AN AUTOMATED THIN FILM SYSTEM FOR CONTROLLING EVAPORATION AND RESISTIVITY MEASUREMENTS

William H. Clark

UMI Number: EP01194

UMI Microform EP01194

ProQuest Information and Learning Company
300 North Zeeb Road
P.O. Box 1346
Ann Arbor, MI 48106-1346

AN AUTOMATED THIN FILM SYSTEM FOR

CONTROLLING EVAPORATION AND

RESISTIVITY MEASUREMENTS

APPROVED:

John Brient

Chairman

Dean of the Graduate School

ACKNOWLEDGEMENTS

My thanks must be extended to Dr. John Brient for hig indulgence and professional expertise in all manners pertaining to this endeavor. I am grateful to Mr. George Moore for extending his experience and knowledge of the thin film apparatus; and to Mr. Carlos Manquero for his aid in comprehending IBM programming techniques. Dr. Donald Bowen is to be thanked for his frequently demonstrated co-operativeness with the Hewlett-Packard programming systems and aids. I greatly appreciate the work of Mr. Karl Marquardt, whose expert knowledge of materials and fabrication techniques were used to construct several portions of the system. Comments made by Mr. Rick Longbrake were useful in eliminating software short-comings. My deepest gratitude must be expressed to Mr. Bruce Andrews and Mr. Jack Graves for the tremendous assistance with the system development and construction, particularly for the film-shutter control by Mr. Andrews and the substrate masks and pc board by Mr. Graves. The assistance given by Mr. Ross Wilson, in the photoetching processes, is greatly appreciated.

TABLE OF CONTENTS

CHAPTER PAGE

LIST OF FIGURES

CHAPTER I.

INTRODUCTION

Although the physics of thin metal films has been of interest for many years, the lack of technology has hindered the collection and analysis of reproducible data. With the advances in high vacuum apparatus and, the advent of high speed electronic computers, the primary obstacles have been removed. This may be evinced by the increasingly sophisticated models being used to describe thin films. Many of the theories are based on the work of Fuchs (1938) and the subsequent modification by Sondheimer (1952). The result is the Fuchs-Sondheimer model of the conductivity of thin metal films or, simply the Fuchs-Sondheimer model. A Method for Fitting the Fuchs-Sondheimer Theory to Resistivity Measurements for all Film Thickness, by G. N. Gould and L. A. Moraga, sketches a least squares technique of determining the bulk resistivity; mean free path length; and, a specularity constant. Appendix F contains the resultant data analysis program and instructions for its' use.

BASIC SYSTEM REQUIREMENTS

Examination of thin film size-effects, by such a model as the Fuchs-Sondheimer theory, necessitates a minimal deviation of all film characteristics other than the thicknesses.

Concurrently, an analysis involving least squares requires nine or more films of different thicknesses. Since films formed in a single evaporation would be expected to exhibit similar characteristics; A system requirement is the simultaneous evaporation of at least nine films of discrete thicknesses. Inadvertant exposure of the films to atmospheric contaminants, particularly H_2O and O_2, can nullify the care taken in satisfying the above requirement. Unless under controlled conditions, comtaminants can change the physical, chemical, and thus, the electrical characteristics in an entirely unpredictable manner. Thus, a second requirement is for the film data (specifically, the electrical measurements) to be taken in situ. One of the most vital, and yet versatile components of a thin film system is that which controls the temperature of the substrate onto which the film is deposited. Surface properties of a film would be expected to effect the specularity constant, while the average grain size may effect the electron conduction. The rate of evaporation in conjunction with substrate temperature, controls a degree of variation in these characteristics. For instance, a rapid evaporation onto a cold substrate results in small grains and an atomically rough surface. Making measurements at various temperatures permits evaluation of the temperature coefficient of resistivity of a film.

Thermal lattice vibrations normally dominate size effects, necessitating measurements at low temperatures. Similarly, variations in temperature vary the reduced thickness parameter (ratio of the film thickness to the electron mean

free path length). Possessing the capacity of temperature control also yields the capability of examining possible annealing processes occurring in the metal films. An evaporation and measurement system[1] was constructed by Mr. George Moore which satisfied many requirements of thin film construction and data extraction.

AUTOMATION

In accordance with the above requirements, an automated thin film system has been constructed that also performs several other tasks; has the capability of following short time period annealing processes; and, performing rapid evaporations which are impossible manually. Basically, the system is divided into two modes of operation: evaporation; and, measurement.

In the evaporation mode, the operator adjusts the system for the overall rate of evaporation; then instructs the controller, (an HP9830A programmable calculator), as to the conditions to be fulfilled. The operator instructs the calculator as to: (1) substrate temperature; (2) whether the first film is to be "just continuous" or not; (3) maximum film thickness; and, if the calculators' choice of thicknesses is not desired, the operator may change any or all of film thicknesses. The operator initiates the deposition

---

[1]For a detailed description of the system see the thesis A System for Thin Film Deposition and Resistivity measurements, by George Moore. It was this system that was modified and automated and his thesis contains a plethora of information not included in this undertaking.

power and the calculator assumes control. Twelve films of the eleven chosen thicknesses are generated by the system. As the films are generated, information on the rates of formation; final film thicknesses; and, resistivity data (taken on the twelfth film at the corresponding thickness) are stored in the calculator. After the films have been generated, the stored data is printed out on a page printer.

Switching to the measurement mode: (1) ac line voltage, frequency and their standard deviations are determined; (2) the operator answers several questions, posed by the calculator, to determine the range of control the calculator is to have. Although keyboard operation and control can be assumed at almost any time, it is feasible to only tell the calculator the first, last, and incremental steps, of desired substrate temperatures at which resistivity measurements are to be made.

A liquid nitrogen reservoir is automatically kept full; the calculator checks for equilibrium at the desired substrate temperature; and, when reached, performs four probes, forward and reverse resistivity measurements; choosing the optimum of two possible film-current conditions. At a given temperature, the calculator consumes approximately one and a half minutes making the data measurements. (Necessitating 132 switching steps.) As a further check on equilibrium, the substrate temperatures determined by three thermocouples, are recorded at the beginning and the end of each resistivity measurement cycle. The data is output on a page-printer and, if desired, stored on a magnetic tape

cassette. After the third temperature point, a least squares TCR analysis on each film is also printed out. At any time, resistivity measurements can be made under non-equilibrium conditions, without affecting the equilibrium measurements or the least squares TCR data, in order to investigate such phenomena as annealing. A system clock logs the time of each major step, or measurement, and the system shuts down after the last measurement at the last temperature.

Chapter II examines the various components of the system; how they operate, and the generalities of how they interface to form the automation sequence.

The third chapter is intended as an operators manual on how to use the system to the fullest of its capabilities. Frequent references will be made to the appendixes, containing system schematics; symbol explanations; tables of patch cord pin-designations and cross references; software listings and program explanations; and, the listing and operating instructions for the Fuchs-Sondheimer data analysis program.

CHAPTER II.

THE AUTOMATED SYSTEM

Although the apparatus construced by Mr. Moore satisfied the basic requirements of a thin-film system, maintaining a manual control over the apparatus and the data extraction imposed several difficulties and limitations. When performing film depositions one had to monitor a frequency counter, judge when a film had reached the proper thickness, manually shutter the film from the evaporative stream, record the time and record the frequency; thus operator response time precludes both film-depositions at high evaporative rates and accurate film-formation data. Perhaps the most debilitating facet of the manual system was the length of time involved in data extraction; frequently, more than an hour would be devoted to making the electrical measurements at a single temperature. The time involved in making these measurements thus tended to limit the number of temperature points at which to take data, and in conjunction, imposed difficulties of maintaining a temperature equilibrium. Moreover, it prevented analysis of any annealing or quenching phenomena which might occur in the metal films. The purpose of automating the system was to minimize these limitations; and in addition, provide a means of eliminating errors which usually occur in the manual tabulation and manipulation of raw data.

A Hewlitt-Packard, (HP), 9830A programmable calculator operates as the automated-system controller. The calculator contains an eight kilo-word memory and is supplemented with specialized ROMs - read only memories - allowing interfacing with peripheral devices (via an HP bus) and, enhanced software capability. The calculator is directly interfaced with five peripherals: an HP 3490A Digital Multimeter (DMM); an HP 5345A Frequency Counter; an HP 59303A Digital to Analog Converter; an HP 59308A Timing Generator; and, an HP 59306A Relay Actuator. The relay actuator and a relay bank forms the indirect interface between the calculator and: (1) a film-shuttering control; (2) a scanner, which connects the appropriate signal (temperature, film-voltage, and film-current information) to the DMM: (3) an HP 6213A Power Supply which, in union with the scanner, supplies current to the desired film; and (4) a latching circuit to parallel the DMM inputs with an HP 419A Null Voltmeter. Figure 2.1 depicts a modular diagram of the automated system. Appropriate modules are further subdivided and examined in detail in subsequent portions of this thesis. Listings and explanations, of the calculator programs, are contained in Appendix E.

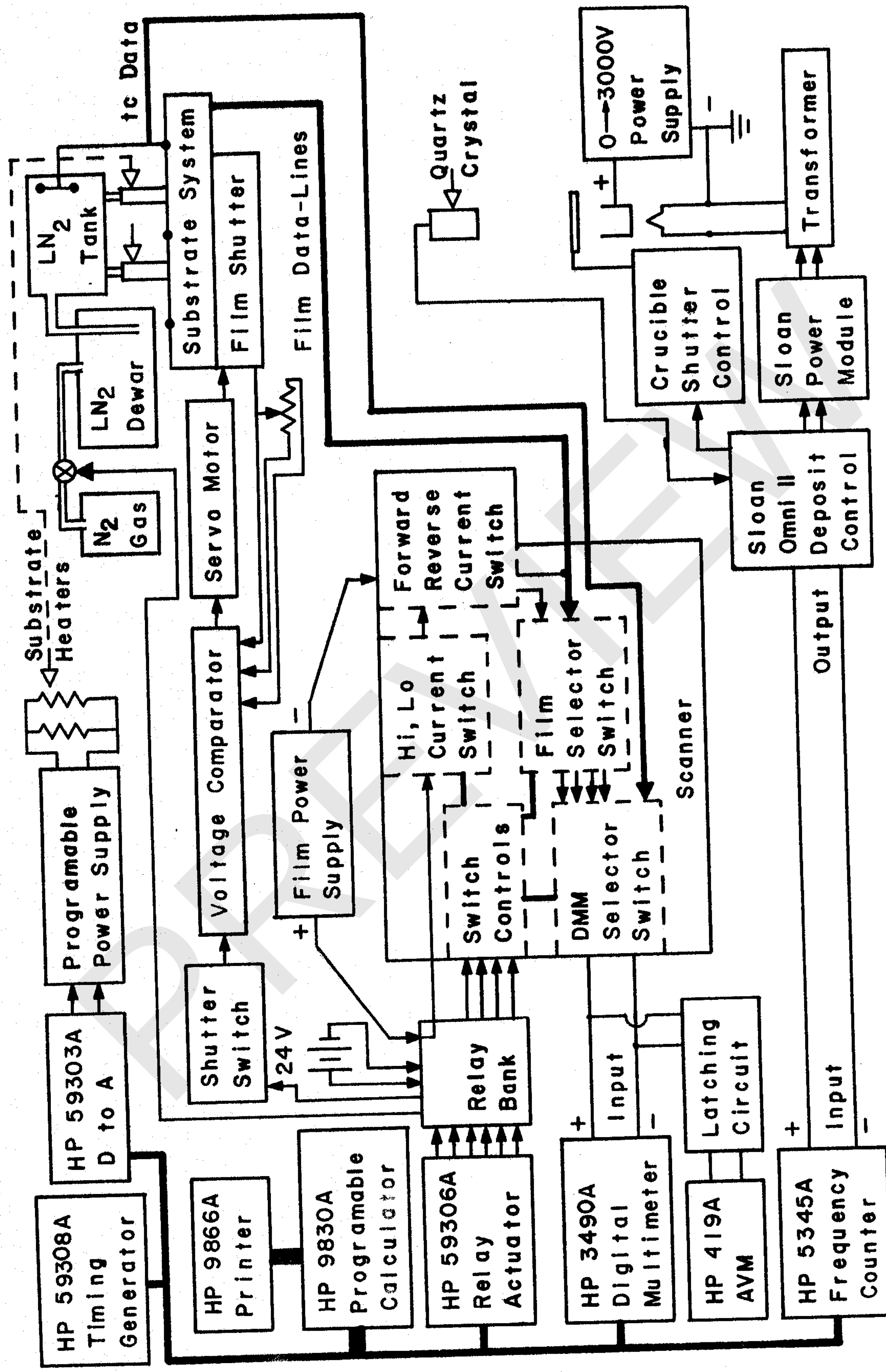


Figure 2-1

The Automated System

SINGLE EVAPORATION

An electron bombardment system heats a crucible containing the metal to be evaporated. The potential difference between the electron source and crucible is variable to 3000 volts and the electron source is a filament whose current is determined by a Sloan Omni II Depositor Control. This unit utilizes a quartz crystal, placed within the evaporant stream and on the plane of the film substrates, as a signal input. The resonant frequency of the quartz (which varies with the mass of the evaporant condensed upon the crystal) is mixed with an internal source to yield a resultant beat frequency. The beat frequency can be initialized to zero and is responsive up to 100 kilo-hertz. This frequency is approximately related to the thickness of the metal deposited upon the crystal, by the equation:

$$(1) \qquad \Delta f = \rho \Delta T/2,$$

where: f is the frequency change; T is the thickness change (deposited upon the crystal); and, rho is the density of the material being evaporated. The Sloan device utilizes the frequency change in attempting to maintain an operator determined rate-of-evaporation, measured in hertz per second. A meter movement indicates the frequency, although an output terminal is available for the connection of an external measurement device. The meter also contains a "high limit"

setting; when the frequency (which corresponds to a thickness) reaches this setting the electron source is turned off and an internal relay is actuated. Connected to this relay is a crucible-shutter control. The control has three settings: open, closed, and automatic. When the switch is put into the open position, a shutter, situated immediately above the crucible, swings away; in the closed position the shutter closes off the evaporant stream. In the automatic position, the shutter is controlled by the Sloan device. When the Sloan applies deposit power, the shutter opens; when the frequency reaches the high-limit setting, the shutter closes.

In Figure 2.2, the basic components of the evaporative process, and the vacuum chamber, are displayed. A section of the crucible power system (The Sloan Power Control Module), is controlled by the Sloan Omni II, and supplies the power to the electron source (the filament) needed to maintain the desired rate-of-evaporation. The substrate system is examined in the Measurement section of this chapter; while the liquid nitrogen reservoir, thermal conductors, and heating resistors are contained under the Temperature Control division.

Film Shutter

Utilizing a least squares method of analysis requires a minimum of nine distinct data points. Investigation of size effects, require that the films differ, essentially, only in thickness. Thus one desires a system to form at least nine films of distinct thicknesses within a single evaporation.

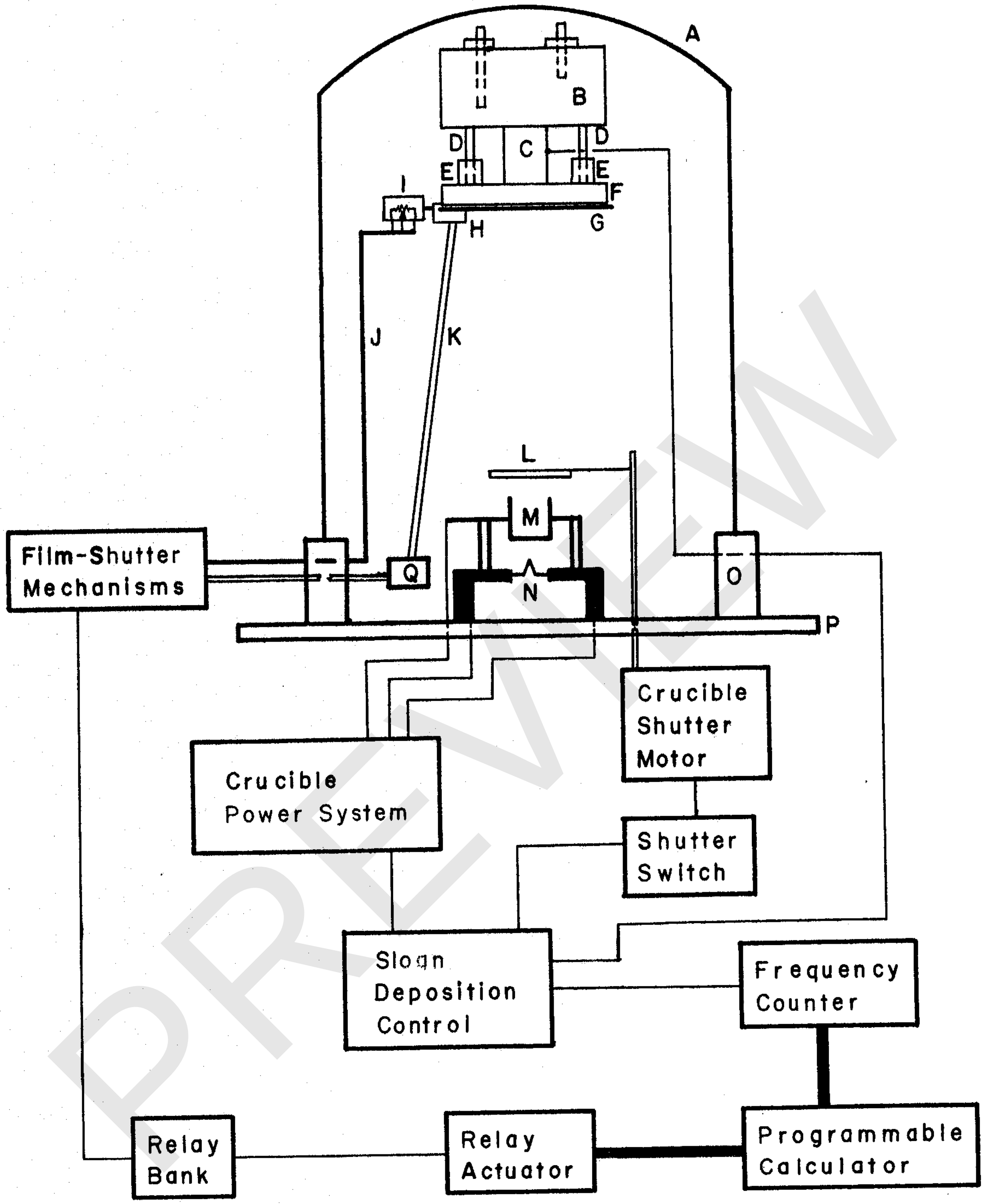


Figure 2.2
The Evaporation System
(Key on Following Page)

A. Glass Bell Jar

B. Liquid Nitrogen Reservoir

C. Quartz Crystal Monitor

D. Metal Posts (Thermal Connectors)

E. Power Resistors (Heaters)

F. Substrate System (Substrate Holder, PC Board, Mask, Mask Holder)

G. Film Shutter

H. Worm Gear; Connecting Driveshaft to Film Shutter and the Three Turn Pot

I. Three Turn, 1000 ohm Potentiometer

J. Cable Harness, for Electrical Feedback from Three Turn Potentiometer

K. Mechanical Linkage: Drive Shaft and U Joints

L. Crucible Shutter

M. T_1B_2-BN Crucible

N. Tungten Filament

O. Vacuum Chamber Collar

P. Vacuum Chamber Base Plate

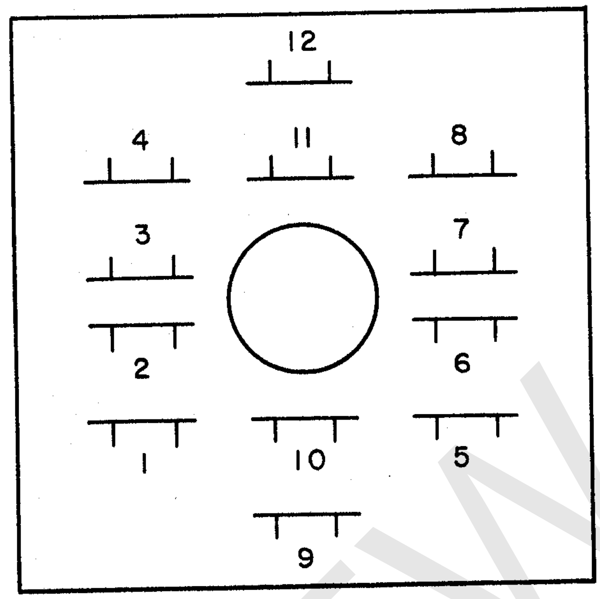
Q. Right Angle Spline

Given the film arrangements in Figure 2.3a, it is possible to evaporate 12 films of 11 different thicknesses without shuttering the quartz crystal from the evaporant stream. Such a film shutter is displayed in Figure 2.3b. The shutter is situated immediately below - and attached to - the substrate system; and, can completely block the metal evaporant from the film substrate. The numerals in Figure 2.3a corresponds to film identification, and the order in which the films are shuttered. Film number one is shuttered first and is thus the thinnest film; films number eleven and twelve are never shuttered; and, are thus the thickest films.

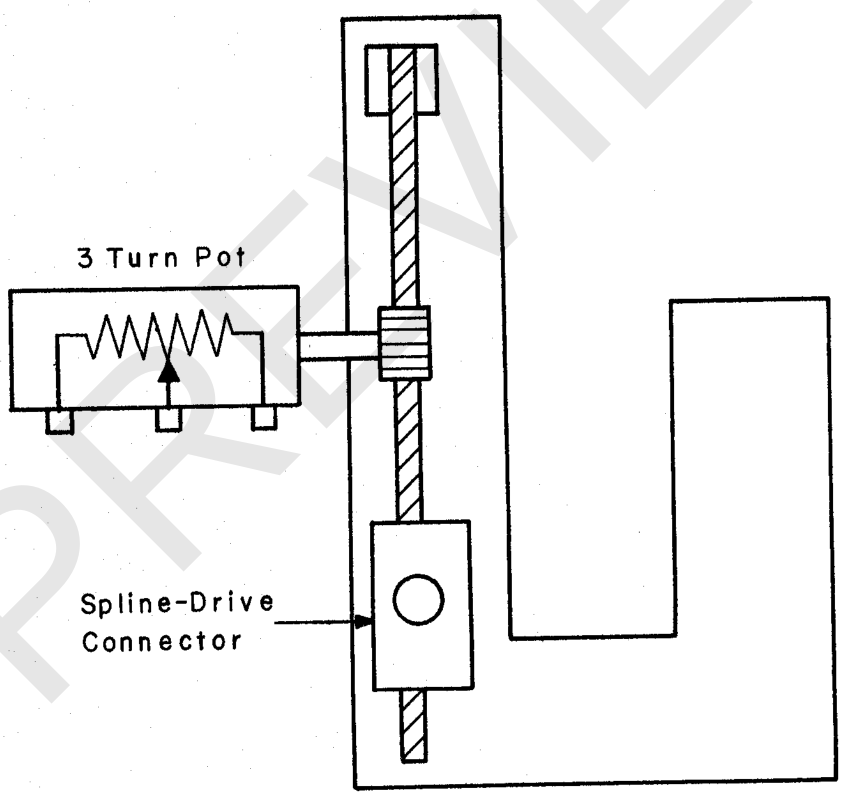
Film Shutter Control

Movement of the film shutter is achieved by utilizing a portion of the control system of a Heathkit, model EU20B, Servo Recorder. The control system, (See Figure 2.4)[2], was modified by connecting two servo motors in parallel (to obtain greater torque), and physically displacing the servo motors, and the three turn potentiometer from the control circuitry. The three turn, 1000 ohm, potentiometer has been fitted with a gear and the gear contacts a threaded shaft, (See Figure 2.3b), fixed to the film shutter. The threaded shaft is also meshed to a mechanical linkage, (consisting of a drive shaft, two universal joints, and a right angle spline), which feeds through the vacuum collar and is geared to the

[2]This figure was adapted from illustrations and descriptions located on pages 26-27 of the model EU20B Servo Recorder operations manual.



(a)



(b)

Figure 2·3
(a) Film Layout
(b) Film Shutter

servo motors. Movement of the servo motors thus; rotates the mechanical linkage; displaces the film shutter; rotates the potentiometer shaft; which alters the resistance setting of the potentiometer. The pot is in turn electrically connected to the control system as a feed-back unit. Details involving the shuttering system are in Appendix B.

Control system. When the input voltage terminals, (not to be confused with the ac line voltage), of the servo recorder are shorted together, a zero position control can be set to provide a zero point reference voltage developed across the three turn pot. This adjustment stipulates that the output of the chopper is zero when the input voltage is zero; and, in the original system was used to position a stylus at an arbitrary reference point on the chart paper.

A dc voltage applied at the input terminals is compared, by the chopper, with the reference voltage. The output of the chopper is amplified and drives the servo motors; which moves the film shutter; changes the resistance of the pot; and, in so doing, changes the reference voltage. Travel direction of the servo motors - and thus the film shutter - is controlled by the phase of the chopper output, which is determined by the algebraic difference between the input voltage and the reference voltage. The operational significance is: when a dc voltage is input, the film shutter moves until the reference voltage developed across the three turn pot is equal to the input voltage. A damping control desensitized this equality relation and prevents the film shutter from oscillating about a given point. Use of this