VACUUM CHAMBER EXPERIMENTS
IN
THIN FILM DEPOSITION

by
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ABSTRACT

This work is concerned with the condensation of a metallic vapor onto a substrate of previously treated and suitably masked semiconductor material to form a complete set of electrically conductive pathways of thin metallic film between the various elements of an integrated microcircuit.

The study included (a) formulation of overall specifications for a Thin Film Evaporation Controller for use in conjunction with a vacuum chamber containing a filament heater and evaporant crucible, (b) design and test of the individual subsystems required in the automatic unit, (c) assembly of the component parts into a complete system, (d) performance of system tests to formulate preliminary evaluations, and (e) illustration of the use of the system in a particular integrated-circuit application.

Particular emphasis is placed on the design of the individual subsystems used in the Thin Film Evaporation Controller. The subsystems consist of (a) an Automatic Priming Sequencer, which governs system timing and conditioning of the evaporation material prior to deposition, (b) Deposition-Rate Controller, which commands and controls the deposition rate during an evaporation process and also terminates the process at a preselected film thickness, and (c) a Power Amplifier, which controls the a-c supply voltage applied to the evaporation filament heaters.
CHAPTER I

INTRODUCTION

Thin film metallization by vacuum evaporation is assuming an ever increasing role in integrated circuit technology today. Through the use of thin film techniques, connecting leads, surface electrodes, and passive components are applied to present day integrated circuits. Because of the extremely small physical dimensions, which must be realized, various thin film properties need to be under control if films are to be reproducible (Holland 1956).

Several parameters which have an important role in the reproducibility of film properties are: the pressure and composition of the gas atmosphere in which films are deposited, the temperature of the substrate, the physical properties of the "evaporant", the duration of the deposition process, and the rate of evaporation. The control of the last two parameters is considered in this study.

The rate of evaporation is one of the most important parameters in determining film structure. If a film is deposited at varying rates, it tends to granulate. When the rate of arrival of metal vapor at a surface is uniform and high, the film formed has a smaller grain size, resulting in more predictable surface properties (Levinstein 1949). It has been shown (DeSilva 1960), that under controlled conditions a constant rate of evaporation is obtained by supplying a constant power to the evaporation heaters.
The studies reported herein are based on a method of rate-feedback control, which provides on a self-correcting basis, the heating power necessary to maintain a preselected evaporation rate in a vacuum chamber for a predetermined time interval. Also provided is an automatic priming sequencer which, just prior to the actual evaporation process, accomplishes the steps of (a) warming up the heater filament and crucible at a predetermined rate for a preset time interval, (b) holding the crucible temperature constant at the precalculated arithmetic mean of the melting and evaporation temperatures of the evaporant, for a preset time duration, and (c) thereafter the control of the crucible heating power is provided by the deposition-rate controller.

Following an outline of the total system and its overall specification, the details of the subsystem designs will be presented. The system performance is discussed in conjunction with some typical results obtained from experimental tests.
CHAPTER II
SYSTEM DESCRIPTION

2.1 System Specifications

From an examination of the requirements of various projects in the Solid State Engineering Laboratory, an acceptable set of specifications for the Thin Film Deposition Controller appear to be as follows:

a) Minimum detectable film thickness - 25 Å.
   Maximum detectable film thickness - 20,000 Å.

b) Minimum detectable evaporation rate .2 Å/sec.
   Maximum detectable evaporation rate 2,000 Å/sec.

c) Accuracy for thickness and rate measurements - 5 per cent.

d) Control system response time (from a 0 rate to within 5 per cent of desired rate) - 10 seconds.

e) Thickness termination within 5 per cent of desired thickness.

f) Automatic priming sequence variable from 30 seconds to 3 minutes, ±25 per cent.

g) Compatibility with various vacuum systems.

The block diagram of the complete vacuum deposition system is shown in Figure 2.1. The system is designed to perform the functions of (a) monitoring of film thickness and evaporation rate, (b) warming-up the evaporation source at a prescribed rate, (c) maintaining deposition
Figure 2.1

Block Diagram of Vacuum Deposition System
rate at a preselected value, and (d) terminating the evaporation process at a predetermined film thickness.

2.2 Functional Description

The system consists of four basic subsystems: The Film Thickness Monitor, Automatic Priming Sequencer, Deposition Rate Controller, and Power Amplifier. A detail description of each subsystem is presented in Chapter III.

In order to obtain the thickness and rate signals necessary to control the deposition process, a commercial film thickness monitor is used, the specifications for which are listed in Appendix A. The monitor utilizes a quartz crystal as the thickness transducer in the vacuum chamber. As the evaporation proceeds, the metal vapor condenses on the quartz crystal. The mass of the deposited film mechanically loads the crystal and causes a reduction in its natural oscillation frequency. By means of an I. F. mixer the crystal frequency is mixed with a sinusoidal reference signal. The difference frequency is then mixed in a second I. F. mixer with a signal from a variable frequency reference oscillator. The resulting beat-frequency sinewave, is then clipped, differentiated, rectified, and averaged to produce a slowly varying voltage which is a measure of the thickness of the metallic film being deposited on the crystal.

The evaporation rate is established by taking the time derivative of the thickness signal. Both the thickness and rate signals are available in analog form from the Film Thickness Monitor.
Prior to the depositing of the evaporant, the Automatic Priming Sequencer initiates and provides the timing for the warming-up and degas intervals. By means of a voltage-integrating amplifier, and temperature of the crucible and evaporant is activated at a controlled rate up to a precalculated temperature. A convenient level is the arithmetic mean of the melting and evaporation temperatures of the evaporant. This temperature is then maintained for a preset time interval to eliminate impurities initially residing within and on the surface of the evaporant. The warming-up rate and corresponding time interval, along with the degas time interval, are each adjusted by means of separate potentiometers.

When the degas interval is completed, the source shutter in the vacuum chamber is automatically rotated open and the control of the evaporant heating power is transferred to the deposition-rate controller.

The deposition-rate controller is designed so that the rate signal from the Film Thickness Monitor is compared with a preset reference voltage by means of a differential amplifier. The output signal from the difference amplifier is a rate-error voltage which is supplied as an alternate input to the integrator upon termination of the degas interval.

The Power Amplifier is a silicon-controlled rectifier (SCR) unit which regulates the rms voltage applied to the evaporant heaters in proportion to the signal output from the voltage-integrating amplifier. Thus, the heater power is made to increase or decrease until the proper evaporation rate is obtained.

In order to determine when the process should be terminated, the thickness signal from the Film Thickness Monitor is compared to a preset
reference voltage. When the desired thickness is reached, the output of the comparator energizes a relay. This relay closes the source shutter in the vacuum chamber, and grounds the input to the power amplifier, thereby terminating the evaporation process.
CHAPTER III
SUBSYSTEM DESIGN

A brief description of the complete control system was presented in Chapter II. Aspects of the design and performance of each subsystem will be treated in this chapter.

3.1 Automatic Priming Sequencer

It has been shown (Dushman and Lafferty 1962) that gases are absorbed on the surface, and are present in the interior, of metals either in a state of solution or in the form of chemical compounds, such as oxides and water. If the metal is not degassed prior to evaporation "sputtering" (expulsion of liquid metal droplets from the surface of the metal) generally will occur. A practical solution to such a problem is to provide a controlled rate of warm-up for the material and also a further period of controlled degassing just prior to evaporation. A typical degas and evaporation cycle which has been found effective (Holland 1965) is illustrated in Figure 3.1.

In order to provide the desired timing cycle the Automatic Priming Sequencer shown in Figure 3.2 was developed. The timing system is designed around two wide-range timing circuits which drive the degas and evaporation control relays (K1, K2, and K3).

The degas-and-evaporation cycle is initiated by manually operating the momentary (push-button) switch S1. This delivers timing
Figure 3.1
Timing Diagram for Priming and Evaporation Cycle
Figure 3.2
Circuit Diagram for Automatic Priming Sequencer
initiation pulses to both timers. For simplicity, the 0.5-to-5 minute timers are referred to as timer No. 1 and timer No. 2. When timer No. 1 is actuated relay K1 is energized, thus applying a constant voltage to the input of the voltage-integrating amplifier. This constant input produces the initial ramp on the degas cycle, shown as segment A-B of the plot in Figure 3.2. Also, when relay K1 is energized its second set of contacts K1b, apply an energizing voltage to relay K3. The K3a set of contacts on relay K3 furnishes a path of parallel operating voltage to the coil of K3. This provides energizing power for K3 when K1 is de-energized. The K3b set of contacts on K3 apply the operating voltage to the output stage of timer No. 2. This allows timer No. 2 to trigger the SCR only when its timing cycle is completed. The segment B-C of the plot in Figure 3.1 occurs between the time when relay K1 is de-energized (by the completion of the cycle of timer No. 1) and the time when relay K2 is energized (by the completion of the cycle of timer No. 2). At the end of the timing cycle of timer No. 2, relay K2 is energized, thus applying the rate error signal to the integrator (producing segment D-E of the plot in Figure 3.1). The K2b set of contacts on relay K2 are also used to apply energizing voltage to the shutter soleniod to rotate open the shutter masking the source.

A schematic representation of one of the two identical timing circuits, is illustrated in Figure 3.3. At the start of the cycle a positive initiation pulse is applied to the gate of the SCR. This initiates the timing cycle by applying the supply voltage \( V \) to the relay driver bus, that is, \( V_{bb} = V \) at time \( t=0 \). With this voltage applied, the
Figure 3.3

Diagram for Timer No. 1 and Timer No.2

$R_1 = 240k$ ohms
$R_{21} = R_{22} = 3$ megohms
$R_3 = 10k$ ohms
$R_4 = 1k$ ohms

$R_5 = 1k$ ohms
$R_6 = 10k$ ohms
$R_{B1} = 100$ ohms
$R_{B2} = 150$ ohms

$D_1 = D_2 = 1N251$
$Q_1 = 2N490A$
$SCR_1 = 2N1596$
emitter voltage \( V_e \) of the unijunction transistor (UJT) begins to rise exponentially toward the voltage \( V \) with a time constant \((R_1 + R_2)C_2\). The equation for the emitter voltage during this time interval is

\[
V_e = V_{bb} \left[ 1 - \exp \left( -\frac{t}{(R_1 + R_2)C_2} \right) \right]. \tag{3-1}
\]

When \( V_e \) exceeds \( \gamma V_{bb} \) by an amount equal to the forward voltage drop of the emitter-base diode of the UJT, where \( \gamma \) is the intrinsic standoff ratio for the particular UJT, the UJT "fires" and discharges the capacitor \( C_2 \) through resistance \( R_{B1} \). From Eq. (3-1), the time delay \( t_d \) between the timing initiator pulse and the firing of the UJT is (using \( V_{bb} = V \))

\[
t_d = (R_1 + R_2)C_2 \exp \left( \frac{1}{1-\gamma} \right), \tag{3-2}
\]

where the forward voltage drop of the UJT emitter base diode has been assumed to be negligible. The delay time \( t_d \) for the timing circuit is thus controlled by the setting of potentiometer \( R_2 \).

Considering its equivalent parallel resistance, the resistor \( R_{B1} \) in series with base-one of the UJT is chosen to be sufficiently small such that the discharge time of capacitor \( C \) is much less than its charging time. The discharge voltage pulse appearing across \( R_{B1} \) is coupled through diode \( D_2 \) and capacitor \( C_1 \) to the SCR cathode. This momentarily reverse-biases the SCR and reduces its current below the minimum holding current. The SCR is therefore switched off and remains in the off condition until another degas-and-evaporation cycle is initiated.
3.2 Deposition-Rate Controller

In order to establish and control a desired evaporation rate, the error amplifier and integrator shown in Figure 3.4 were designed. By means of the difference amplifier, the rate signal from the Film Thickness Monitor, is subtracted from the reference voltage derived from potentiometer $R_{R2}$. The resultant error voltage supplied the input to the integrator. It has been shown (Holland 1965), that a controller response-time equal to or less than 10 seconds is adequate for the evaporation of aluminum. This implies that the $R_{10}C$ product should be approximately equal to 10 seconds. The resistor $R_{10}$ is a one-megohm potentiometer and the capacitor $C$ is chosen as 10 uf's.

Since the initial control signal must be a ramp to provide for the warming-up of the evaporant an alternate input to the integrator is necessary. The timing requirements of the warming-up interval need not be held within precise limits. From experimental data, it was found that a maximum slope of 0.20 volts/second for the integrator output voltage during the warming-up interval was sufficient. This implies that the time constant of the second input should be equal to 25 seconds.

Instead of using a 2.5 megohm resistor, a tee network of resistors was designed with resistor values of $R_7 = 45k$ ohms, $R_8 = 250k$ ohms, and $R_9 = 5k$ ohms. The proper timing sequency for application of each input signal to the integrator is dictated by the Automatic Priming Sequencer.

In most deposition processes, a desired metallization thickness is required. When this thickness is achieved the process is to be terminated within a specified tolerance.
Figure 3.4

Rate Error Amplifier and Voltage Integrator
In order to terminate the deposition process the circuits shown in Figure 3.5 was designed. The thickness signal from the Film Thickness Monitor is coupled into a differential amplifier. The differential amplifier is necessary because the signal from the thickness monitor is taken across a resistor, neither side of which is grounded. The output of the difference amplifier is then compared to a reference voltage using a Fairchild \( \mu A \) 710c comparator.

When the reference voltage is greater than the thickness voltage by 2mv, the output of the comparator is +2.5 volts, but when the thickness voltage is within 2mv, or is larger then the reference voltage the output of the comparator is -.5 volt. The output of the comparator is connected to an inverter, which is used to actuate the SCR relay driver. When the difference between the reference voltage and thickness voltage is less than 2mv, the SCR energizes relay K4. One set of contacts of relay K4 is used to switch the input of the power amplifier from the output of the integrator to a grounded connection. This terminates the power input to the heater. A second set of contacts K4 is used to terminate the energizing voltage to the shutter solenoid, thus rotating the shutter to its original position over the evaporation source. Due to the inductive behavior of the relay coil, switching times of the order of 20-to-40 milliseconds are achieved.

The thickness reference voltage is set by a ten-turn helical potentiometer, this provides the capability of terminating the process at any thickness between 45.5Å and 22,720Å. Accuracies in the range of 2 to 4 per cent can be realized.
Figure 3.5

Thickness Comparison and Termination Circuit
3.3 Power Amplifier

In order to provide power control for the resistance-heated source, the principle of a-c phase control is used, whereby the process of rapid on-off switching connects an a-c supply to a load for a controlled fraction of each cycle of the supply voltage. This has been shown (Gutzwiller 1964) to be a very efficient means for controlling power to a load such as a resistance heater. Phase control of an a-c source can be accomplished conveniently by the gating-on of an SCR at a particular phase angle of an a-c supply frequency. The SCR will then continue conducting for the completion of that half cycle in which it was triggered.

The complete circuit of the power amplifier is shown in Figure 3.6. Basically, the control scheme is one in which, by means of transformer T1, the load (evaporant heating filament) is placed in series with a highly variable "impedance". This variable impedance consists, primarily, of the SCR-diode bridge combination (SCR\textsubscript{1}, SCR\textsubscript{2}, D\textsubscript{1}, D\textsubscript{2}) which, by means of the subcircuit powered by the isolation transformer T2, is made responsive to the slowly varying control signal V\textsubscript{c}. Since the SCR-diode bridge is essentially a short-circuit after the appropriate SCR is "fired", the subcircuit is de-energized on initiation of the triggered load-pulse and re-energized at the beginning of the next half-cycle of the a-c supply frequency. The voltage which is developed across the SCR-diode bridge is coupled by means of the isolation transformer (T2), to a full wave diode bridge (composed of diodes D\textsubscript{3} through D\textsubscript{6}). The output of this diode bridge is then used as the supply
Figure 3.6
Circuit Diagram for Power Amplifier
and synchronizing voltage for the UJT trigger circuit and the voltage-controlled current source. Several voltage waveforms associated with the trigger circuit and current source are shown in Figure 3.7.

Referring to Figure 3.7a, at time $t_1$, the a-c supply voltage reaches the value necessary to cause the zener diode ($D_7$), to conduct current at its constant-voltage level ($V_z$). With this voltage applied to the emitter of transistor $Q_1$, a collector current $I_c$ is established through capacitor $C$. In order to insure the linear dependence of $I_c$ upon the control voltage $V_c$, transistor $Q_1$ is biased such that it remains in its active region throughout its operation.

Because the control voltage $V_c$ varies only very slowly during a half cycle of the a-c supply voltage the collector current can be considered constant during the same half-cycle. Thus the time rate of change $dV_e/dt$, of the UJT emitter voltage is essentially constant and equal to $I_c/C$. When the UJT emitter voltage reaches $V_{bb}$ (the emitter firing potential), the UJT fires (at time $t_2$), discharging $C$ through the pulse transformer $T_3$. This results in a trigger pulse applied to the gates of $SCR_1$ and $SCR_2$. The SCR with a positive potential on the anode is then gated on, thereby removing the supply voltage from the subcircuit. This permits the desired current to flow in the load for the remaining portion of that half cycle. When the supply voltage decreases to a value for which the current in the SCR is less than its specified holding current the "on" SCR ceases to conduct. When the SCR's are not conducting, the supply voltage is again transferred to the subcircuit. This initiates the new cycle, carrying out the desired
Figure 3.7
Voltage and Current Waveforms for Power Amplifier
synchronization of the trigger circuit with the a-c supply. The same events occur in the next half-cycle of the supply voltage, except the second SCR is now gated on to control the current delivered to the load.

If the resistances in the network \((R_1, R_2, R_3)\) of the SCR-controlling subcircuit in Figure 3.6 were all zero, the average power applied to the load would be responsive not only to the control voltage \(V_c\), but also, to any variations in the a-c supply voltage. The indicated non-zero values used for \(R_1\), \(R_2\), and \(R_3\) were selected in accordance with a principle previously established by Cleary (1964). The regulative capability of the circuit is due essentially to the presence of \(R_2\) of Figure 3.6, which elevates the UJT interbase voltage \(V_{bb}\) above the zener voltage. The resistor \(R_2\) in parallel with the series combination of \(R_3\) and the UJT interbase resistance \(R_{bb}\) forms a voltage divider with \(R_1\). The equation for \(V_{bb}\) is thus

\[
V_{bb} = \left[ \frac{V_S}{R_1} + \frac{V_Z}{R_2} \right] \cdot \left[ \frac{1}{\frac{1}{R_1} + \frac{1}{R_2} + \frac{1}{R_{BB}}} \right].
\]

When a prolonged surge in the amplitude of the a-c supply voltage is experienced, the output of the full-wave bridge (diodes \(D_3, D_4, D_5\) and \(D_6\)) also increases, thereby increasing \(V_{bb}\). This increase in \(V_{bb}\) results in an increase in the emitter peak-firing potential, \(V_p = \sqrt{3}V_{bb}\) of the UJT. With an increase in \(V_p\), the triggering of the UJT is delayed, and results in a reduction of the conduction angle of the SCR. The converse is also true; when the line potential is decreased the
conduction angle of the SCR increases. The potentiometer $R_2$ was adjusted to a point where 10 per cent variations in the nominal 115-volt a-c supply voltage produced less than one per cent variations in the measured rms load voltage.

The desired linear relationship between the control voltage $V_C$ and the SCR conduction angle is assured if the collector current $I_C$ of transistor $Q_1$ is a linear function of the control voltage $V_C$. From the circuit parameters shown in Figure 3.6, it is seen that the rate of increase of the UJT emitter voltage $V_e$ (prior to UJT firing) is governed by the equation

$$\frac{dV_e}{dt} = \frac{I_C}{C} = \frac{h_{FE} V_B - V_{BE}}{R_5 + \frac{R_4}{R_5}}$$

(3-5)

in which $h_{FE}$ is the d-c current gain and $V_{BE}$ is the base-to-emitter voltage of transistor $Q_1$. The values of $R_4 = 51.3k$ ohms, $R_5 = 1500k$ ohms, $C = 0.1\mu f$, and $h_{FE} = 200$ give values of $dV_e/dt$ of 1.0 volt/millisecond for $V_C = 0$ and 10 volt/millisecond for $V_C = -10$ volts. These slope values for the rising UJT emitter-firing voltage correspond, in the power amplifier circuit illustrated in Figure 3.6, to SCR conduction angles of $\phi = 0$ and $\phi = 160$ degrees, respectively. This range of conduction angles results in range of rms load-voltage control from essentially zero to 96 per cent of the a-c supply voltage.
CHAPTER IV
SYSTEM OPERATION

In the discussion on subsystem design in Chapter III, there are five operating conditions which are determined by five potentiometer settings. These operating conditions are (a) the rate at which the temperature of the heater filament and evaporant crucible is elevated toward the degas temperature level, (b) the length of the time interval over which the elevation toward the degas temperature level occurs, (c) the length of the time interval over which the constant degas temperature level is maintained, (d) the rate at which the evaporant is deposited on the substrate, (e) and the film thickness at which the evaporation process is to be terminated. The details of the relationships between the potentiometer settings and the five corresponding operating conditions are given in the discussion which follows.

The first three of the above mentioned five operating conditions have previously been designated as the priming interval. In order to precalculate the three potentiometer settings for the priming interval, four properties of the heater filament were determined experimentally. These properties are as follows: the power per unit area required by the heater filament to sustain a desired temperature, the resistivity of the filament at that temperature, the effective filament length, and the effective external heating area. The temperature dependence of resistivity and the power per unit area measured for the particular
tungsten filament used in the system concerned in this report are shown in Figure 4.1. Each set of data points was obtained both with and without the evaporant present on the tungsten filament. The effect of the presence of the evaporant on the filament was found to be essentially negligible.

By use of the experimental results in Figure 4.1, the rms voltage required by the heater filament to obtain a desired temperature is found to be

\[ V_{\text{rms}} = \sqrt{\frac{4W\rho L^2}{d}} \text{ volts,} \]  

(4-1)

where \( \rho \) is the resistivity, \( d \) is the filament diameter, \( W \) is the power per unit area, and \( L \) is the effective filament length. A plot of \( V_{\text{rms}} \) versus temperature is shown in Figure 4.2a. Conversion of an experimentally confirmed, theoretical curve for rms load voltage versus SCR conduction angle (Bath and English 1967) to the present application produced the curve shown in Figure 4.2b. The data plotted in Figure 4.2, thus leads to the curve shown in Figure 4.3, which relates the power-amplifier voltage \( V_c \) to the filament temperature for an a-c supply voltage of 115 volts and for the particular heater filament having an equivalent cylindrical radiating area of 14.3 cm\(^2\), a cross sectional area of .017 cm\(^2\), and a length of 20 cm. By use of the results in Figure 4.3, the control voltage \( V_c \) necessary to sustain a desired degas temperature can be determined.

The particular warming-up rate, that is, the rate at which the control voltage \( V_c \) is elevated, must naturally depend on the material
Figure 4.1

The Temperature Dependence of (a) Power per Unit Area, and (b) Resistivity for a Tungsten Filament.
Figure 4.2

The (a) Ratio of RMS Load Voltage to RMS Line Voltage as Determined by Power Amplifier Control Voltage, and (b) Filament Temperature Dependence on RMS Load Voltage
Figure 4.3

Relationship Between Filament Temperature and Power Amplifier Control Voltage $V_C$
being evaporated. From the results of several laboratory trials it was found that a warming-up interval of approximately 30-seconds to one-minute and a degas interval of 30-seconds were adequate for the conditioning of both aluminum and platinum. If the two intervals were adjusted to be either longer or shorter than those specified above, the evaporant would wet the filament unevenly and drop off prior to evaporation.

Referring to Figure 3.2, it is seen that the potentiometer \( R_{R1} \) controls the rate of increase \( \frac{dV_c}{dt} \) of the control voltage \( V_c \) during the warming-up interval, as governed by the equation

\[
\frac{dV_c}{dt} = x \left[ \frac{V_{R9}}{C(R_7R_8 + R_7R_9 + R_8R_9)} \right], \tag{4-2}
\]

where \( 0 \leq x \leq 1 \) corresponds to the setting of potentiometer \( R_{R1} \). For the values of \( V_R = 5 \) volts, \( C = 10 \mu F \), \( R_7 = 45 \) k ohms, \( R_8 = 250 \) k ohms, and \( R_9 = 5 \) k ohms, this equation reduces to the formula \( \frac{dV_c}{dt} = (0.2)x \) volts/second, which is plotted in Figure 4.4. Thus, the required setting of the warming-up potentiometer \( R_{R1} \) is obtained from Figure 4.4 simply by precalculating the control voltage slope \( \frac{dV_c}{dt} = V_{cd}/t_w \) from the warming-up time interval \( t_w \) and the desired degas control voltage level \( V_{cd} \). The warming-up time interval \( t_w \) is controlled by adjusting potentiometer \( R_{21} \) in timer no. 1, which is illustrated in Figure 3.3. Potentiometer \( R_{21} \) is a single-turn linear potentiometer (zero to three-megohms) which is set according to time calibrations on the front panel of the Thin Film Evaporation Controller.
Figure 4.4

Relationship Between Power Amplifier Control Voltage $V_c$ and Setting of Warming-Up Potentiometer $R_{R1}$
With the completion of the warming-up time interval, the Power Amplifier control voltage is held constant during the degas time interval allowing the impurities initially residing within and on the surface of the evaporant to be eliminated. The degas time interval \( t_d \) is controlled by adjusting potentiometer \( R_{22} \) in timer No. 2, which is illustrated in Figure 3.3. Since both timing circuits are initiated at the same instant, timer No. 2 must be set for a time interval equal to the sum of the warming-up time and the degas time intervals \( (t_w + t_d) \). Potentiometer \( R_{22} \) is also set according to a time calibration on the front panel of the Thin Film Evaporation Controller. This completes the three potentiometer settings which govern the priming interval. There remains now two potentiometers to be set. These potentiometer settings will govern the rate at which the evaporant is to be deposited and the final thickness at which the evaporation process is terminated.

The rate reference potentiometer \( R_{R2} \), shown in Figure 3.4, is set in conjunction with the evaporation rate scale of the Film Thickness Monitor. The Film Thickness Monitor has three ranges to which the evaporation rate scale can be set, but for each range the corresponding analog output voltage always varies between zero and 5 volts. Therefore, the rate reference voltage was made variable between zero and 5 volts by using a ten-turn potentiometer connected to a 5 volt reference level. Thus, when the desired evaporation rate corresponds to \( y \) per cent of the monitor-rate scale, the rate potentiometer \( R_{R2} \) is set at \( y \) per cent of its total resistance. This leaves only the thickness potentiometer to be set.
The thickness potentiometer $R_{r3}$, shown in Figure 3.5, is set by following the same procedure as was used for the evaporation rate potentiometer. The thickness scale of the Film Thickness Monitor has four ranges, but again the corresponding analog output voltage varies between zero and 5 volts for all scales. Therefore, another ten-turn potentiometer $R_{R3}$ is connected to the same 5 volt reference level as the rate potentiometer. Thus, if the terminating thickness corresponds to $z$ per cent of the thickness scale of the monitor, then the thickness potentiometer $R_{R3}$ is set at $z$ per cent of its total resistance. The Thin Film Evaporation Controller is now ready to initiate and control to completion an evaporation process.

A step-by-step operating procedure, covering both the Film Thickness Monitor and the Thin Film Evaporation Controller, is given in Appendix B.
CHAPTER V

EXPERIMENTAL RESULTS AND DISCUSSION

The subsystems described in the foregoing discussion were interconnected as a total "thin-film-evaporation system", and various performance parameters of the system were investigated.

In order to verify that the system was operating within its specifications, several evaporation trials were initiated and controlled to completion. During each process, data was plotted of (a) the normalized rate error, that is, the ratio of rate-error to desired rate, (b) the rms voltage supplied to the heater filaments, (c) the final film thickness as compared with the desired thickness, and (d) the final film thickness resulting from a given monitor frequency shift.

Referring to specification (d), in Chapter II, the control system is to have a response time of 10 seconds for a step change in rate, that is, from a zero-rate to within 5 per cent of desired rate in 10 seconds. A plot of the normalized rate error and a profile of the filament supply voltage are shown in Figure 5.1. Figure 5.1a verifies that the evaporation rate is within 5 per cent of the desired rate at the completion of the first 10 seconds of evaporation. Figure 5.1b illustrates, that if, a constant power is delivered to the heater filaments, this will result in an approximately constant evaporation rate. That is, the evaporation rate has reached its desired level when the filament voltage has reached its stable-final value.
Figure 5.1

The (a) Normalized Rate Error, and (b) Filament Voltage for an Evaporation Process Controlled at an Evaporation Rate of 50 Å/Second
The second specification to be verified was the 5 per cent tolerance placed on the final film thickness at which the process is terminated. Figure 5.2 shows the specified thickness tolerance envelope within which the process should be terminated, corresponding to a desired film thickness. All results were within the specified 5 per cent limits, except at film thicknesses above 1500 Å. The error resulting above 1500 Å was attributed to undesired heating of the monitor crystal in the vacuum chamber, which results in a variation of the natural frequency of the crystal.

In order to verify that this error was due to the heating of the crystal, several evaporation trials were conducted using only the Film Thickness Monitor and a manual controller. The first set of trials, the results of which are shown in Figure 5.3, were conducted with no heat shield on the crystal, while in the remaining set of trials the crystal was completely shielded except for a small opening to the crystal face. The resulting error at large film thicknesses was found to be reduced in the second set of trials. One solution to the crystal heating problem would be to build a water-cooled jacket around the monitor crystal. This would maintain the crystal at an approximately constant temperature throughout the evaporation process.

As a final test of the complete vacuum deposition system, a thin film of aluminum was deposited on a previously treated and suitably masked silicon wafer (Wise 1967) to provide the bonding surfaces for the Unijunction transistor shown in Figure 5.4. The Thin Film Evaporation Controller was set to maintain a deposition rate of 25 Å per second and
Figure 5.2

Terminal Film Thicknesses for Several Evaporation Trials
Figure 5.3

Experimental Correlation Between Film Thickness and Frequency Shift as derived from the Film Thickness Monitor
Figure 5.4
Photograph of Unijunction Transistor Metallization Patterns
to terminate the evaporation process at a desired thickness of 1000 Å. The results of the metallization were as follows: a deposition rate of 24.5 Å per second was maintained during the process, and the terminal thickness achieved was 1030 Å. These results were well within previously stated specification.

On the basis of the studies conducted thus far, it appears that the system described performs the overall control necessary for evaporation and deposition of selected thin metallic films. The experimental tests performed with the total system in operation confirmed that the accuracy of the control achieved by the interconnected subsystems was within the tolerances sought for in the design. Future improvements might include (a) simplified device for initiating and controlling the priming interval, (b) an automatic device for stabilizing the temperature of the film-thickness-monitoring crystal, and (c) possible incorporation of integral-plus-proportional control of the Power Amplifier.
APPENDIX A

SPECIFICATIONS FOR COMMERCIAL ITEMS

Specifications for the Edwards Film Thickness Monitor and the Burr-Brown, 1507 Operational Amplifiers are presented in this section.

**Edwards Film Thickness Monitor**

**Thickness Measurements**

- Minimum detectable frequency shift: 10 Hz
- Maximum frequency shift before resetting zero: 50 kHz
- Crystal frequency shift: 150 kHz max.
- Frequency shift repeatability on same range: 1% of fsd
- Frequency shift drift over any 15 minute period: less than 100 Hz

**Rate of Thickness Change Measurement**

- Minimum detectable rate of thickness change: 0.05 divisions/sec.
- Maximum rate of thickness change: 10 divisions/sec.
- Rate of change accuracy on any one range: 1% of fsd

**Electricity Supply Required**

- Power Requirements: 90-130V or 200-240V
- 50-60 Hz
- 10 Watts max.
Burr-Brown, 1507 Operational Amplifier

Open Loop Specifications

- Rated Output - Voltage: 10 volts
- Rated Output - Current: 2 ma
- Input Current Offset: 10 na

Closed Loop Specifications

- Input Impedance: 50MHz

Power Supply Requirements

- Rated Supply Voltage: 15 volts DC
- Supply Drain: 10 ma
APPENDIX B
OPERATING PROCEDURE

The purpose of this Appendix is to present a step-by-step operating procedure for both the Film Thickness Monitor and the Thin Film Evaporation Controller, which are shown in Figure B.1.

Steps
1. Turn on the power to the Film Thickness Monitor and Thin Film Evaporation Controller. Allow a ten minute warm-up period before making potentiometer settings.

Film Thickness Monitor Adjustments
2. Calculate the frequency shift \( \Delta F \) which corresponds to the desired film thickness \( T \) using the following equation:

\[
\Delta F = 0.815 \frac{T}{\rho},
\]

where \( \rho \) is the density of evaporant.

3. Select the frequency shift range which results from the above calculation (choose \( \Delta F \) to be approximately the half scale reading).

4. Rotate the frequency shift "set zero" control fully clockwise until considerable friction commences. Now turn the set zero control slowly counter-clockwise until the meter indicates
Figure B.1

Photograph of Thin Film Evaporation Controller
exactly 10 per cent of full scale deflection. If the 10 per cent point is missed repeat step 4 again.

5. Select the rate scale on the Film Thickness Monitor which corresponds to approximately twice the desired rate of evaporation.

6. Push in and hold in the "set zero" push-button under the rate meter and adjust the "set zero" control until the rate meter read zero.

7. Calculate the arithmetic mean of the melting and evaporation temperatures for the material to be deposited.

8. Refer to Figure 4.3 to obtain control voltage level $V_{cd}$ necessary to obtain the mean temperature.

9. Adjust timer No. 1 for the desired warming-up time interval $t_w$ (30 second-to-one minute is adequate for most commonly used evaporants).

10. Calculate $V_c/t_w$, and refer to Figure 4.4 to obtain the setting for the warming-up rate potentiometer.

11. Adjust timer No. 2 for desired degas time (30 seconds is adequate for most commonly used evaporants). Note: If evaporant is held in a crucible, both timers should be set for maximum time interval.

12. Set the thickness potentiometer of the Thin Film Evaporation Controller as follows: (a) if the desired thickness corresponds to $y$ per cent of the frequency shift scale on the monitor, (b) set the thickness potentiometer at $y$ per cent of its total resistance.
13. Set the rate potentiometer of the Thin Film Evaporation Controller as follows: (a) if the desired rate corresponds to $z$ per cent of the rate scale on the monitor, (b) set the rate potentiometer at $z$ per cent of its total resistance.

**Process Initiation**

14. Complete the following sequence on the Thin Film Evaporation Controller to start the evaporation process.
   a. Press in RESET push-button
   b. Press in START push-button

If the evaporation process must be terminated prior to completion press in the STOP and then the RESET push-buttons.
REFERENCES


