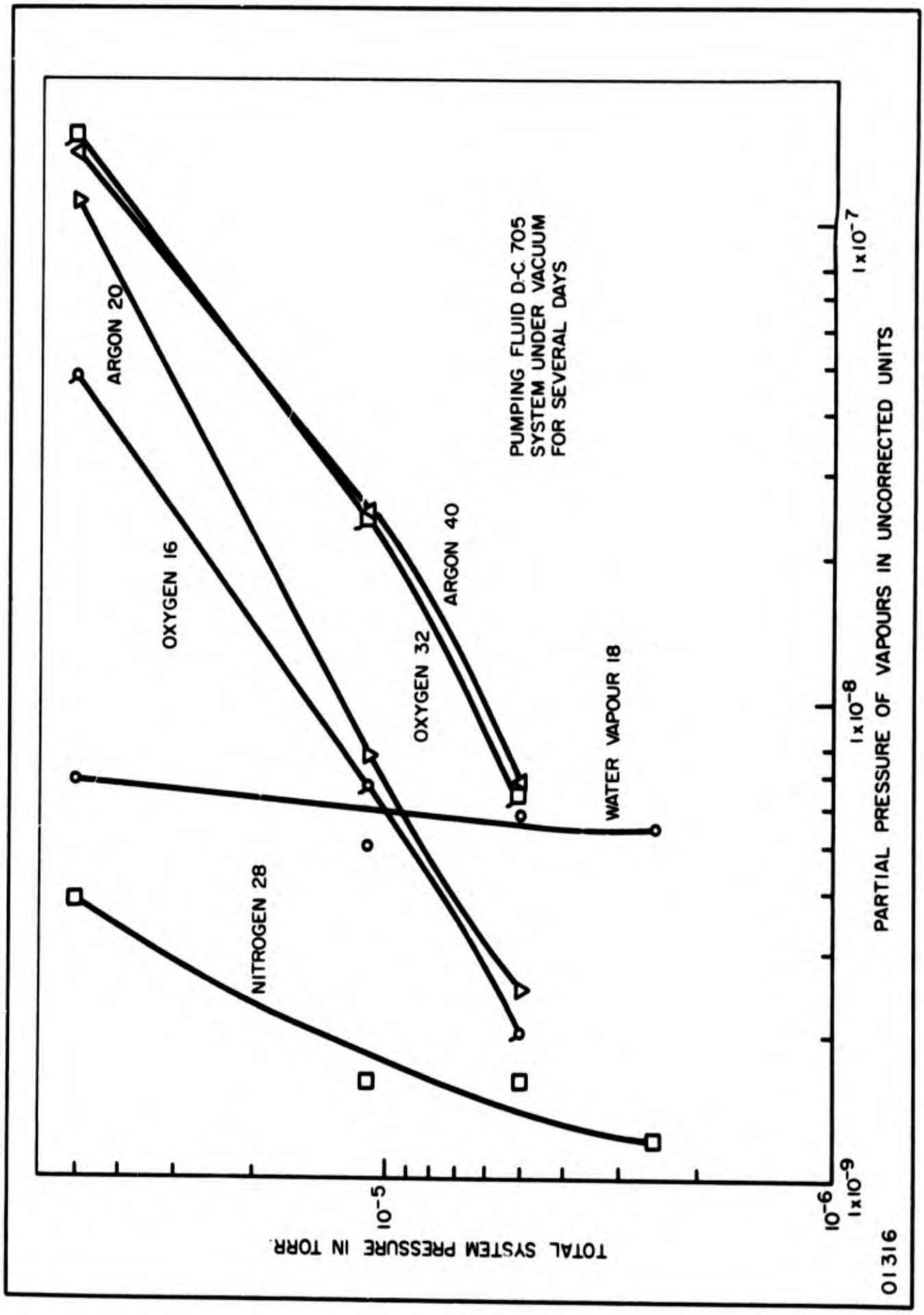


Fig. 4. System Total Pressure Adjusted by Admitting O_2/Ar

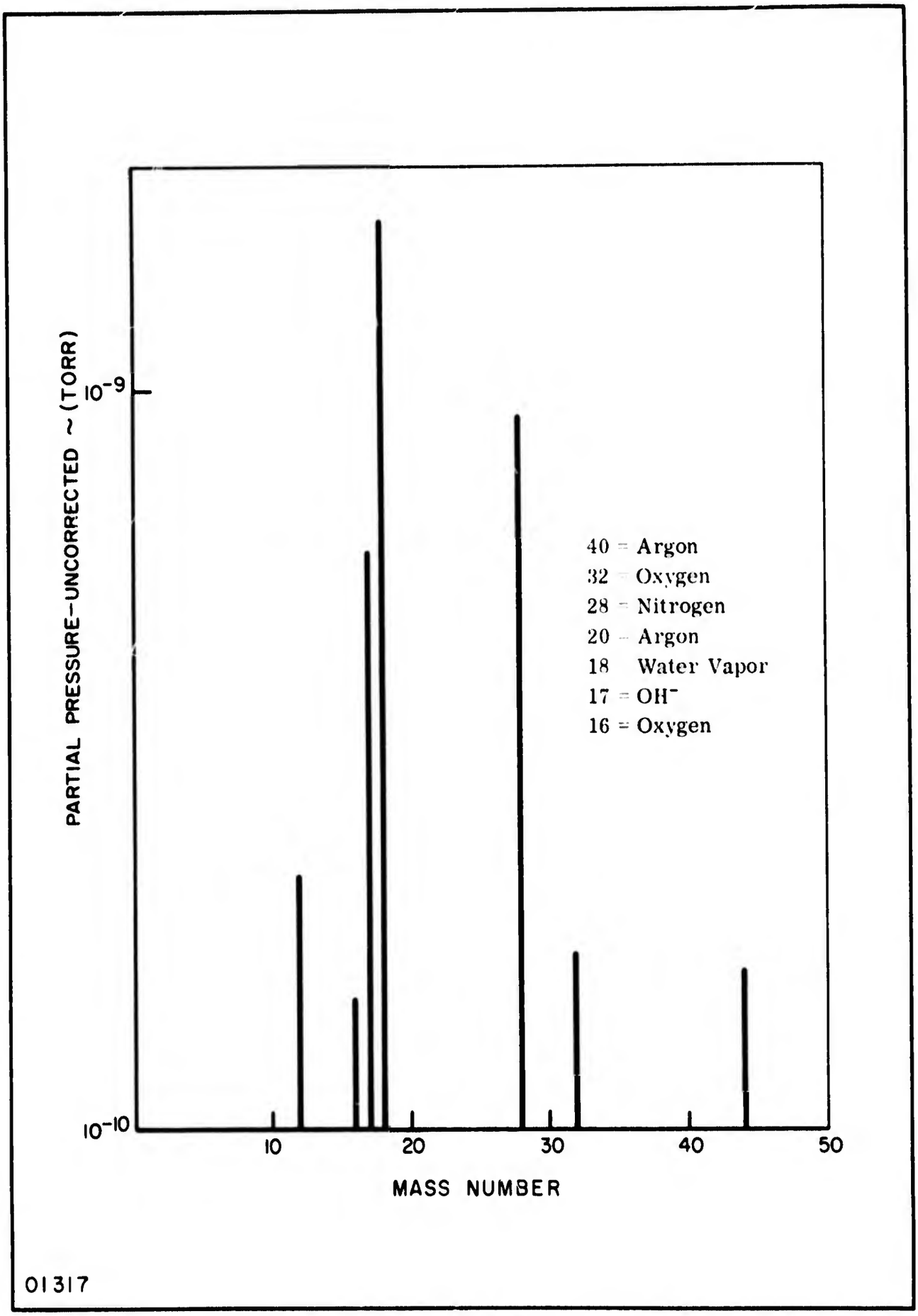


Fig. 5. Mass Scan of Major Constituents

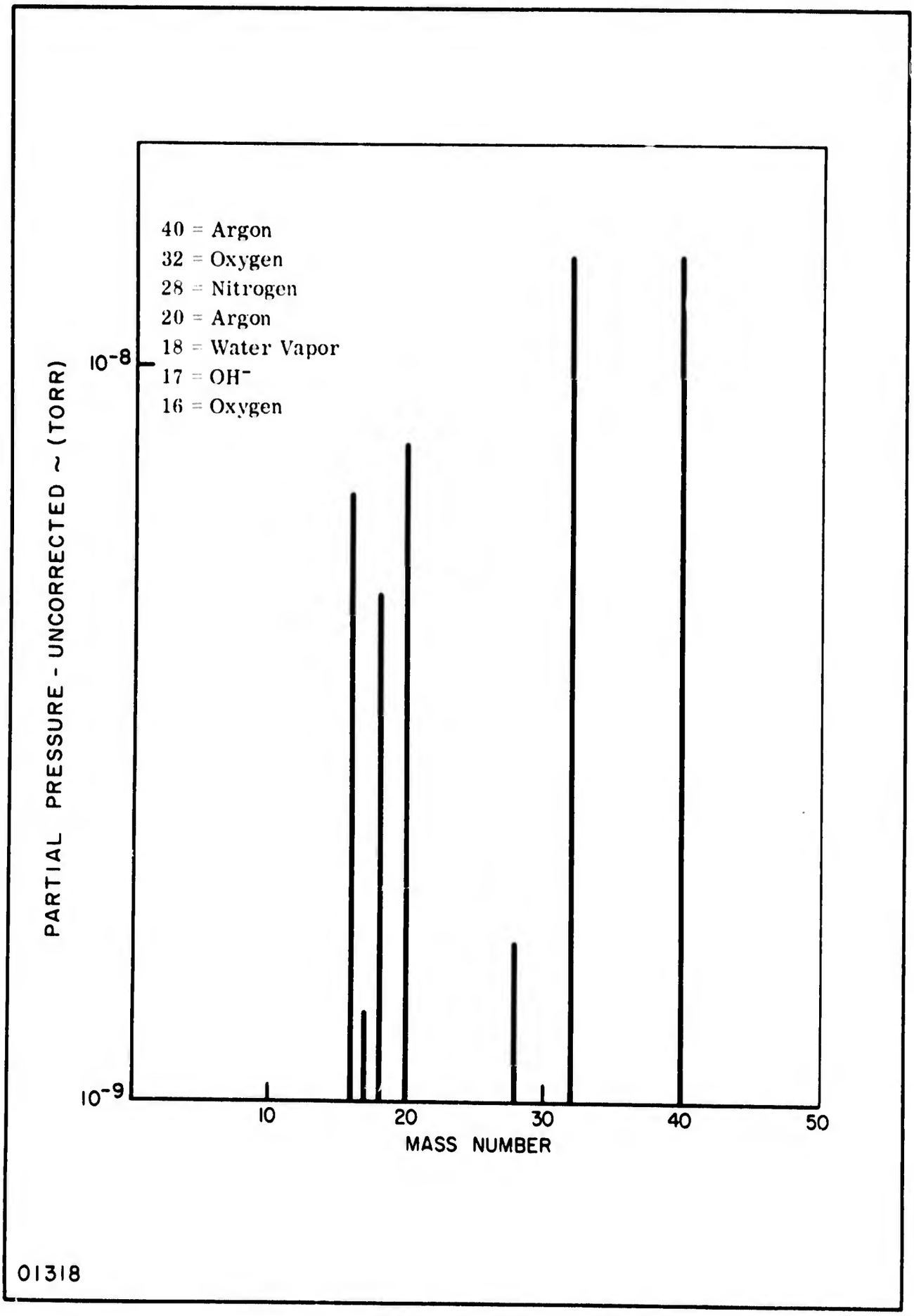


Fig. 6. Mass Scan of O_2/Ao 50%/50%

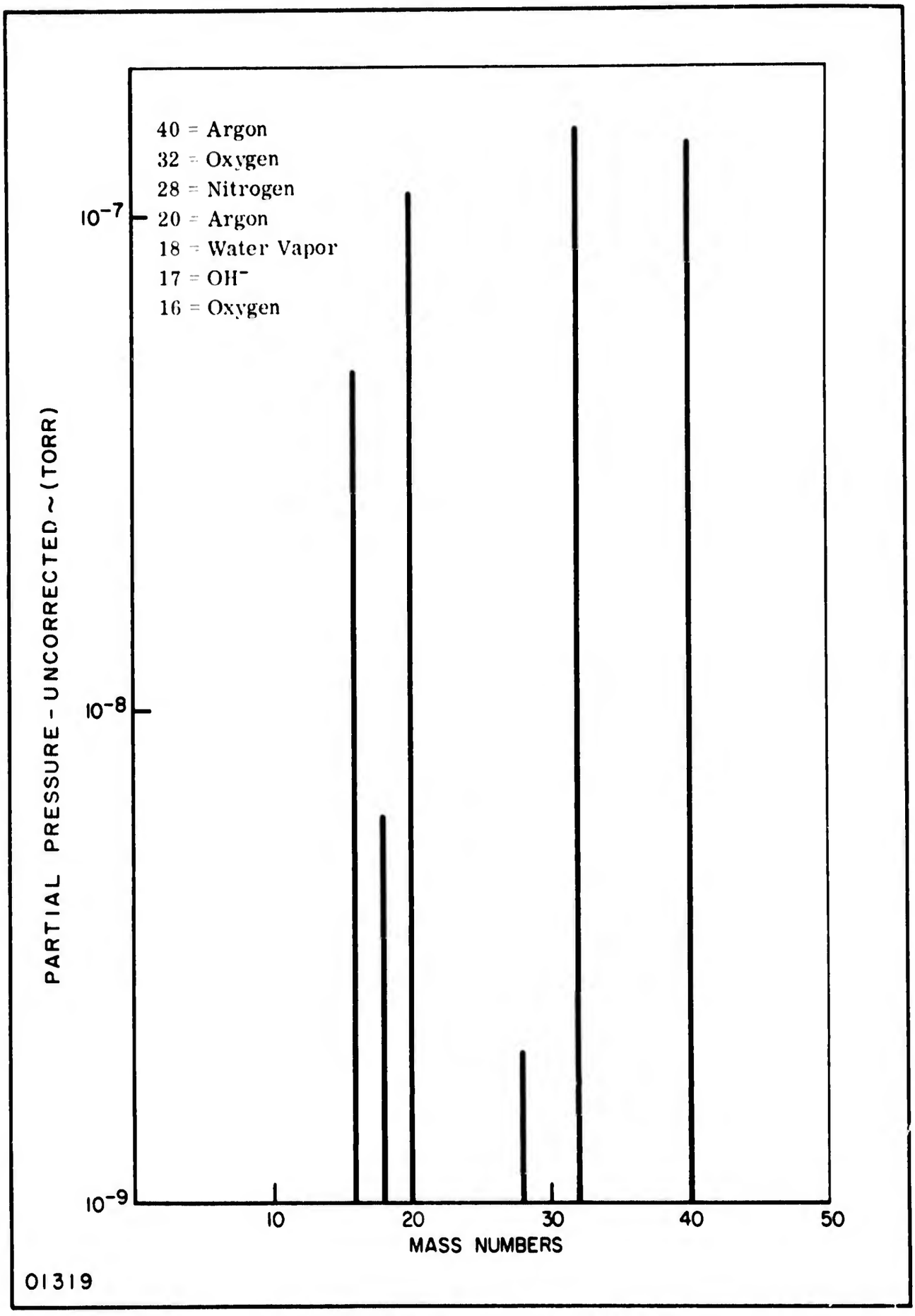


Fig. 7. Mass Scan of O_2/Ao 50%/50%

When the pressure moderator is the gate valve, then the partial pressures of the individual gases vary with pumping speed as would be expected.

When the pressure is adjusted by means of gas admittance, then the partial pressures of the other species remain at substantially the same level provided the system has been well outgassed or has been pumped for prolonged periods (order of days) without breaking the vacuum. In the case of a system which has recently been exposed to normal (wet) laboratory atmospheres the partial pressure of water vapor, in particular, rises with rising total pressure.

A scan was made in which the system pressure was adjusted from 1×10^{-6} to 1×10^{-5} torr by admitting argon/oxygen and from 1×10^{-5} to 1×10^{-4} torr by adjustment of the gate valve (Fig. 8). In this case, water vapor makes a significant contribution to the total system pressure. During this scan the pressure in the backing line was 3.5×10^{-2} torr and assuming a speed of 12 cubic feet per minute for the backing pump, a theoretical effective pumping speed of 560 liters per second is calculated for the diffusion pump, which is slightly less than half the manufacturer's quoted speed for the pump.

The conditions during an actual sputtering run are worse, the diffusion pump is throttled back with the gate valve and the effective pumping speed is calculated to be 375 ccs per second. Therefore, it may be assumed that the sputtering atmosphere contains a large unpredictable percentage of water vapor which degrades the reliability of the process.

Process reliability is further degraded by contamination of oil vapor caused by backstreaming. Investigation of backstreaming has been hampered by the fact that the mass spectrometer will not measure masses greater than 70, but within the limits of the instrument extensive measurements were made.

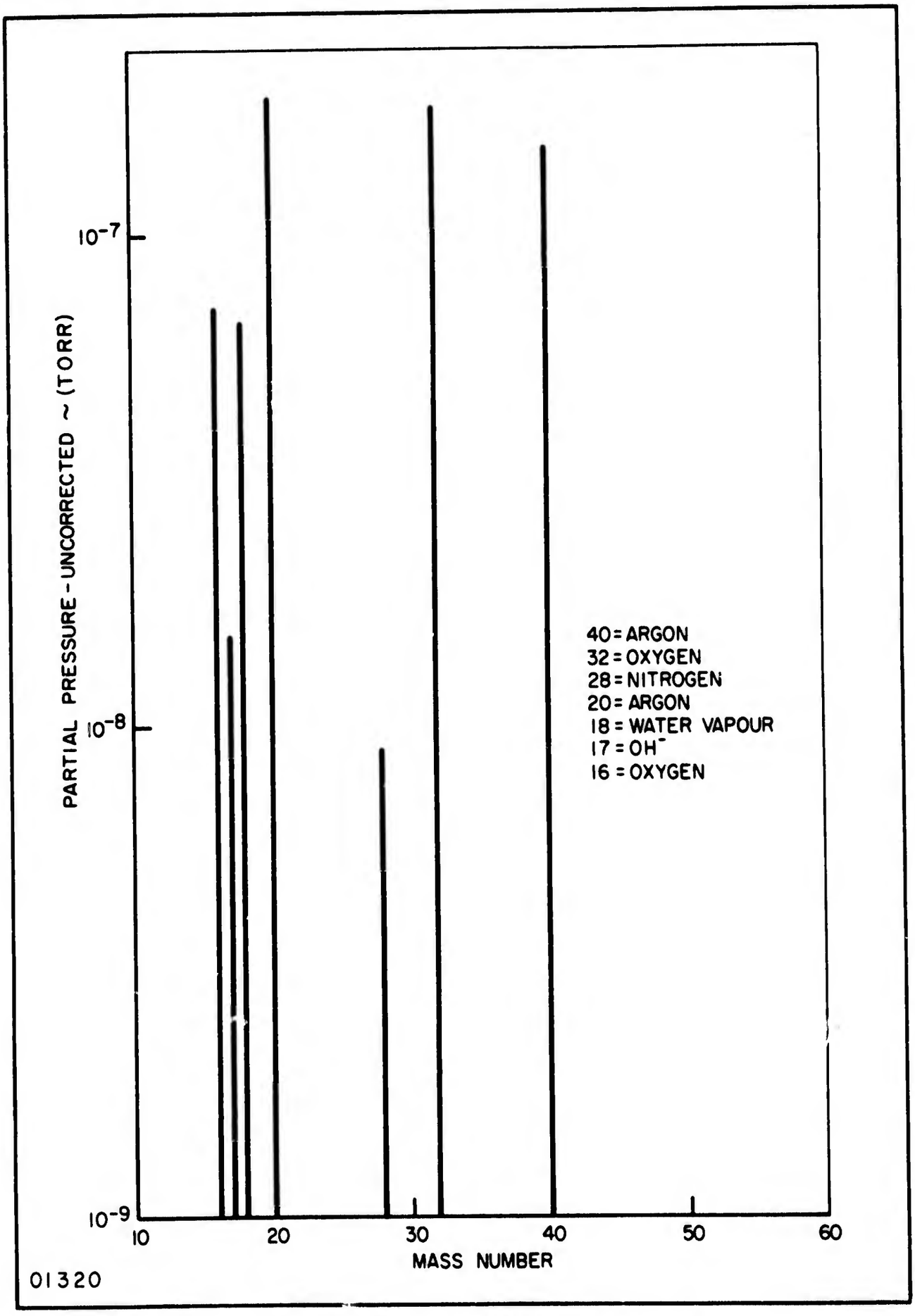


Fig. 8. Mass Scan of Major Constituents

It has been established that proper maintenance is essential in order to eliminate the back streaming, which adversely affects the adhesion of the film.

3. Discussion

From the work described, it was concluded that major sources of inconsistency in the sputtering process are water vapor and backstreaming of oil vapor.

The water vapor contamination may be reduced by reducing the internal surface area of the sputtering chamber to the practical minimum. If this is done, there is a further advantage that the sputtering chamber volume is also a minimum which will make automatic regulation of gas dosage (a desirable feature) to be more accurately controlled. (It is desirable that the ratio system volume to pumping speed be less than 1 for good pressure control.)

At present, backstreaming is prevented by the use of a liquid nitrogen filled cold trap; it is both inconvenient and expensive to maintain such a trap in the filled condition for periods longer than eight hours per day. Therefore, the practice at present is to allow the trap to warm up overnight with the main gate valve closed and the power to the diffusion pump either turned down to less than 40 per cent normal power or cut off completely.

Under these conditions the possibility of oil contamination creeping up the walls of the diffusion pump and through the cold trap to the underside of the gate valve under the action of surface tension forces may not be overlooked. For this reason, the vacuum systems used for sputtering are dismantled and thoroughly cleaned every 3-4 weeks.

The effeciency of trapping of a liquid nitrogen trap depends upon the mean free path of gas molecules in the system being much longer than the spacing between the cooled surfaces of the trap to insure a high probability of collision. In normal vacuum ambients with a total pressure of 1×10^{-6} torr, the mean free path for air is about 50 meters and the stated condition is satisfied. When sputtering chamber pressures of 5×10^{-2} torr are used, the mean free path for air is about 0.1 cm. Under these conditions, a cold trap is a less effective means of preventing backstreaming.

4. Sputtering System Design

Serious consideration has been given to the design of a more suitable sputtering system and the following specification was devised:

1) System to have minimum practical volume.
2) Cathode to be water-cooled.
3) Anode to be heated.
4) Cathode/anode spacing to be readily adjustable.
5) A substrate plate which may be biased with respect to the anode to be available.
6) System to have a shutter close to the cathode.
7) System to be free from oil backstreaming.
8) Automatic control of gas admittance (regulated by sputter current).
9) Water vapor contamination minimized.
10) High voltage d-c supply to be stable and have low ripple.
11) Main chamber to be metal and at anode potential to prevent outgassing due to ion bombardment of chamber walls.

A design for a sputtering chamber which meets the relevant requirements is shown in Fig. 9. The main chamber is an aluminum prefabricated box with internal dimensions 10 inches by 10 inches by 3 inches (volume 0.17 cubic feet).

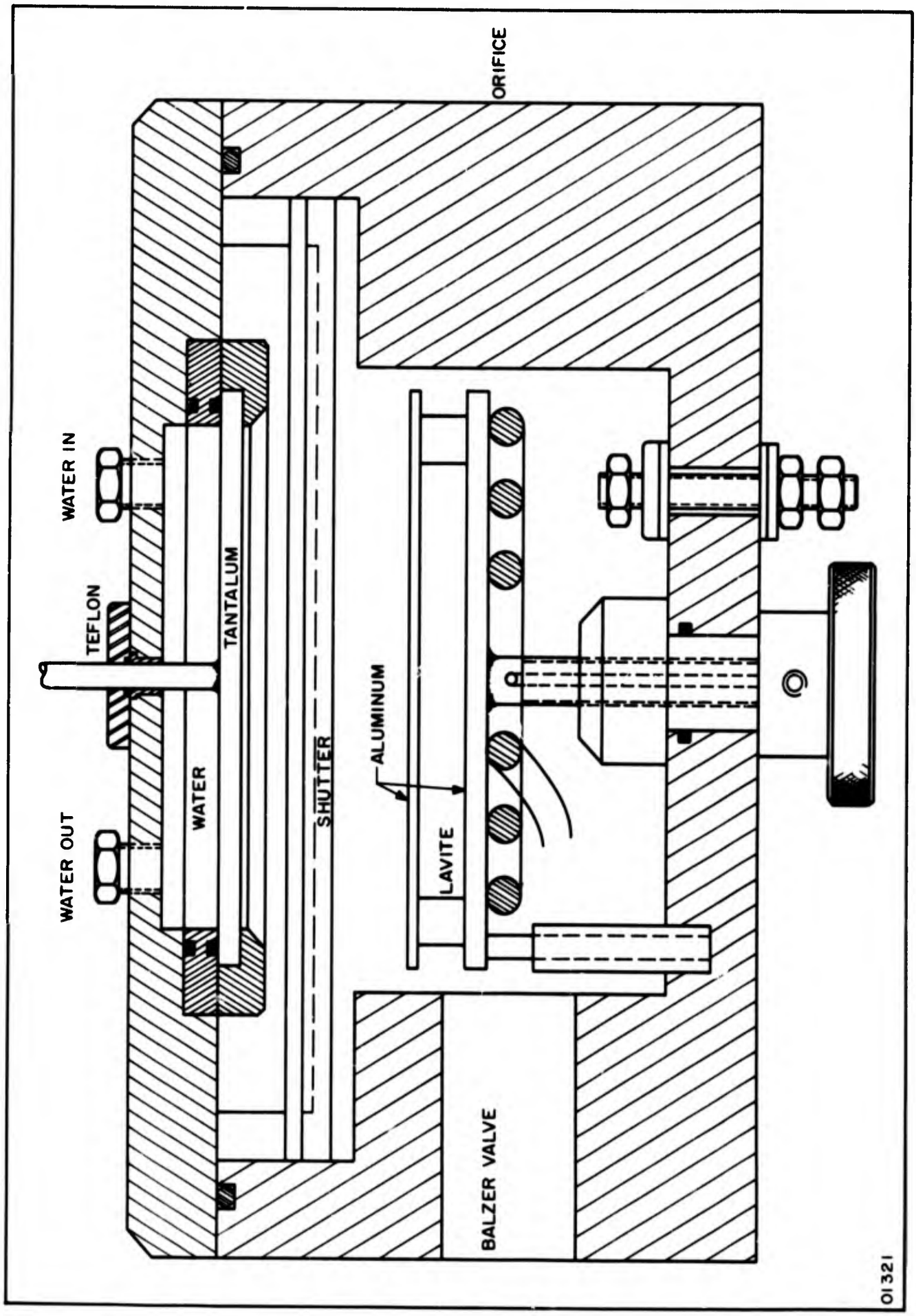


Fig. 9. Clean Dry Sputtering Fixture

The anode position may be adjusted without breaking the vacuum. The shutter design is based on that used for stop diaphragms on cameras and has the merit of being extremely compact.

The pumping system is shown in Fig. 10. This uses a small diffusion pump (speed 20 liters/sec) and a large capacity backing pump (speed 25 m^3/hr) so when large volumes of gas at sputtering pressures are being pumped by the diffusion pump the critical backing pressure is unlikely to be exceeded. The diffusion pump is fitted with a water-cooled baffle to prevent backstreaming but as an additional safeguard, an ion baffle is also used. This device was originally produced for use with rotary backing pumps to prevent backstreaming. The trapping mechanism of the ion baffle occurs when hydrocarbon molecules backstream from the pump in the direction of the vessel being evacuated and an electrical cold cathode discharge created by an inverse magnetron, polymerizes the oi' as a result of ion collision. This causes a solid CH-polymerized layer to form on the surface of the cathode.

A second ion baffle is incorporated in the roughing line to prevent backstreaming while the chamber is being roughed. By using ion baffles and proper selection of pumps, oil contamination backstreaming should be minimized. It is intended to control the system pressure by means of gas admittance and no control is intended by throttling of the diffusion pump.

If this does not afford sufficient control then it will be a simple matter to incorporate a diaphragm with a hole providing a known conductance to reduce the effective pumping speed to a level where gas admittance is a practical control.

The entire vacuum chamber will be mounted inside a dry box to minimize the effects of water vapor contamination so that when the sputtering system is opened to atmospheric pressure for loading, the system will not be exposed to water vapor.

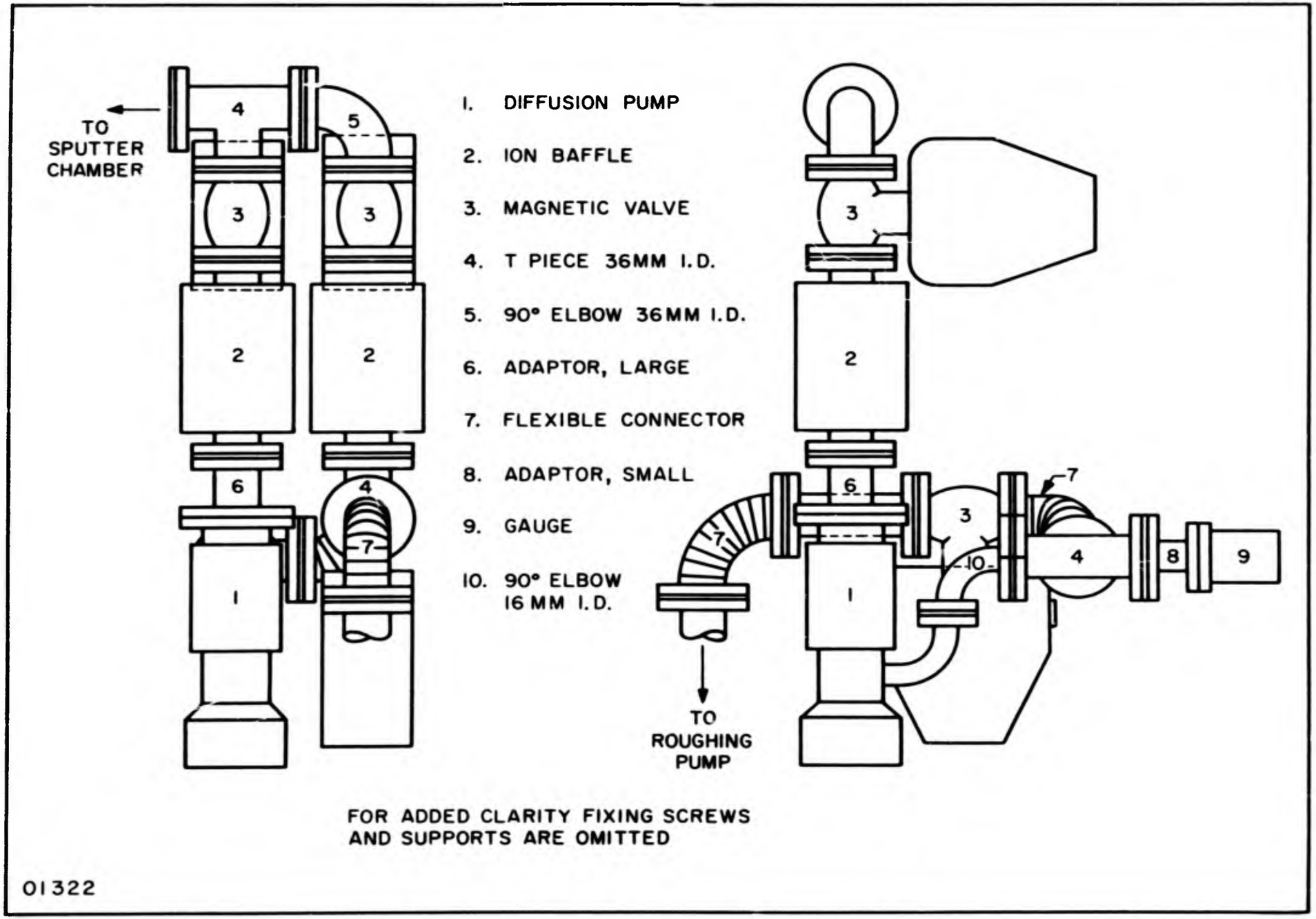


Fig. 10. Pumping System for Sputtering Plant

This procedure will also minimize the effects of dust and prevent finger contamination from reaching the system.

It is expected that the system will be operable in December and the automatic gas dosage and high stability power supply will be in operation during the first quarter of 1966.

B. RESISTANCE MONITORING DURING DEPOSITION

An investigation has been conducted on resistance monitoring slides that can be used to monitor sheet resistance of deposited resistive films in glow discharge. The criteria for the desired monitoring slides are:

1) The monitoring slide must have the same surface characteristics as the substrate being deposited. Ceramic and glass monitoring slides are not acceptable since the surface roughness factors and sticking coefficients are not the same as the oxidized silicon substrate being deposited.

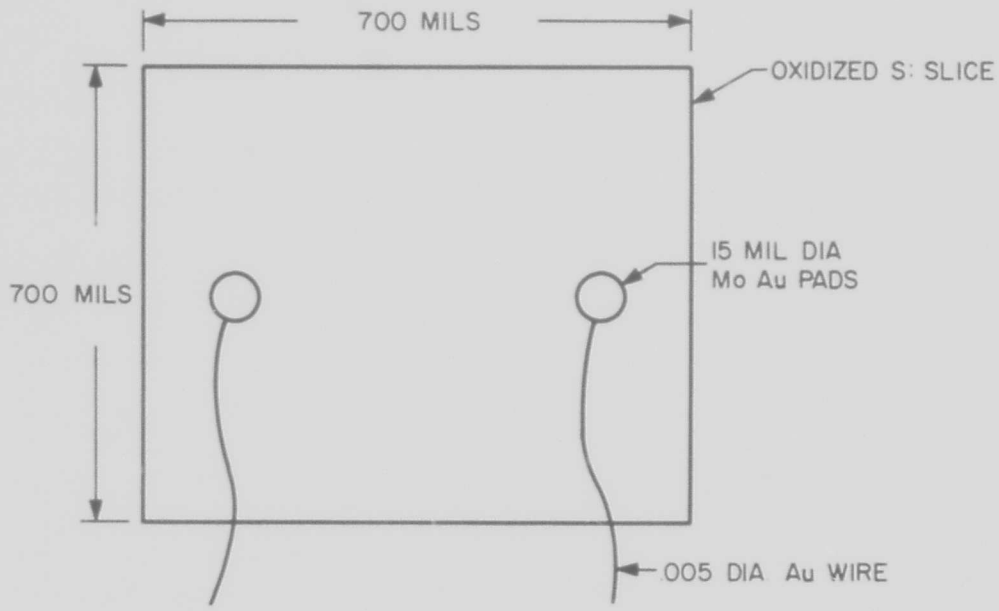
2) The monitoring slide must be small; therefore, not disrupting the charge distribution of the glow discharge. This requirement negates the use of overlay masks since mask holders are bulky and would disrupt the charge distribution.

3) The slide must be inexpensive and readily replaceable.

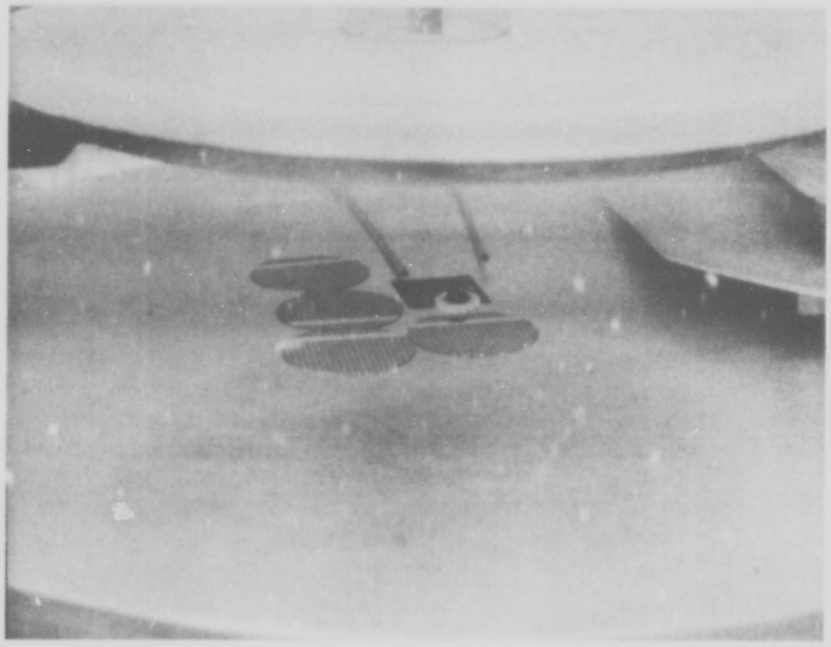
4) The resistance monitoring equipment should be accurate and the circuit should operate at low currents in order not to affect the resistance of the monitoring slide.

5) The monitoring slide should operate continually during the deposition in glow discharge.

The monitoring slide shown in Fig. 11 fulfills all of the above requirements except for the last one. A 700-mil square slice is scribed out of a rejected silicon slice, thus making use of inexpensive starting material. The silicon is thermally oxidized and the monitoring slices have the same surface characteristic as the substrates



(A) SCHEMATIC



(B) PICTORIAL

01323

Fig. 11. Resistance Monitoring Slide

to be deposited. Molybdenum gold is evaporated and etched providing ohmic contacts for the sputtered film. Since a photo-etch step is used to define the ohmic contacts, the contacts are located in the same place on each slide.

Five-mil diameter gold wire is soldered to the contacts and connected to a Non-linear System, Series 4800 Digital Ohmmeter. Since the ohmmeter is a self-balancing wheatstone bridge with guard circuit, a maximum of 6X can be applied to the monitoring slide which will not alter the deposited film properties.

The criteria that could not be met for the optimum monitoring slide is the ability for continuous monitoring. The glow discharge establishes a field across the slide, thus giving a fallacious measurement. The operation that works satisfactorily for monitoring is to deposit for 15 seconds, stop the glow, turn on the monitoring slide, deposit for 15 seconds, etc. The sheet resistance is monitored every 15 seconds which allows adequate control.

Figure 12 shows data points for films deposited at 200 Ω/□ and 1000 Ω/□. The reading on the monitoring slide is shown to be slightly less than one square. The sheet resistance readings were taken after the deposited films had been etched and processed for ohmic contacts.

This technique has proven both accurate and useful for monitoring deposited resistances.

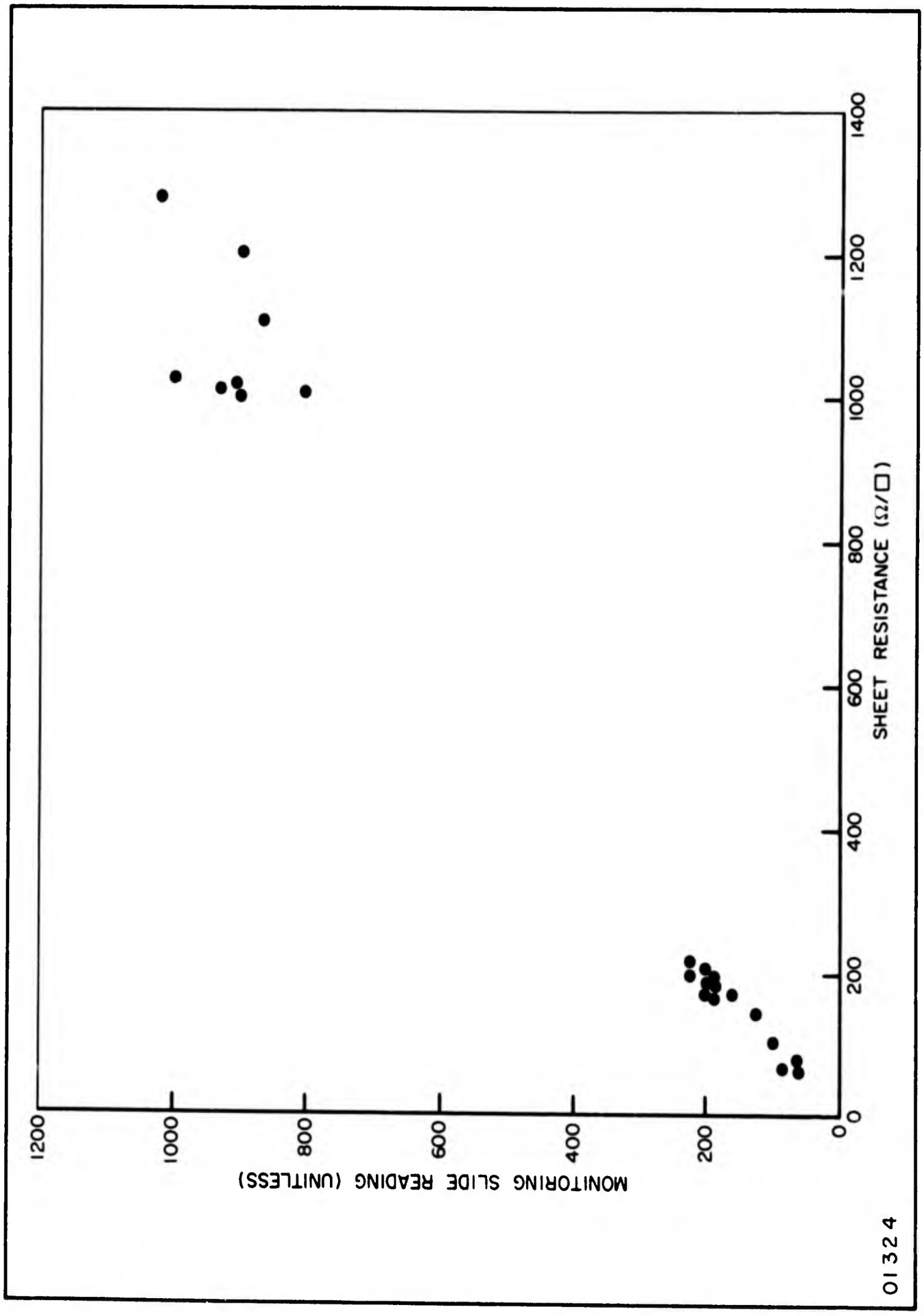


Fig. 12. Monitoring Slide Readings vs Sheet Resistance

SECTION III

OXIDATION EXPERIMENTS ON Ta/Ta_2O_5 THIN FILMS

A. GENERAL

Reactively sputtered Ta/Ta_2O_5 thin-film resistors show large increases in resistance when heat-aged in air. The work reported here is an attempt to clarify the oxidation mechanisms both at the surface and at grain boundaries in the bulk of the film. The results of oxidation experiments are compared with the results obtained on tantalum films reported in the literature.

B. EXPERIMENTAL WORK

Ta/Ta_2O_5 films were prepared under the conditions outlined in Table 1. Resistors having an initial value of about 1 k were made from the 25 Ω/□ material, and about 10 k from the 300 Ω/□ films. These resistors were then oxidized in air at various temperatures. Figure 13 shows the result of these heat agings. It is informative to propose a simple model to see if the growth of tantalum pentoxide on the surface of these films will account for the large changes in resistance. Figure 14 shows how Ta_2O_5 might grow on top of the film. Notice that the film increases in thickness as pentoxide is formed. This is due to the high ratio of molar volume of pentoxide to tantalum.[1/] The thickness of oxide (y) is related to the thickness of tantalum (x) by the relation

$$y = 2.5\ (x_o - x)$$

Table 1. Summary of Sputtering Conditions for the Ta/Ta_2O_5 Resistors Used in Heat Age Experiments

Results Reported In	Approximate Sheet Resistance and Thickness	Sputtering Rate	Total Pressure During Sputtering	Cathode Voltage and Current	Flow Rate Settings 99.99% Ar 0.01% O_2	Flow Rate Settings 100% Ar
	25 Ω/□, 7000 Å	400 Å/Min.	~ 45 μm	-3000 V 100 mA	14	100
	300 Ω/□, 1100 Å	120 Å/Min.	~ 45 μm	-2900 V 100 mA	70	0
	200 Ω/□, 1700 Å	450 Å/Min.	~ 45 μm	-2900 V 100 mA	70	0
X-Ray Scan Data, Samples 1, 2, 3, 4	200 Ω/□, 1700 Å	450 Å/Min.	~ 45 μm	-2900 V 100 mA	70	0

In all runs the anode was biased -20 V with respect to ground.

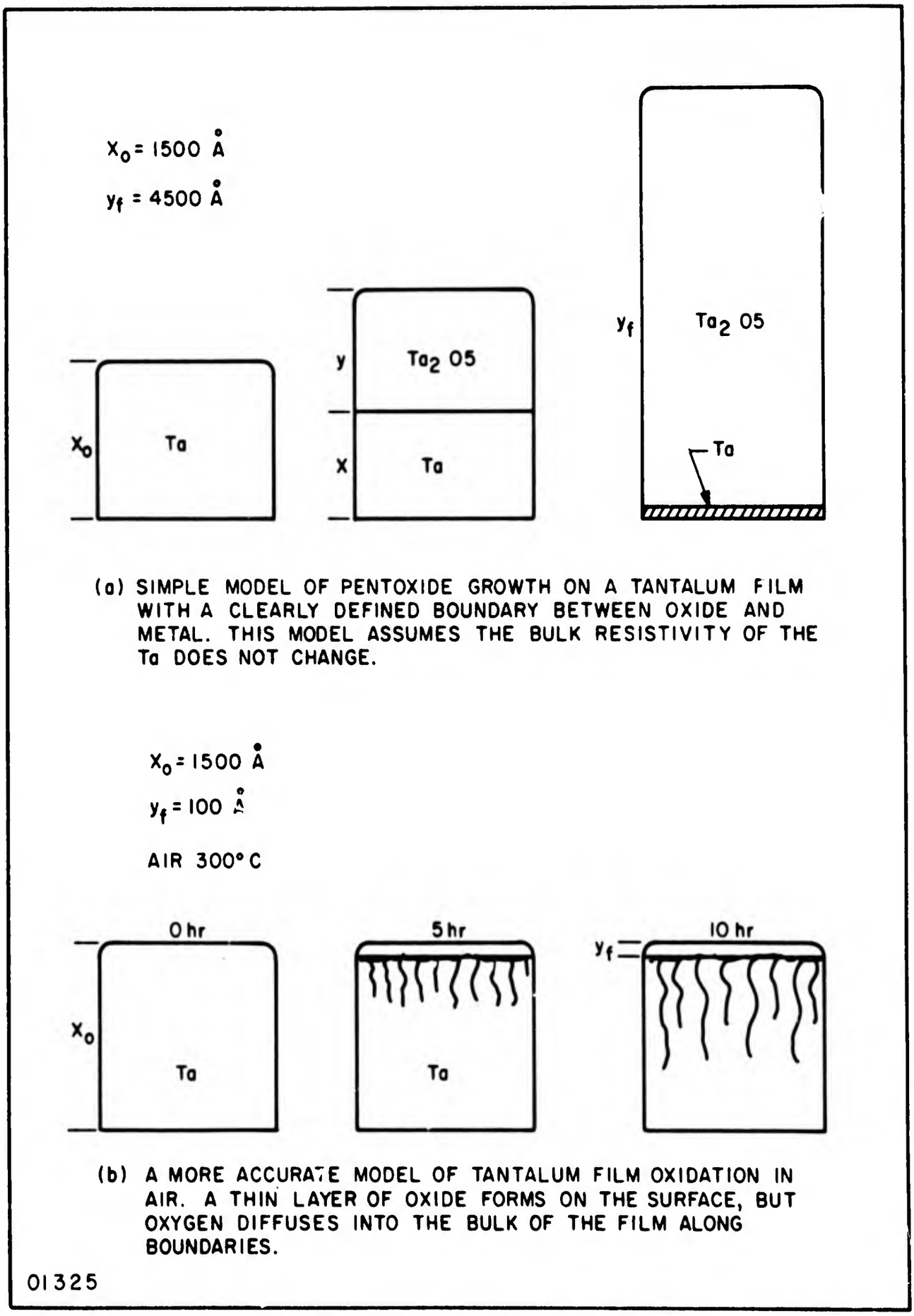


Fig. 13. Tantalum Oxidation Modules

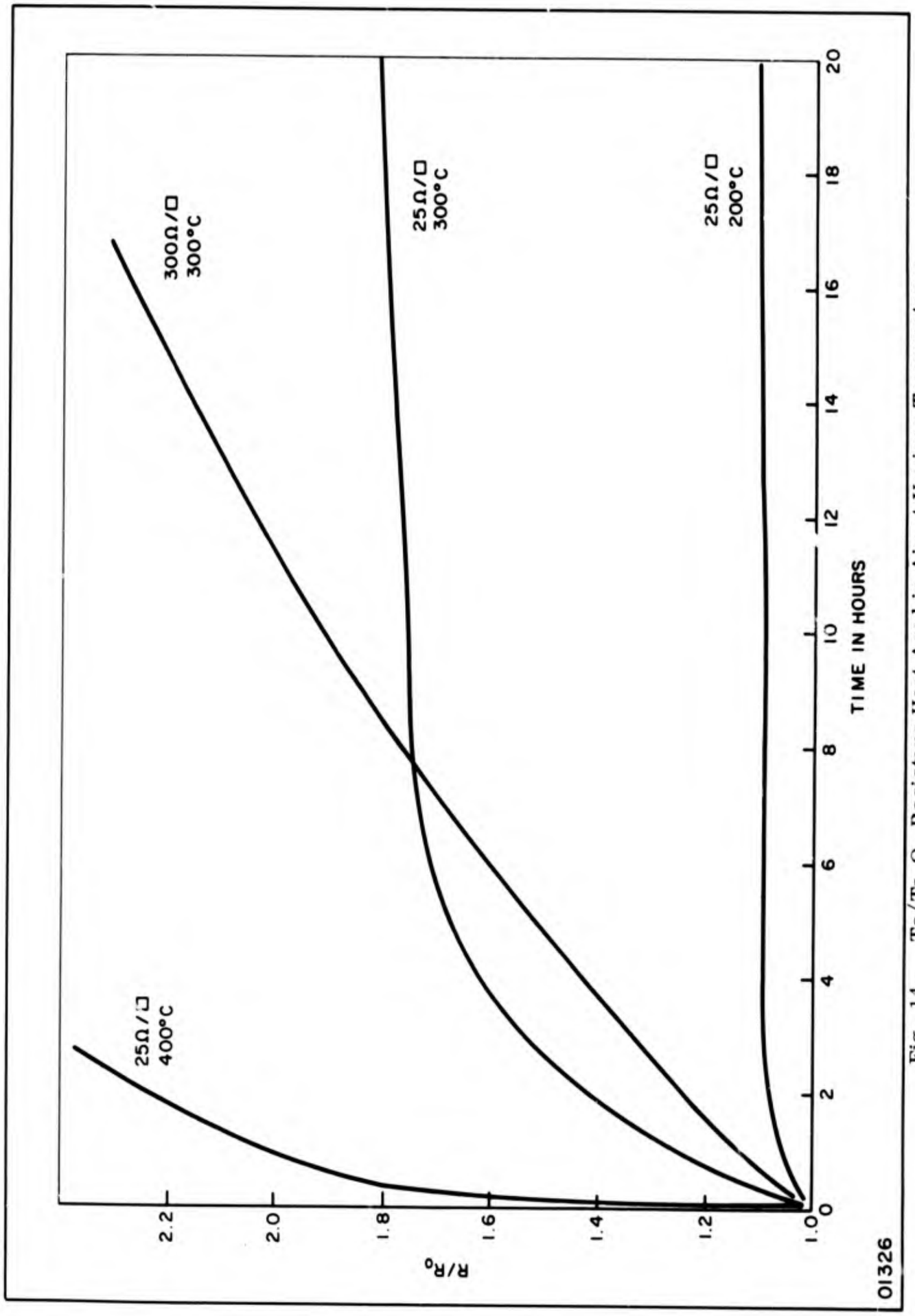


Fig. 14. Ta/Ta_2O_5 Resistors Heat Aged in Air at Various Temperatures

Using this formula, the resistance of the film as a function of y, assuming the pentoxide has zero conductivity, would be:

$$\frac{R}{R_o} = \frac{1}{1 - y/2.5\, x_o}$$

where R_o is the starting resistance, and x_o the thickness of the tantalum film before oxidation. For the resistance of the film to increase by a factor of two, the oxide would have to be $1.2\, x_o$ thick. This would require an oxide thickness after 10 hours of heat aging of 1700 Å on the 300 Ω/□ sample shown in Fig. 14. Published data on the oxidation of Ta would predict an oxide of no more than 100 Å. This value is based on oxidation work by Vermilyea.[2] Basseches[3] has done oxidation work on sputtered Ta films and reported oxide growth on the sputtered films may be slower by as much as a factor of 10 compared to bulk Ta. The 100 Å figure is then an upper limit on the oxide thickness of a Ta film heat aged for 10 hours at 300°C.

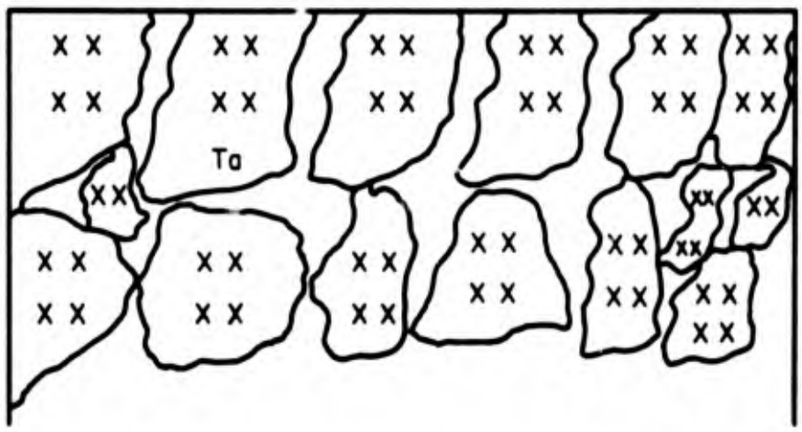
Obviously surface oxide formation alone does not account for the large changes in resistance on heat aging in air. Oxygen can diffuse into the grain boundaries of the sputtered films forming Ta_2O_5, thus creating high resistance paths. Hirai[4] has measured the conductivity of sputtered Ta films as a function of depth from the surface of the film. His measurements show that the bulk resistivity of a 400 Å Ta film increased by a factor of two when heat aged in air for five hours at 300°C. An oxide layer of 70 Å was formed on the surface of the film. Hirai concludes that oxygen diffusing into grain boundaries, rather than surface oxide formation is responsible for the large changes in resistance. Experiments by Maissel[5] support this conclusion. A more reasonable model for oxidation by air is shown in Fig. 13b.

C. HIGH VACUUM HEAT AGE

Oxidation experiments in air point out that pentoxide formation at grain boundaries cause large increases in resistance. In Ta films at low oxygen concentrations, oxygen atoms are incorporated interstitially into the tantalum lattice. At higher concentrations, Ta_2O_5 begins to form in layers around small Ta grains, as shown in Fig. 15.[6] Another oxidation mechanism might be oxygen diffusing out of the Ta grains to form larger volumes of Ta_2O_5. This additional oxide would decrease the number of conduction paths, increasing the resistivity of the films.

To confirm this, heat aging runs on reactively sputtered Ta films at $< 5 \times 10^{-6}$ Torr were performed. Electron diffraction patterns and X-ray reflection scans of these sputtered films before heat aging showed the films to be largely noncrystalline with trace amounts of Ta_2O_5. If heat aging in high vacuum produced more, and stronger Ta_2O_5 peaks in the X-ray scans, it would indicate that more pentoxide was forming in the film. Figure 16 is a facsimile of three X-ray diffraction scans. The top scan is of bulk Ta metal. This metal was then oxidized in air at 300°C. The middle scan is of this oxidized sample. The new peaks are identified as Ta_2O_5, since they agree well with published X-ray spectra of this oxide. The bottom scan is of a sputtered Ta film heat aged in vacuum at 300°C for 17 hours. Several peaks in this spectra agree with the Ta_2O_5 spectra. Because of preferential orientation of the film on the substrate, not all the pentoxide peaks are observed.

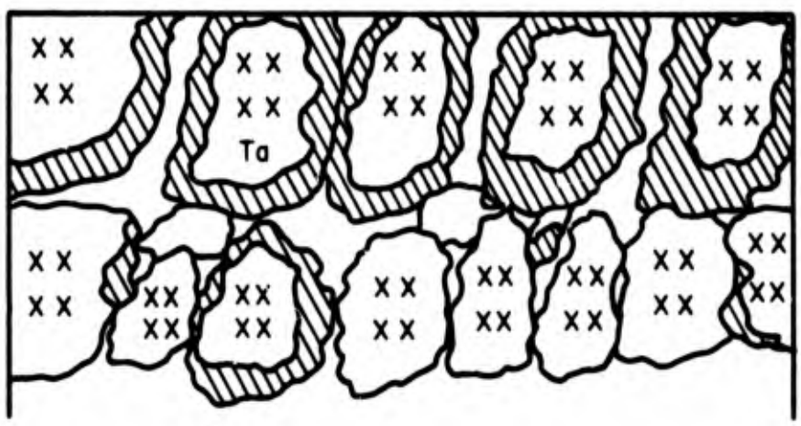
Table 2 summarizes the results of X-ray analysis on bulk Ta metal and sputtered Ta films heat aged in vacuum. The large number of Ta_2O_5 peaks seen in the film spectra (samples 2 and 3) are evidence that oxygen incroporated interstitially into these films while sputtering, forms additional volumes of Ta_2O_5 when the films are heat aged at temperatures above 250°C. Figure 17 shows the change in resistance of 200 Ω/□ material heat aged at $< 5 \times 10^{-6}$ torr at different temperatures. The increase in resistance is attributed to pentoxide formation in the film.



INTERSTITIAL OXYGEN - X

AVERAGE GRAIN SIZE - 150 °A
AVERAGE GRAIN SEPARATION - 10 °A
AT LOW OXYGEN CONCENTRATION DURING SPUTTERING ($<10^{-5}$ TORR)
OXYGEN IS INCORPORATED INTERSTITIALLY INTO THE Ta GRAINS.

(A) LIGHTLY DOPED Ta FILM



INTERSTITIAL OXYGEN - X
Ta_2O_5 -

AT HIGH OXYGEN CONCENTRATION ($>10^{-4}$) Ta_2O_5 FORMS IN LAYERS
AROUND THE Ta GRAINS

(B) HEAVILY DOPED Ta FILM

01327

Fig. 15. Oxygen Doped Tantalum Films

Table 2. Summary of X-ray Spectra Peaks Observed in Different Samples
d-spacings in Å

Si	3.13												1.043	
Ta			2.338				1.653			1.35	1.168			0.954
Ta_2O_5		2.36		2.02	1.80	1.582		1.551	1.427			1.215		
1	X			X[w]				X*					X	
2	X	X		X					X		X	X	X	
3	X	X		X				X*	X		X	X	X	
4	X							X*					X	
5			X				X				X			
6			X	X	X	X	X		X	X	X			X

1. Oxygen doped Ta film, 300 Ω/□, 1200 Å. No heat aged.
2. Sample (1), but heat aged 5 hr at 300°C in vacuum (< 5×10^{-6} Torr).
3. Sample (1), but heat aged 17 hr at 300°C in vacuum (< 5×10^{-6} Torr).
4. Sample (1), but heat aged 15 hr at 200°C in vacuum (< 5×10^{-6} Torr).
5. Ta metal substrate.
6. Ta metal substrate heat aged in air for 15 hr at 300°C.

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w - indicates weak peak.

* - The 1.551 Å peak identified as Ta_2O_5, may be a peak at 1.56 Å sometimes seen in single crystal Si.

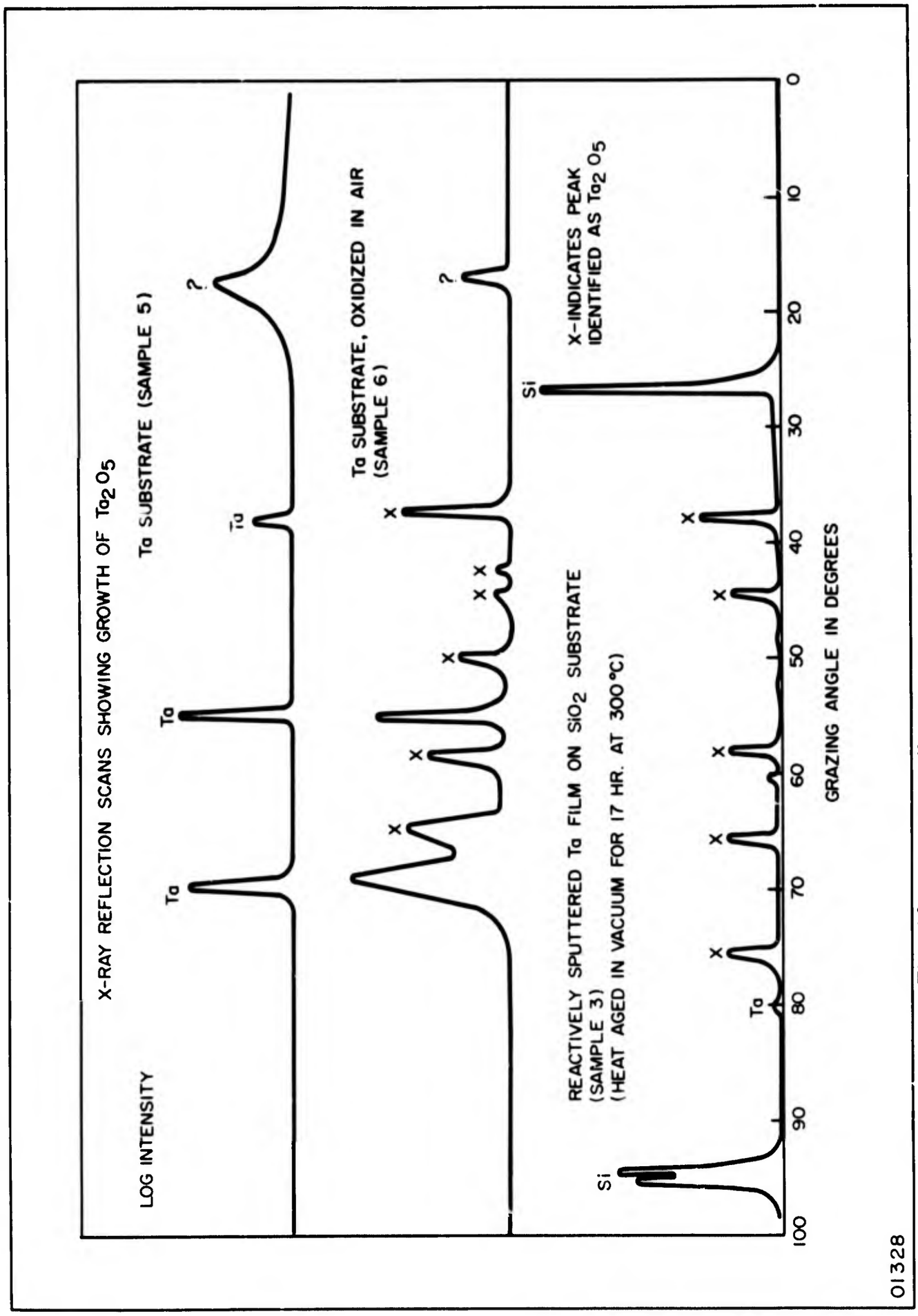

Fig. 16. X-Ray Diffraction Scans Showing Growth of Ta_2O_5

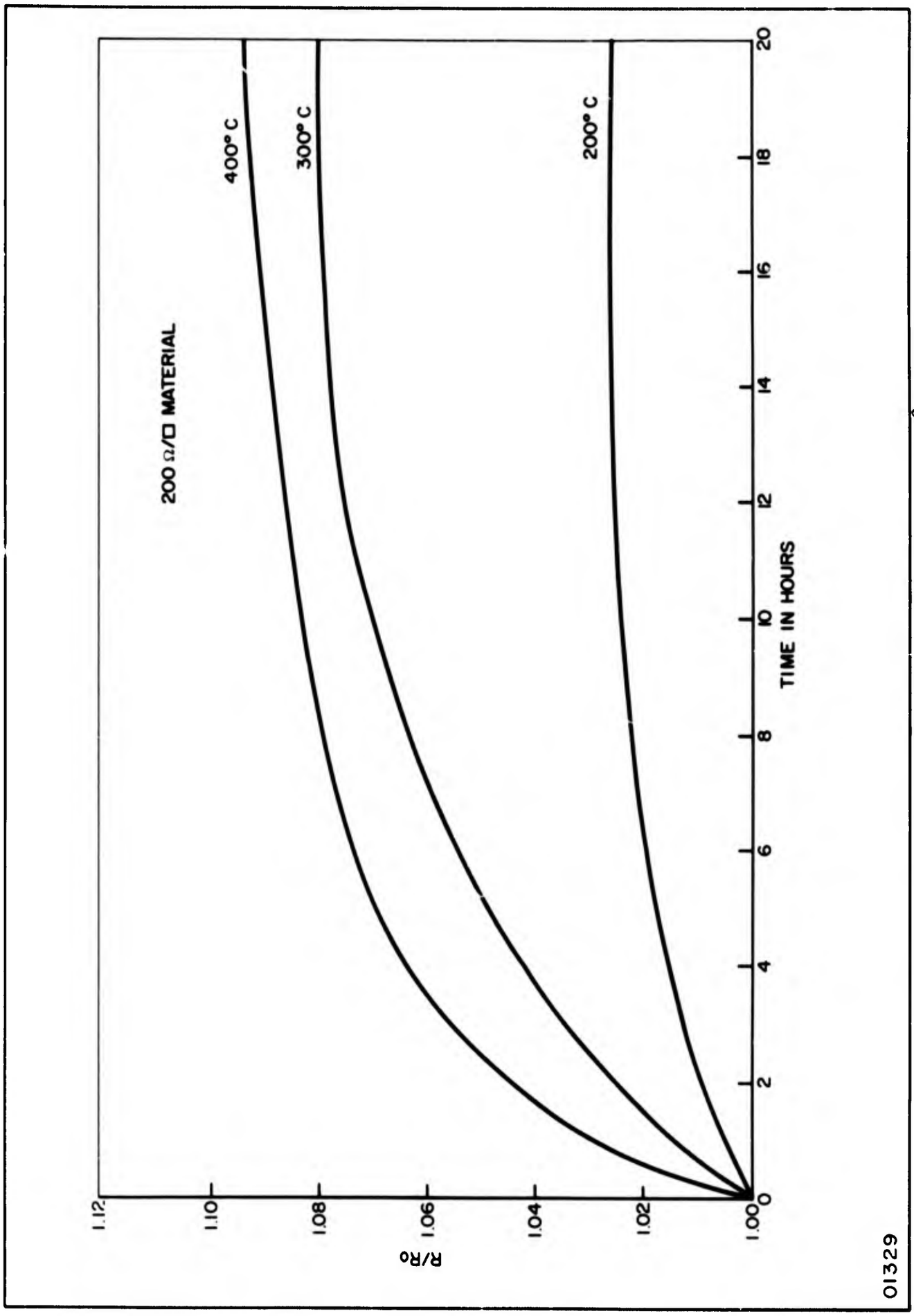

Fig. 17. Tantalum Resistors Heat Aged at $< 5 \times 10^{-6}$ Torr

The X-ray spectra of some samples, notably (3), appeared to fit published spectra of Si_3Ta_5. The peaks of Si_3Ta_5 and Ta_2O_5 are quite close. Si_3Ta_5 was ruled out for the following reason.

To have the intensity that was observed in these scans, the unknown would have to be a major constituent in the film (on the order of five per cent of the film volume). The Ta films were sputtered on Si slices that had at least a 2000 Å layer of SiO_2 on the surface. To form Ta_3Si_5 one must propose the following reaction, or admit the possibility of very large cracks in the oxide:

$$37\ Ta + 15\ SiO_2 \rightarrow 6\ Ta_2O_5 + 5\ Si_3Ta_5$$

The free energy of this reaction can be calculated using published values of ΔF°[7] and ΔH°[8]. The calculated value of ΔF for the reaction is + 2 K cal/mole Ta at 25°C and + 4 K cal/mole Ta at 400°C. This positive value of ΔF indicates the reaction is not favorable to go in the direction indicated. These calculations are supported by Revesz[9] who states that the reduction of SiO_2 by a metal is not probable.

D. CONCLUSIONS

The diffusion of oxygen into grain boundaries of sputtered tantalum films is a major source of resistivity increase when these films are heat aged in air at temperatures above 200°C. Interstitial oxygen incorporated into the film during reactive sputtering forms additional volumes of pentoxide when these films are heated. These pentoxide volumes reduce the number of conduction paths in the film, thereby increasing the resistivity.

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SECTION IV

PROCESS CONTROL INVESTIGATION

A. GENERAL

A process control investigation has been conducted to determine the ability to reproduce a set process for fabricating 1000 Ω/□ resistors and > 0.35 pF/mil^2 capacitors. The objective of the investigation is to complete the combined resistor and capacitor process on oxidized silicon substrates, determining yields and process problems. The process was terminated at the final resistor stabilization bake process immediately prior to scribing and mounting.

B. PROCESS FOR RESISTORS AND CAPACITORS

The process used in this investigation is:

1) Starting material - P-type silicon slices 1.00 inch in diameter.
2) Thermal oxidation of silicon - 8000 Å thick.
3) Clean slices - 180°C H_2SO_4 and boiling HNO_3.
4) Bake - vacuum at 180°C - 2 hours.
5) Evaporate aluminum - 200°C substrate temperature, 5000 Å thick.
6) Etch capacitor bottom plates in H_3PO_4, HNO_3, HA_c etchant at 40°C.
7) Reactively sputter Ta_2O_5 dielectric
   - Voltage - 2000 V
   - Current - 100 mA
   - 50% O_2 and 50% Ar
   - Pressure - 50 μm

Cathode anode spacing - 1.75 in.

Time - 260 min.

Anode bias - 0

Cathode diameter - 2.75 in.

8) Etch holes in Ta_2O_5

3 parts HF

2 parts HNO_3

7 parts HA_c

9) Reactively sputter Ta/Ta_2O_5 resistive material

Voltage - 2600 V

Current - 150 mA

1000 ppm O_2 in Ar

Pressure - 80 μm

Cathode anode spacing - 1.75 in.

Time - 9 min.

Anode bias - 20 X

Cathode diameter - 6.5 in.

10) Etch resistive film with

3 parts HF

2 parts HNO_3

19 parts HA_c

11) Evaporate aluminum - 5000 Å thick

12) Etch capacitor top plates and resistor contacts with H_3PO_4, HNO_3, HA_c etchant.

13) Etch taps - resistor adjustment

The process steps (5) through (8) are eliminated when resistors are being fabricated without capacitors.

C. 1000 Ω/□ Ta/Ta_2O_5 RESISTORS

The requirement for the thin-film resistor is fabrication of a stable 1000 Ω/□ resistor with a tolerance of ± 20 per cent. The test vehicle bar layout is shown in Fig. 18 and the resistor marked R_1 is the resistor tested in most of the investigation. Resistor R_1 ix 160 mils long and 1.0 mil wide yielding a resistor value of 160,000 Ω.

In order to accurately estimate the process control, six lots of material were processed. All lots were processed for 1000 Ω/□ resistors and two lots were processed for resistors and capacitors.

Figure 19 shows the results of the stabilization bake for three of the six lots. Since the resistors are designed for 160,000 Ω ± 20 per cent, the area between the dashed lines of Fig. 19 is the objective for the final average resistance of the slices.

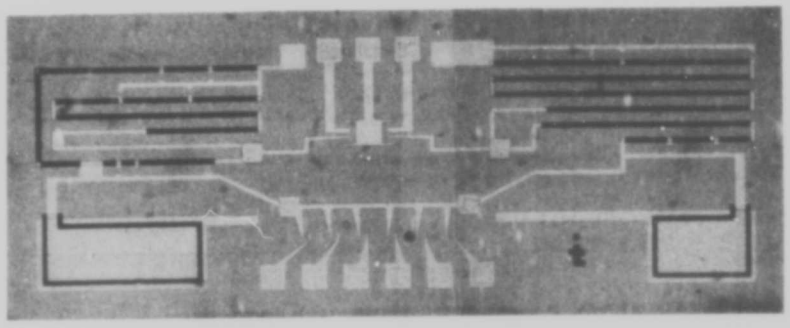

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Fig. 18. Test Vehicle for 1000 Ω/□ and > 0.35 pF/mil^2 Thin-film

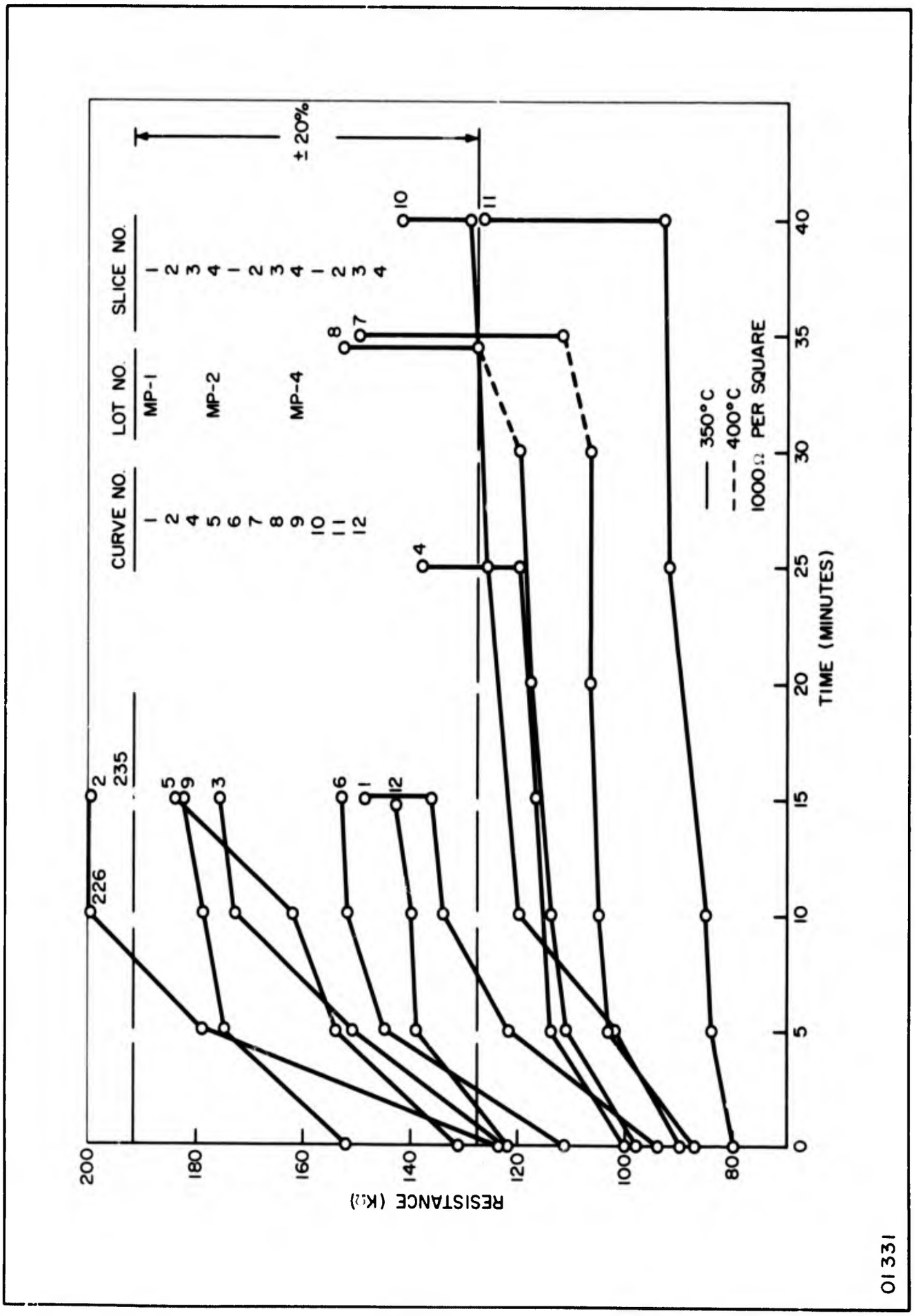


Fig. 19. Ta/Ta_2O_5 Resistor Stabilization Bake

The data was taken in the following manner:

1) 10 resistors on each slice were tested after contacts were defined and before the stabilization bake.

2) The slices were baked at five or 10 minute increments and the same ten resistors tested previously were again tested, averaged and plotted in Fig. 19.

3) At approximately 15 minutes cumulative bake time, the resistors appeared stable and were tested again. 25 resistors were tested for the final average.

4) If the sheet resistance was low, slices 4, 7, 8, 10 and 11, the slices were baked for longer times and tops removed.

5) The top removal process is accomplished by a KMER-etch step where 15 per cent or 30 per cent of the resistance is added to each resistor on each bar on the slice. This is illustrated in Fig. 20 for one resistor. If tap 1 is etched and removed, 15 per cent additional resistance is added to the resistor and if taps 1 and 2 are etched and removed, 30 per cent additional resistance is added to the resistor. The result of this step is shown in Fig. 19 by the vertical lines for slices 1, 4, 7, 8, 10 and 11.

At the end of the stabilization bake and tap removal process, the resistor yields and distributions were taken.

The cumulative yields for the six lots is 62 per cent as shown in Table 3 of the resistors at 1000 $\Omega/\square$ $\pm$ 20 per cent. Lots three and five had a total of three slices that the yield is 0 per cent. In an actual process, the resistive films would be stripped from these slices and redeposited, thus increasing the over-all yield.

An important observation to make is how the failure resistors are lost. That is, do the resistors fail randomly or do the resistors fail in groups? The importance is realized by a simple calculation of the two conditions,

1) If the resistors fail randomly then the over-all yield of 62 per cent must be raised to the power of the number of resistors that will be designed into an integrated circuit bar to calculate the bar yield. This follows since if one resistor fails, then the integrated circuit bar fails. If three resistors are designed into the circuit the yield for resistors would be 24 per cent.

Table 3. 1000 Ω/□ Ta/Ta_2O_5 Resistor Process Yields

Objective: 1000 Ω/□ ±20%
160 000 Ω ±20%

Lot Number	Slice Number	No. Units Tested	Yield ±20%	R(Ω)
MP-1	1	25	76	152 000
	2	25	52	196 000
	3	25	88	167 000
	4	25	100	149 000
	TOTAL	100	79%	
MP-2	1	25	56	197 000
	2	25	72	161 000
	3	25	96	155 000
	4	25	92	158 000
	TOTAL	100	79%	
MP-3	1	25	52	205 000
	2	25	28	179 000
	3	25	0	–
	TOTAL	75	27%	
MP-4	1	25	68	177 000
	2	25	92	143 000
	3	25	52	127 000
	4	25	88	159 000
	TOTAL	100	75%	
MP-5	1	25	0	418 000
	2	25	0	503 000
	3	25	84	174 000
	TOTAL	75	28%	
MP-6	1	25	56	141 000
	2	25	68	141 000
	3	25	86	140 000
	TOTAL	75	70%	
CUMULATIVE TOTAL		525	62%	

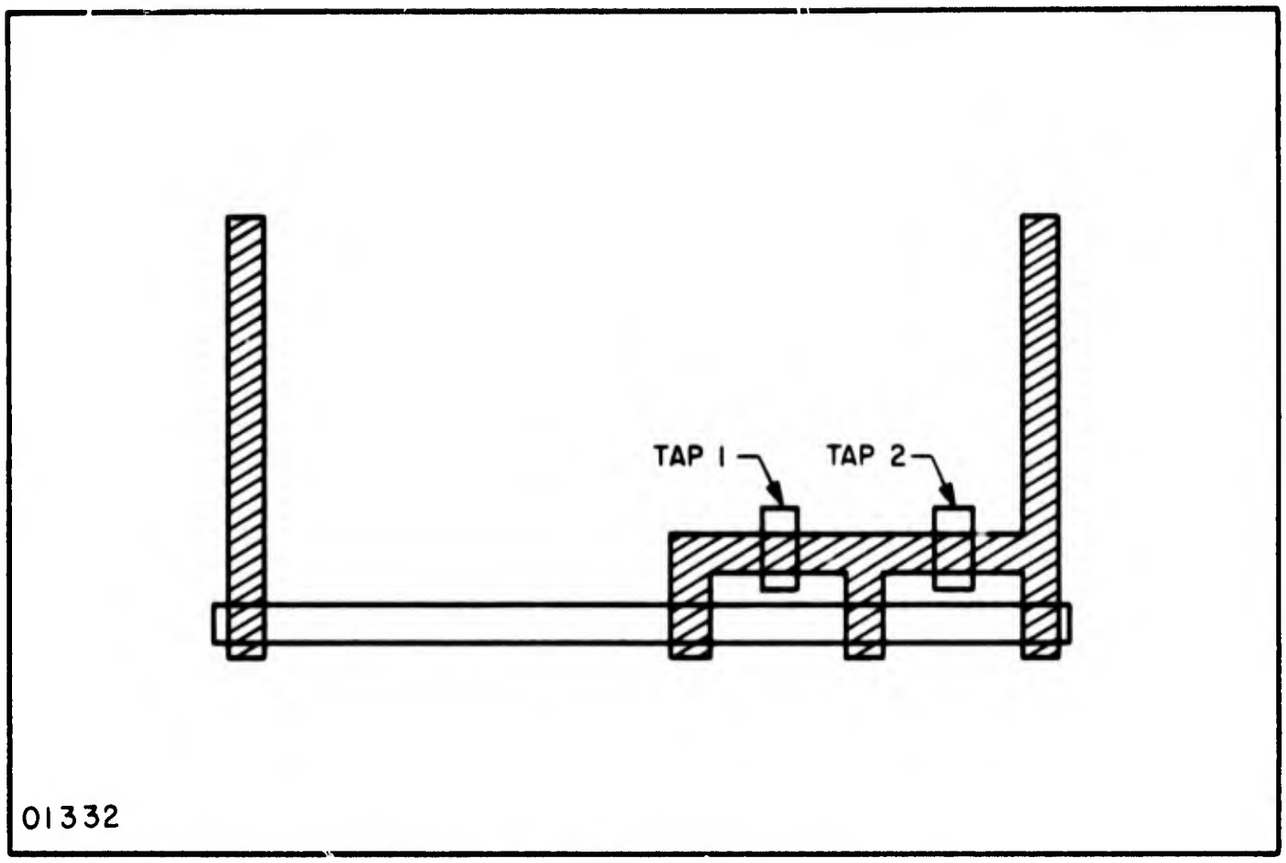
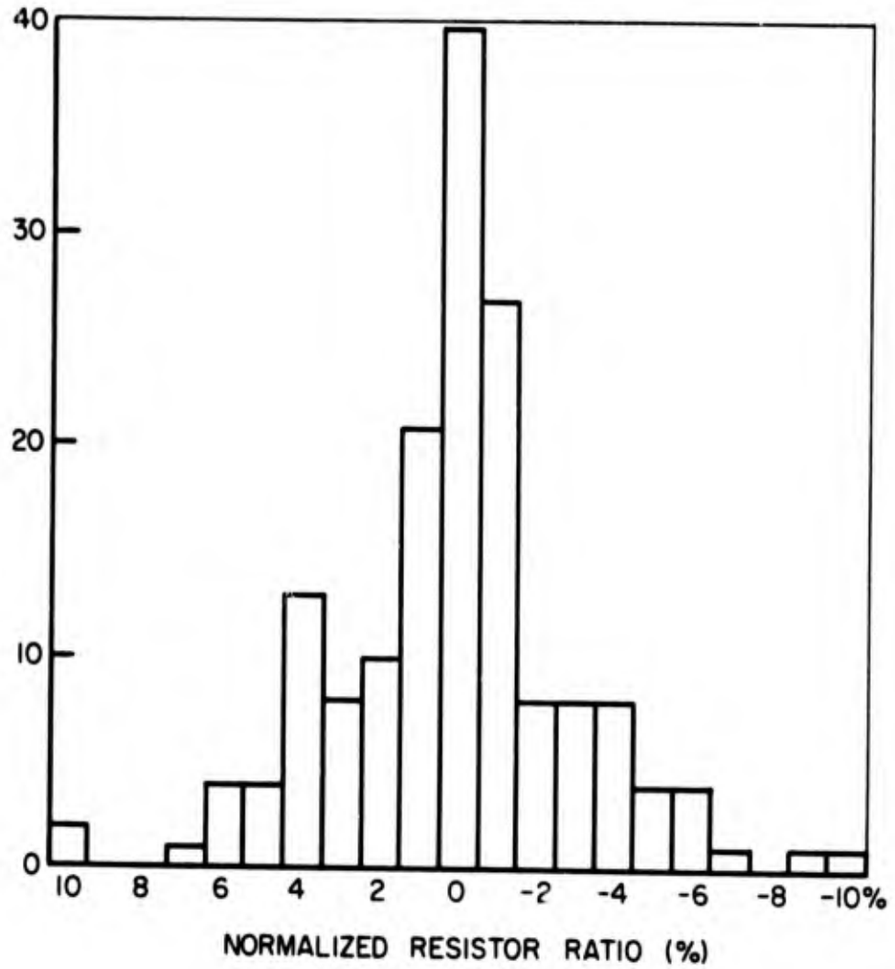


Fig. 20. Thin-film Resistors with Taps

2) If the resistors fail in groups, which will be shown to be true, then the fabrication of two or more resistors instead of one will add little to the yield loss. The test vehicle had three resistors that were convenient to test.

Table 4 shows test results from testing three resistors/circuit on four slices. The test yield for the one 160,000 Ω is 72% and for the three resistors is 68%. if a straight line relationship is assumed for yield degradation per added resistor, then the figure of 2 per cent/resistor can be assumed to be an approximate figure.

Figure 21 shows a histogram of normalized resistor ratios indicating that on each hybrid monolithic circuit bar, the majority of all the resistors have a ratio within three per cent of the designed value. This is more readily shown in Fig. 22 which illustrates the yield to a particular resistor ratio requirement. The dotted line indicates that 92 per cent of the ratios determined are less than ± 5 per cent of the designed value.



RATIO OF 1000 Ω/□ Ta/Ta_2O_5 RESISTORS:
FOUR RESISTORS/CIRCUIT:
RATIOS $R_1/R_2 \cong 2.8$
$R_1/R_3 \cong 4.1$
$R_1 = 160{,}000\ \Omega$

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Fig. 21. Histogram of Normalized Resistor Ratios

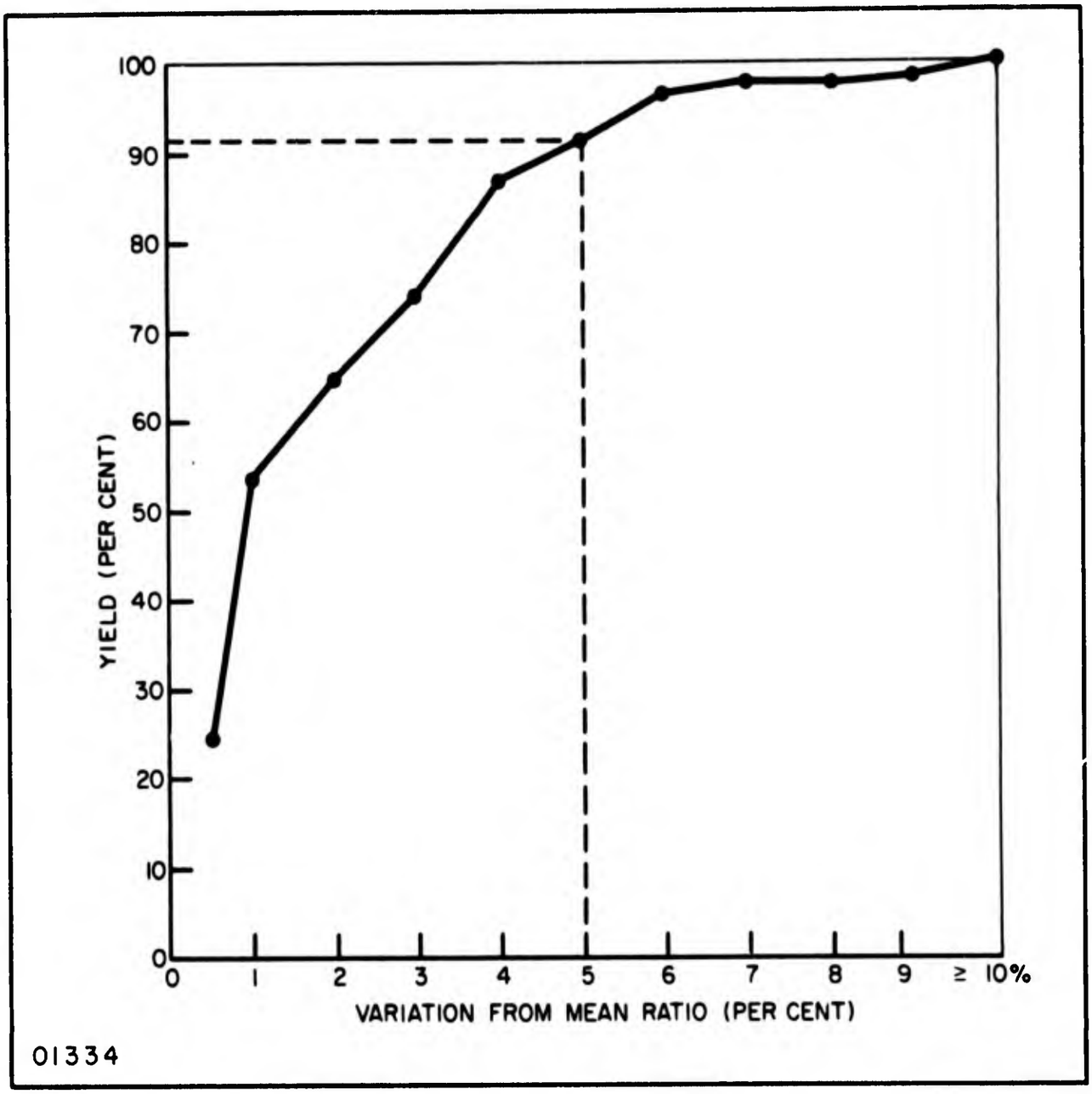

Fig. 22. Resistor Yields for Various Resistor Ratios

D. 1000 Ω/□ RESISTORS AND 0.35 pF/MIL^2 CAPACITORS

Lots five and six were processed for both thin-film resistors and capacitors. The deposited dielectric was designed for 0.45 pF/mil^2 since the specification is >0.35 pF/mil^2. Table 5 shows the yields for the two lots. The resistor yield is 46 percent, capacitor yield 85 percent, and the cumulative yield is 38 percent.

It is important to note that the resistor yield is 46 percent as compared to 62 percent for the average of the six lots tested for resistors only. This indicates that

a reduction in resistor yields is expected when both resistors and capacitors are fabricated on the same slice.

Table 6 shows the results from the resistor stabilization bake. The capacitors were given a 45-minute bake cycle and the capacitance, df, and voltage breakdown are not significantly different. Also, the yield is 90 percent before and after bake, indicating no catastrophic yield reduction.

Table 4. Yields for Four Resistors

1000 Ω/□ Ta/Ta_2O_5 Resistor						
Lot	Slice	No. Units Tested	Yield			
			R-1	R-2	R-3	Bar
MP-36	1	75	44	48	72	44
	2	75	100	100	100	100
	3	75	58	46	52	44
	4	75	84	96	92	84
		300	72%			68%

Yield estimate based on R-1 is 72 percent and actual yield is 4 percent lower.

Table 5. Compatible Resistor and Capacitor Processing

Combined Thin-film Resistor/Capacitor Process Yields Objective: Resistors 1000 Ω/□ ±20% Capacitors 0.35 pF/mil^2 > −20%								
Resistors					Capacitors			
Lot	Slice	No. Units Tested	Yield ±20%	R(Ω)	No. Units Tested	Yield ±20%	Cap (pF/mil^2)	Cumulative Yield
MP-5	1	25	0	418 000	20	90	0.45	0
	2	25	0	503 000	20	90	0.44	0
	3	25	84	174 000	20	90	0.43	76
TOTAL		75	28%		60	90%		25%
MP-6	1	25	56	141 000	20	90	0.43	50
	2	25	68	141 000	20	90	0.44	61
	3	25	86	140 000	20	60	0.48	52
TOTAL		75	70%		60	80%		54%
CUMULATIVE TOTAL		150	49%		120	85%		38%

Table 6. Ta_2O_5 Capacitors – Heat Age

Heat Age: 10 min, 300°C 35 min, 350°C Air					
Condition	No. Units Tested	Cap pF/mil^2	V_{BD}	df	Yield (%)
Before Heat Age	20	0.48	68	0.007	90
After Heat Age	20	0.50	77	0.01	90

SECTION V

RESISTOR THERMAL TRIM AND STABILIZATION

Thermal trim and stabilization techniques have been investigated to provide a means of gross adjustment of resistor values while stabilizing the resistive films. Two techniques have proven adequate for gross adjustment of resistor values and one technique has shown to be satisfactory for both trimming and stabilizing the resistive films.

The first technique involves heating a slice by infrared in pulses of 20- to 30-second duration. This has been reported by Kuo[10] for adjusting resistor values. Although the technique is quite adequate for rapid gross adjustments of resistor values, stresses are set up in the film and resistance change on life test is greater than for the resistors from the same lot that were not heat treated. This is shown in Fig. 23.

The second method for thermally trimming the resistors is to provide a bake cycle at 300°C, 350°C, or 400°C, depending on the resistance change required. The temperature of the bake determines how much the resistance changes. The mechanism for effecting a resistance change is oxidation of the film surface, oxidation along grain boundaries, and annealing structural defects. This process inadvertently stabilizes the resistive film against further change on life test. Resistors stabilized in this manner are shown in Fig. 23 and change much less in resistance than either the infrared stabilized resistors or the unstabilized resistors.

The high-temperature bake technique shows promise for both adjusting resistor values and stabilizing the films.

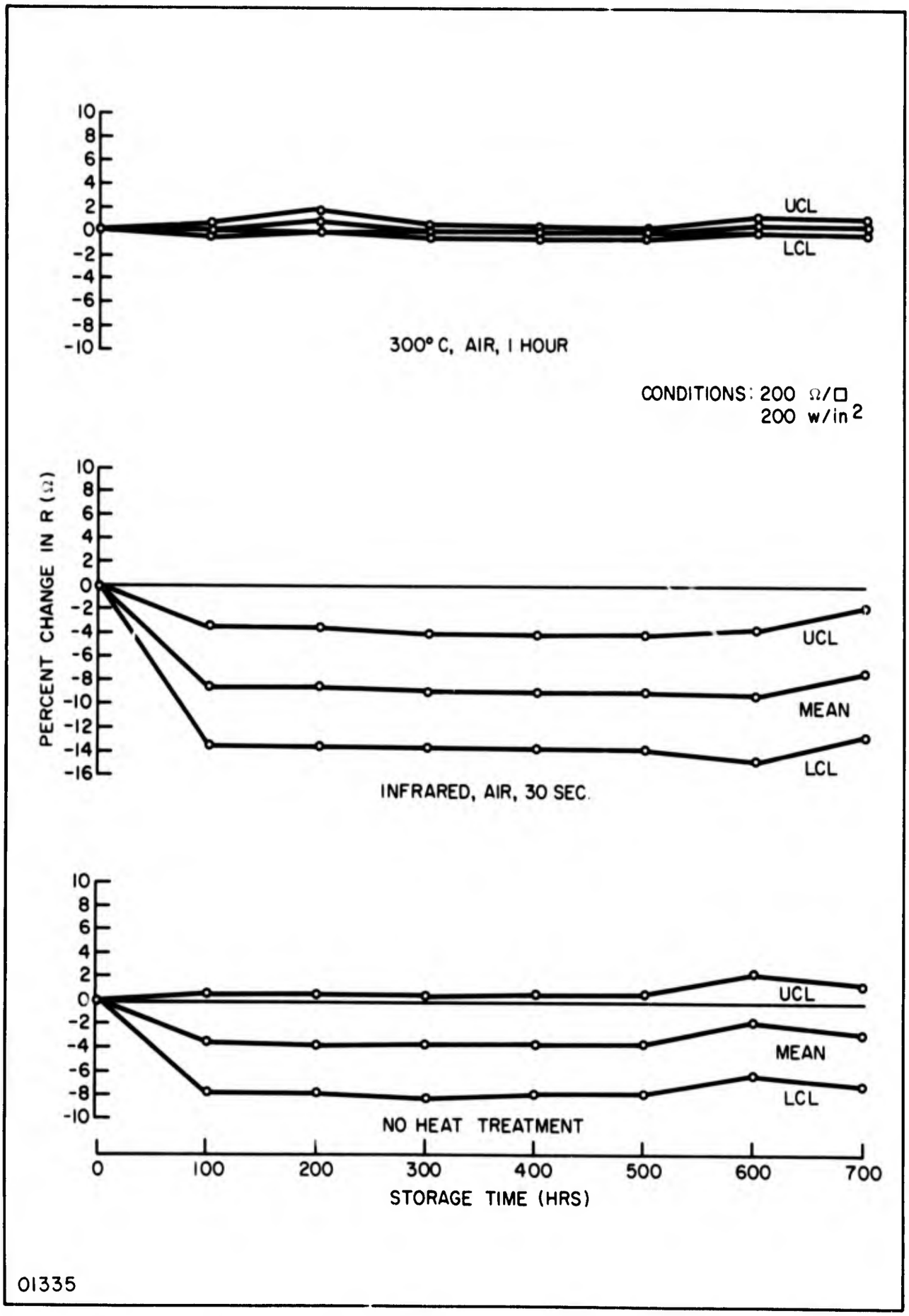

Fig. 23. 200 Ω/□ Ta/Ta_2O_5 Resistor Life Test

SECTION VI

TEST VEHICLE — 455 KHz AMPLIFIER

A severe problem has been encountered in the process for fabricating the amplifier and effort during this reporting period has been expended solving this problem. Since both thin-film resistors and capacitors are being used on this amplifier, the process must have steps to delineate a desired geometry for these devices. The delineation of the dielectric has proven to be a major problem.

Etching has been shown to be a satisfactory method[11] on previous test vehicles to establish holes in the Ta_2O_5 film. However, previous test vehicles had large dimension holes (4 by 4 mils) to be etched and slight undercutting could be tolerated with no yield loss. The 455 KHz amplifier requires etching 1.0 by 0.5 mil holes through the Ta_2O_5 film with < 0.5 mil undercutting and a satisfactory etchant has not been found for this application.

An alternate method for forming holes in the Ta_2O_5 film is making use of a mask to deposit the Ta_2O_5 through. The first attempt was totally unsuccessful when metals such as Al, Mo or Au were deposited on a slice as a reverse deposition mask. The inability to remove the metal mask by etching negated further investigation with this technique.

A technique that has proven successful is forming a KMER mask on the slice and sputtering the Ta_2O_5 through the holes in the KMER.

The process used is:

1) Spin high viscosity KMER on slice
2) Air dry — 30 min. 90°C
3) Expose and develop pattern

4) Bake — 15 min., 150°C, 28 μm pressure

5) Etch to remove KMER residue from bottom plates

6) Bake — 15 min., 150°C, 28 μm pressure

7) Sputter Ta_2O_5 < 100°C substrate temperature

8) Strip KMER mask — J-100*.

There are two critical steps in this process. The first is the time, temperature and pressure relationship for KMER bakeout. The purpose of the vacuum bake is to vaporize the solvents from the KMER to negate outgassing in glow discharge. However, a drying process of higher temperature or lower pressure can fix KMER so that it cannot be removed in the subsequent J-100 step.

The second critical area of the process is to insure that the KMER residue is completely removed from the capacitor bottom plate prior to oxide deposition. There are a variety of ways that KMER residue can be left in the open areas:

1) If the KMER is not developed sufficiently, residue will remain.

2) Solvents can be baked out of the KMER and deposited in the open areas during the vacuum bake cycle.

3) Outgassing during the sputtering cycle can contaminate the sputtered oxide.

These sources of contamination can be minimized by the outlined process.

Several lots of capacitors have been processed using the KMER reverse mask technique with promising results. The quality of the dielectric film is not degraded by the KMER, and the geometry of the dielectric can be delineated by the KMER technique.

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*Resist Strip J-100, IRC Laboratory, 811 South Sherman, Richardson, Texas 75082.

SECTION VII

CONCLUSIONS

A. AMBIENT GAS CONTROL

The residual gas investigation has indicated that the sputtering ambient gas must be controlled to eliminate water vapor. Much of the erratic control of the deposition parameters can be attributed to change in water vapor concentration in the ambient gas. A special sputtering fixture has been designed to minimize this contamination.

B. OXIDATION EXPERIMENTS ON Ta/Ta_2O_5 FILMS

The oxidation experiments on the Ta/Ta_2O_5 resistive films depict a structure that can be altered to aid in control of the temperature coefficient of resistance. In order to fabricate resistive films up to 25,000 Ω/□, a complete understanding of the structure of the film is required to increase the sheet resistance and minimize TCR.

C. PROCESS CONTROL

The present process for fabricating 1000 Ω/□ resistive films and >0.35 pF/mil^2 capacitive films is adequate to yield a 38% process. This yield should increase considerably with increased process control with the recently designed sputtering fixture.

D. THERMAL TRIM

A satisfactory process has been established for using a thermal trim technique to adjust resistor values while stabilizing the films.

E. 455 KHz AMPLIFIER TEST VEHICLE

The Ta_2O_5 etch problem has been solved by using a reverse KMER mask to deposit through. Effort will continue on fabricating the amplifier.

SECTION VIII

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13 ABSTRACT

Basic investigations in the residual gas contamination during sputtering and oxidation mechanism have yielded information that contributes to increased process control. Also, the oxidation experiments depict a structure that can be modified to aid in temperature coefficient of resistance control.

A process control investigation for fabricating 1000 Ω/□ resistors and > 0.35 pF/mil^2 capacitors on the same silicon substrate has been completed. The results show the yield for the 1000 Ω/□ ± 20% tolerance resistive film is 62% and for both the resistive and capacitive films, the yield is 38%.

An etch problem on the 455 KHz amplifier has delayed fabrication of this circuit. The problem has been solved by using a KMER mask to deposit the Ta_2O_5 dielectric through.

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	ROLE	WT	ROLE	WT	ROLE	WT
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