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SIZE EFFECTS AND ELECTRON MICROSCOPY
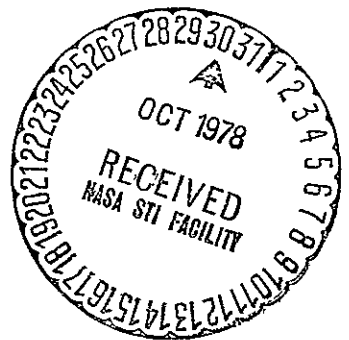
OF THIN METAL FILMS

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SIZE EFFECTS AND ELECTRON MICROSCOPY

OF THIN METAL FILMS

by

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THESIS

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INTRODUCTION

A thin metal film is a metal deposited onto a substrate with thickness ranging from several angstroms to several microns. The metal is deposited by means of vacuum evaporation, sputtering, or chemical deposition. Substrates come in varied materials such as glasses, ceramics, singlecrystal, and metal substrates. The choice of substrates used depends on the type of study to be conducted since the substrate has a significant effect on the growth and electrical properties of the thin film.

For the research conducted in this thesis, all films are deposited by resistive heated evaporation in an oil diffusion pumped vacuum system (ultimate $\simeq 10^{-7}$ torr). Growth of the thin film begins with the condensation of the metal on the substrate. Initially the atoms condense on the substrate surface in the form of small nuclei. These nuclei grow as the evaporation continues and form small islands. These islands grow and eventually, as the evaporation continues, coalesce and form a network of connected islands leaving voids throughout the film. Through continued deposition these channels are filled and form a continuous film. The growth from nuclei to a continuous film is highly dependent on the deposition parameters, evaporation rate as well as substrate material and substrate temperature.

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Figure 1 shows the growth stages of a film[1] while Figure 2 shows the dependence of grain size on various deposition and annealing parameters.[2]

Resistivity measurements are taken on thin films to observe size effects. For size effects to be observed the mean free path must be of the order of the thickness of the film. This ratio is designated by $K_o = a / l_o$, where a is the thickness and l_o is the mean free path. The range in which one would like to take measurements is where $.1 \lesssim K_o \lesssim 10$. This range of K_o can be approached by taking measurements of one film over various temperatures, since l_o can be lengthened or shortened by the lowering or raising of the temperature of the film, by measuring films of different thicknesses at one temperature or a combination of different films at different temperatures. For the metals gold[3] and silver[4] the ranges of l_o are:

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[1]Leon I. Maissel and Reinhar Glang, Handbook of Thin Film Technology (New York: McGraw Hill Book Company, 1970), pp. 8-33.

[2]Kasturi L. Chopra, Thin Film Phenomena (New York: McGraw Hill Book Company, 1969), p. 183.

[3]K. L. Chopra and L. C. Bobb, "Electrical Resistivity Studies on Polycrystalline and Epitaxially Grown Gold Films," Acta. Metal., 12 (1964), pp. 807-811.

[4]V. P. Nagpal and V. P. Duggal, "Geometrical Size Effect on Polycrystalline Silver Films," Thin Solid Films, 9 (1972), pp. 313-316.

Figure 1. Stages of film growth.

Figure 2. Dependence of grain size on (a) substrate temp-
erature; (b) rate of deposition; (c) film thickness; and
(d) annealing temperature.

NUCLEATION   NUCLEI GROW   COALESCENCE

CHANNELS   HOLES   ⟶ CONTINUOUS FILM
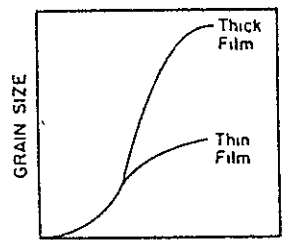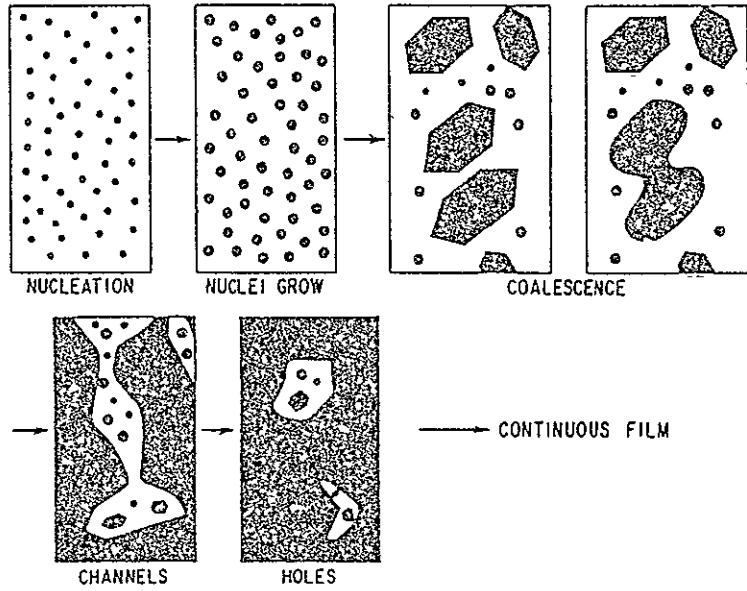
(a) SUBSTRATE TEMPERATURE
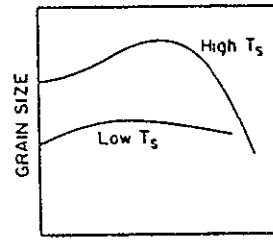
(b) DEPOSITION RATE
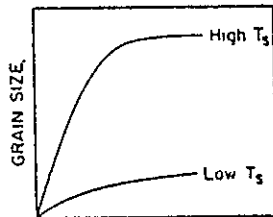
(c) FILM THICKNESS
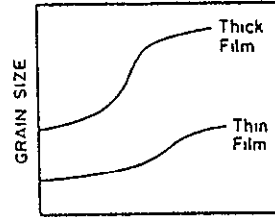
(d) ANNEALING TEMPERATURE

Gold	Silver

$l_o \simeq 400\text{\AA}$ at 298K $\qquad$ $l_o \simeq 570\text{\AA}$ at 300K

$l_o \simeq 1400\text{\AA}$ at 78K $\qquad$ $l_o \simeq 3200\text{\AA}$ at 77K

Therefore measurements are taken on a series of films of different thicknesses in a range of temperatures, although the minimum thickness of a film is seriously restricted at high temperatures. In studying thin metal films, those that are consistently analyzable and well studied are epitaxial films. These consist of single crystal or highly oriented films with large grains. Thus when surfaces are smooth no size effects are observed on these epitaxial films. If these films are grown simultaneously with polycrystalline films in the same evaporation then they would have the same amount of impurities. Therefore a difference between the two could arise from grain scattering in the polycrystalline films.

In the analysis of size effects in the epitaxial films the theory of Fuchs-Sondheimer[5] is used in which no grain boundary scattering is considered. In analyzing the polycrystalline films the theory of Mayadas and Shatzkes (M-S)[6] is used since it also incorporates the scattering of the grain boundaries. The resistivity of an epitaxial film

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[5]E. H. Sondheimer, "The Mean Free Path of Electrons in Metals," Advances in Physics, 1 (January, 1952), pp. 1-42.

[6]A. F. Mayadas and M. Shatzkes, "Electrical and Resistivity Model for Polycrystalline Films," Physical Review B, 1 (February, 1970), pp. 1382-1389.

with a smooth surface will approach that of the bulk mater-
ial. Thus the measured resistivity of an epitaxial film will
give the ρ_o to be used in the M-S analysis of the polycrys-
talline films. Knowing the value of ρ_o', l_o can also be
determined. Often ρ_o and l_o will be different from those of
the actual bulk values but ρ_o and l_o of the epitaxial films
should be similar to that of polycrystalline films when they
are evaporated simultaneously.

To grow epitaxial and polycrystalline films simul-
taneously in the same evaporation different substrates and
deposition temperatures will be required. An epitaxial film
is usually grown on a crystalline substrate such as mica or
NaCl at elevated temperatures while the polycrystalline films
are grown on non-crystalline substrates at room temperature.
The epitaxial films must be held at a temperature such that
the evaporated atoms have enough mobility to orient them-
selves along the substrate surface. Usually this tempera-
ture is well above that of the agglomeration point (this is
the temperature where the film begins to revert back to the
island structure of its earlier growth).

Looking again at Figure 1 one sees that there is a
variation of grain size with substrate temperature. Thus it
would be an advantage to be able to vary the substrate
temperature of the non-crystalline substrate independently
of the crystalline substrates in order to form films of
different grain size. Modifications have been made in the
experimental apparatus to carry out this type of study.

EXPERIMENTAL APPARATUS

In the research conducted for this thesis the ex-
perimental apparatus utilized is basically the same as that
described in the theses of Longbrake[7] and Clark.[8] In the
description of the apparatus emphasis will be on changes and
modifications.

Instrumentation

Control of deposition and measurement of films is
carried out through the use of a Hewlett-Packard, HP 9830A,
calculator interfaced with a number of peripheral devices.
Figure 3 is a block diagram of the automated system. The
major change in the peripherals is the replacement of the
custom built scanner by Clark with the HP 3495A scanner and
the addition of a temperature control device, DTC-2 Oxford
Digital Temperature Controller.

The DTC-2 monitors a platinum resistance thermo-
meter and the error between the desired temperature and the
measured temperature is used to control the amount of power

[7]R. L. Longbrake, "Size Effect and Annealing in
Thin Metal Films," Master's thesis submitted May, 1977 to The
University of Texas at El Paso, Physics Department.

[8]W. H. Clark, "An Automated Thin Film System For
Control of Evaporation and Measurement," Master's thesis sub-
mitted August, 1976 to The University of Texas at El Paso,
Physics Department.

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Figure 3. Block diagram of computer automated system.

HP 59303A Digital to Analog

Oxford DTC 2 Temp Controller

Programable Power Supply

Resistive Substrate Heaters

LN₂ Supply

LN₂ Tank

Substrate System

Film Shutter

HP 9866A Printer

Translator Control

Stepping Motor

HP 9830A Programable Calculator

Film Power Supply

Film Data Lines

TC Data Lines

HP 59308A Timing Generator

Quartz Crystal Oscillator

HP 3459A Scanner

HP 3490A Digital Multimeter

input

Evaporation Source

HPIB · IEEE Standard 488-1975 Interface Bus

HP 5345A Frequency Counter

Sloan Omni II Controller

Sloan Power Module

Transformer

to the heaters. The desired temperature is set by inputing

a signal of 0-5 volts into the DTC-2 using a digital to

analog converter. In addition the DTC-2 outputs a voltage

proportional to the temperature measured which is read by

the digital volt meter (DVM) and sent to the HP 9830A which

allows the temperature of the platinum resistance thermometer

to be monitored and recorded.

In the deposition system changes consist of the

addition of a thermocouple to the quartz crystal oscillator

housing and a source shutter. These attachments were made to

enhance the calibration accuracy of the Sloan thickness

monitor.*

The automated system is capable of evaporating

twelve films of eleven different thicknesses and making

resistance measurements from a temperature of 100K to 500K.

Vacuum and Substrate Systems

In this phase of the apparatus the change has been

rather dramatic. First the vacuum system used is a 45.72 cm

diameter and 80 cm high bell jar with a 15 cm Veeco, Model

VE-770, diffusion pump instead of the previous smaller sys-

tem, 10 cm diffusion pump and a 31 cm diameter 50 cm high

bell jar. The distance of the source from the substrate has

gone from 31 cm to 54 cm. With this increase there was a

smaller variation in film thickness over the substrate area

utilized.

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*See Appendix.

Initially the substrate system used was the same as
that described by Longbrake (see Figure 4). Since we wished
to vary the temperature of the two substrates independently,
the copper substrate holder was split into two separate
pieces. Several evaporations were carried out with this sys-
tem while still in the smaller vacuum chamber. A temperature
difference of only 55K was obtained. At this point the sub-
strate system was redesigned and moved to the larger vacuum
chamber. In order to increase the difference in temperature,
the copper posts were replaced with stainless steel tubes
capped with a copper plug at the ends. Through threaded
holes in the copper plugs the tank was attached to the split
substrate holder (see Figure 5). As shown in the figure the
tubes are asymmetrical in length so as to enhance the temp-
erature difference between each side of the plate. The idea
being that when the tank is full of liquid nitrogen (LN_2)
both tubes will be at the same temperature. But when the
tank is filled to below the protruding tube the side of the
substrate attached to it can be heated while the other side
continues to be cooled by LN_2.

This system operated well at a temperature of 400K
for the hot side while on the cold side the temperature was
200K. In the metals to be investigated the high temperature
side must be above 400K. As the temperature on the hot side
was raised above 400K we were unable to maintain independent
temperature control and the temperature difference rapidly
decreased. Several data runs were taken on this system but

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Figure 4. Substrate system.

LN$_2$ Reservoir

Fill and Discharge Lines

Metal Posts

Power Resistors

Set Screw

Set Screw

Substrate Holder

Terminal Block

Substrates

Spring Contacts

Film Mask

PC Board

Mask Holder

Film Shutter

Shutter Guides

Screws
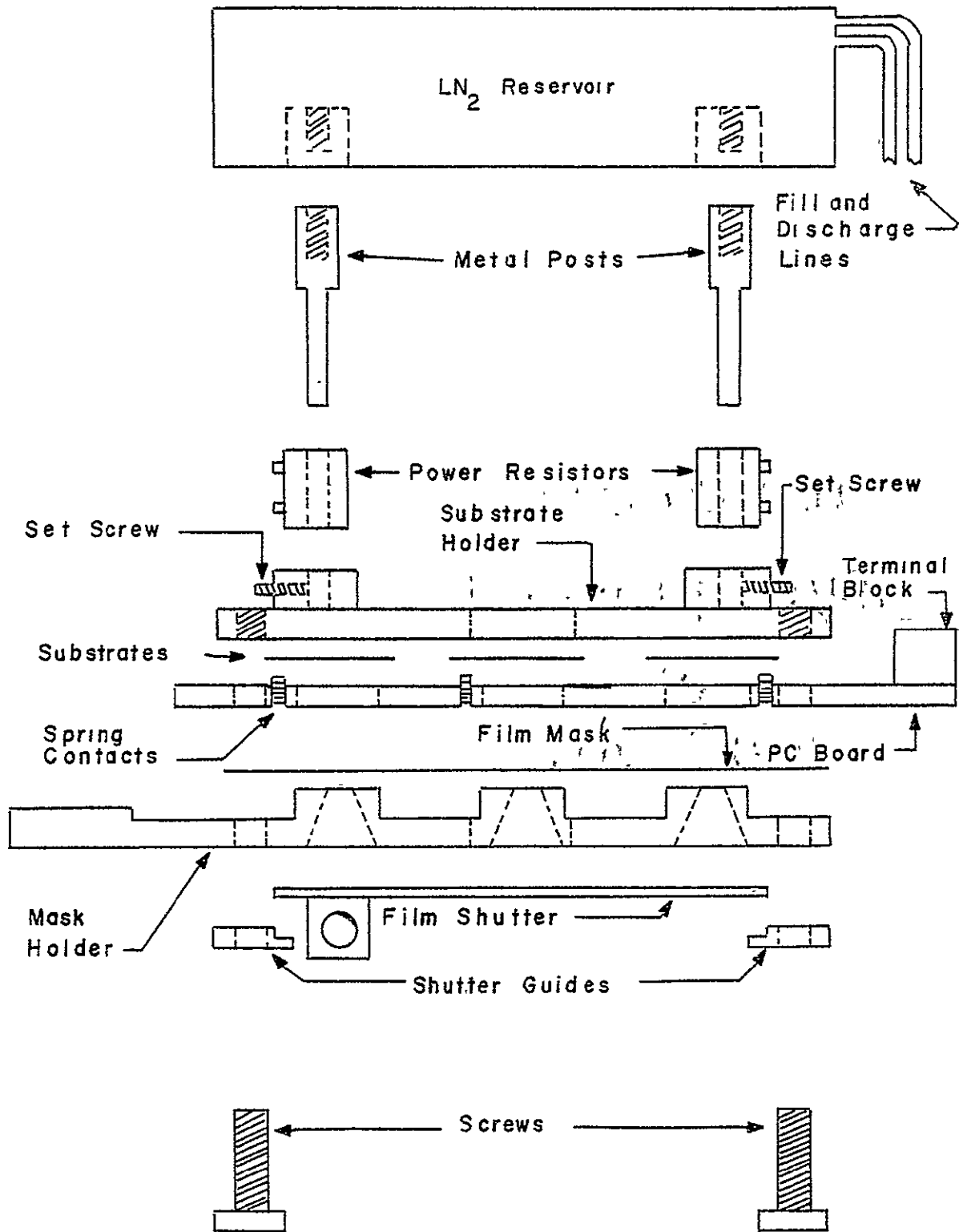
Figure 5. Split substrate system.

LN$_2$ In

N$_2$ Out →

LN$_2$

Heater

Copper Plugs

Heater

Cu Rod

Cu Rod

Copper Substrate Holder

Crystalline Substrates

Non Crystalline Substrates

Film Mask

Printed Circuit Board with Spring Contacts

Mask Holder

Shutter Mechanism
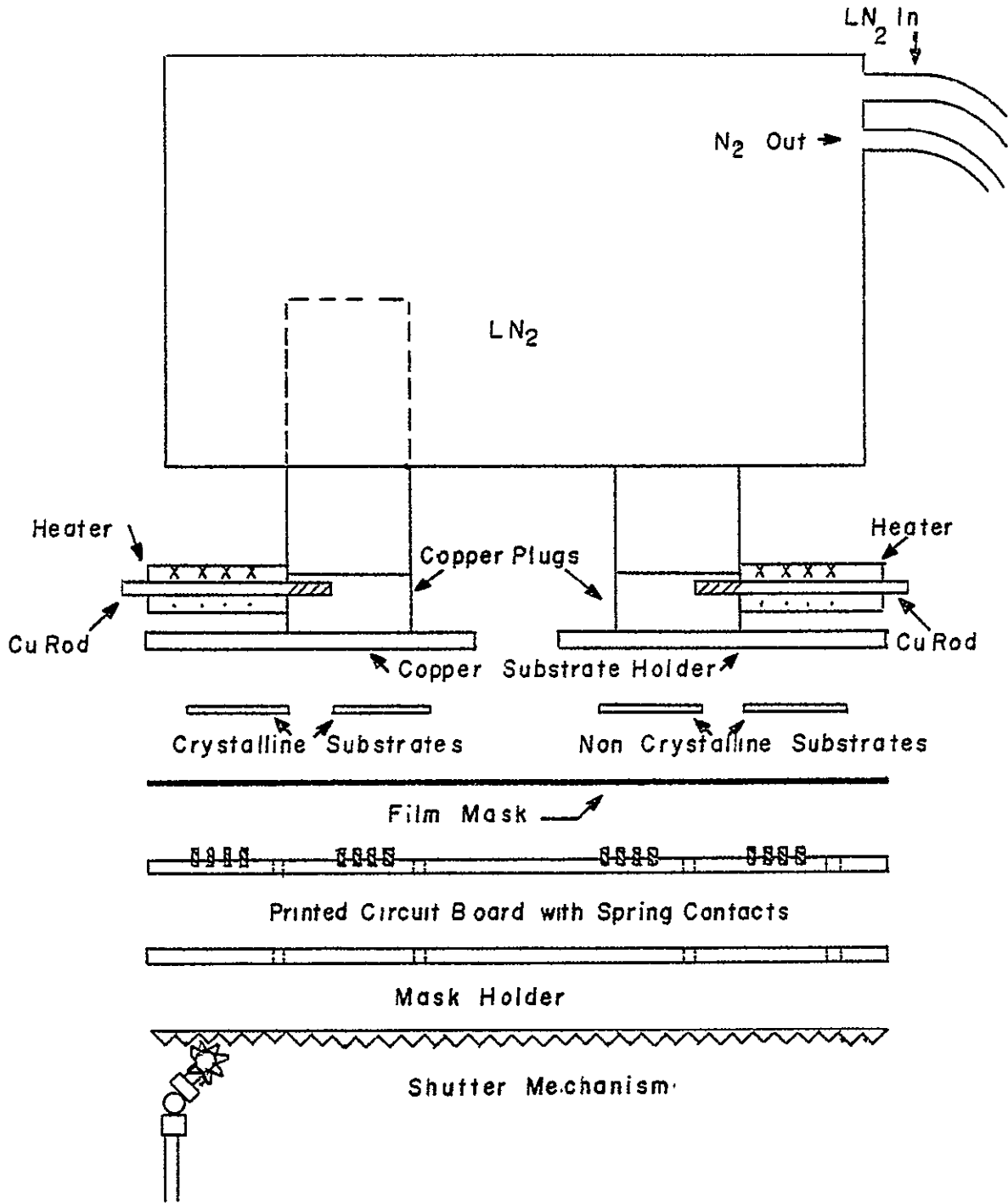
in the deposition mode no attempts at differences in sub-
strate holders were attempted.   In addition a continuous
substrate holder was used to insure uniform temperature
throughout the substrates.   The evaporations in this system
were all done at 450K.   Although this system was not used in
the mode for two different substrate temperatures, it was a
substantial improvement in extending the range of tempera-
tures measured down to 100K.

     In analyzing the past systems the problem was that
there was not a complete isolation of one side from the
other.   A substrate system was designed with a separate heat-
ing and cooling system for each part of the split substrate
holder.   Figure 6 shows this new system.   The tank and all
the tubing are stainless steel.   The tanks are isolated from
one another by glass ceramic rods (Corning-Macor) which joins
the two separate tanks and give the substrate system a very
rigid structure.   When this system was being proposed the
objective was to obtain temperature differences of at least
200K when the heated side of the substrate holder was raised
to at least 450K.   Also to enhance the temperature control at
low temperatures (100-200K) one must be able to raise the
temperature of the substrate holder while liquid nitrogen is
in the tanks.

     In testing this new substrate system a temperature
difference of 375K was achieved while maintaining the heated
side at 480K and cooled side at 105K.   It is also possible to
raise the substrate to 230K while the tank is full of LN_2.

Figure 6. Split Substrate System.

Heater   Copper Substrate Holder   Heater

Crystalline Substrates   Non Crystalline Substrates

Film Mask

Printed Circuit Board with Spring Contacts

Mask Holder

Shutter Mechanism
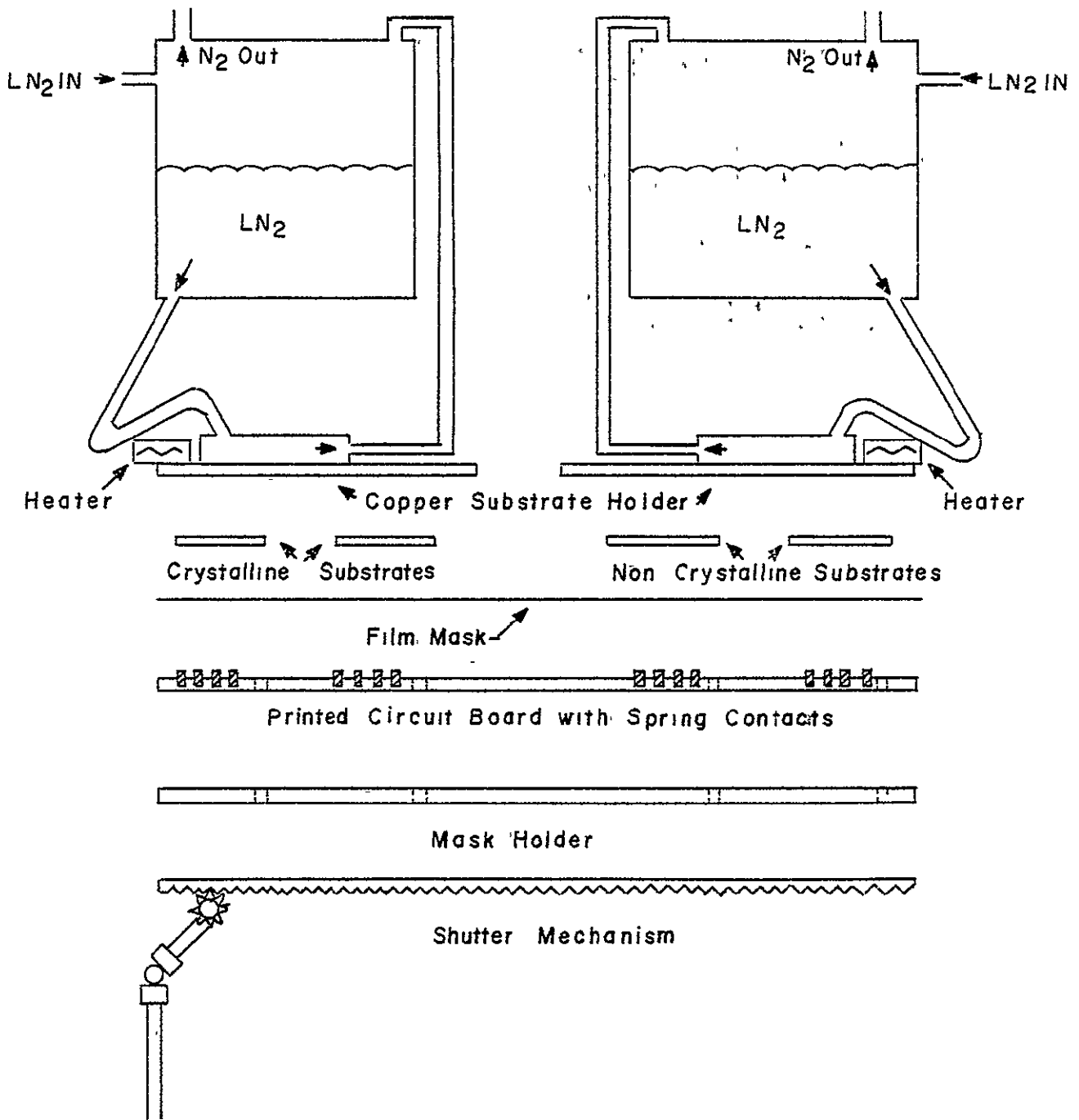
EXPERIMENTAL PROCEDURE

From start to finish there is approximately three weeks of work involved in one experimental run. It is divided among the preparation of the system, data taking, and analysis (measurement of film dimensions, reduction of resistivity data, taking of TEM pictures, measurements of grain size and determining degree of epitaxy).

System Preparation

In preparing for an experimental run the chamber and all components within must be thoroughly cleaned. Substrates to be used are cut to proper dimensions and cleaned.

Since several metals are used in the research, all traces of these must be removed from the components within the vacuum chamber before another evaporation takes place. Even if the same metal is to be studied the cleaner the system the higher vacuum attainable. Chemical etchants are used in cleaning components with large surface areas in the vacuum chamber while the smaller components are cleaned with Micro Cleaning solution in an ultrasonic cleaner. Once the system is reassembled an alcohol wipe is applied to the large surfaces just before putting it under a vacuum.

A variety of substrates are used in the research carried out. These include pyrex glass (cover slips), Corning 7059 glass, fused quartz, mica and NaCl. For all but

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the NaCl the preparations are the same. First they are cut to the proper dimensions using a tungsten carbide scribe on the glass and quartz although the mica is easily cut with scissors. The dimensions needed for one film and its metalizations are 2 x 2 cm. The thicknesses of the substrates are 1.59 mm for the fused quartz, .178 mm for the pyrex glass, .0076 mm for the mica and .533 mm for the Corning 7059 glass. Once cut to the proper dimensions, they are stored submersed in a Micro Cleaning solution until they are to be used. Just before they are to be attached to the substrate holder the container in which they are being stored is placed in the ultrasonic cleaner and the substrates are agitated for five to ten minutes. The mica is an exception to this and is left to be agitated less than one minute. If the mica is left in the ultrasonic cleaner for a longer period of time the agitation will begin to fragment the substrates. Next the substrates are rinsed off with distilled water and blown dry with a hot air gun, being careful not to leave any water spots on the substrates. They are now ready to mount on the substrate holder.

The NaCl was 2 x 2 x 5.02 cm when purchased so that thickness could be varied. The thickness depends on whether the NaCl is to be used alone or in combination with other substrates which would limit the useful range of thickness. Varying the thickness begins with first cleaving a substrate from the NaCl crystal. It is then ground down to close to the desired thickness and then polished to the final

dimension. The grinding process utilized a varying assort-
ment of lapping paper, ranging in grit size from 100 to .3
microns, and a tool on which the NaCl is mounted for grind-
ing and polishing. The polishing is done on a kit designed
for the polishing of NaCl windows. Since the thinnest film
to be studied is approximately $150\overset{o}{A}$ the defects on the sur-
face must be much less than $150\overset{o}{A}$.

The above procedure can be avoided if the NaCl is
evaporated onto mica substrates. Koch and Vook[9] report that
a $100\overset{o}{A}$ film of NaCl evaporated at room temperature on mica
will allow smooth epitaxial films of silver to be grown.

For growing epitaxial films, the crystalline sub-
strates must be at high temperatures during deposition. The
choice here is whether the non-crystalline substrates are to
be at the same temperature as the crystalline substrates or
at a different temperature. A continuous copper substrate
holder would be used for maintaining all films at the same
temperature during deposition. A split substrate holder
would be used if the non-crystalline substrates were to be
held at another temperature.

Once the substrates are attached to the substrate
holder, there is an initial evaporation of metalizations for
making electrical contact with films. If a different metal
is used for these metalizations than is used for the films

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[9]F. A. Koch and R. W. Vook, "Smooth Detachable, (111) Monocrystalline Ag Films," J. Appl. Phys., 42 (1971), pp. 4510-4511.

then the vacuum system is recleaned. The substrate system as shown in Figure 6 is now assembled and ready for film deposition.

Evaporation

The resistive heater evaporation source is a Sylvania Integral Tungsten Alumina Evaporation Crucible. Those with an especially wide opening are used so as to insure a more uniform evaporation.

Before the deposition of any films the substrate system is raised in temperature to bake off and outgas impurities. If there was to be an evaporation at high temperatures or annealing at high temperatures the system should be raised above that point during the outgassing procedure. Usually it is raised beyond the highest temperature to be used in either the deposition or measurement phase. The system is allowed to sit at this high temperature for a period of eight to ten hours while under a vacuum.

At the completion of this final cleaning procedure the substrates now are lowered to the deposition temperature. If a split substrate holder is being used the LN$_2$ reservoir is filled and the heaters for that side of the substrate holder turned off. The HP 9830A calculator then monitors the temperature of the substrate holder (hot side if the split holder is being used), and will indicate when the deposition controller should be activated. After the deposition controller is switched on the HP 9830A is in full

control until the deposition of films is complete, at which time a manual shutter over the source is engaged. See Clark, et al.,[10] for programing details.

When substrates of different material are used the shutter mechanism covers the films on the non-crystalline and crystalline substrates at the same time. Therefore there will be twelve films of six different thicknesses. The films are numbered from one to twelve and the pairs of films of corresponding thicknesses, from thinnest to thickest, are #1 and #5, #2 and #6, #3 and #7, #4 and #8, #9 and #10, #11 and #12.

[10]W. H. Clark, S. J. Brient, R. L. Longbrake and J. Elon Graves, "Automated Apparatus for Evaporation and in situ Measurements of Multiple Thin Films," Rev. Sci. Instrum., 49 (1978), pp. 24-30.

MEASUREMENT

When the film deposition is complete the heaters
are turned off and the substrates are allowed to cool down to
near room temperature ($\simeq$ 90 minutes) before the addition of
LN_2 which starts temperature cycling. This is for the case
of the continuous substrate holder and the hot side of the
split substrate holder. This procedure helps to conserve
LN_2 and reduce thermal shock to substrates and films. When
the split holder is being used then immediately after comple-
tion of film deposition LN_2 is added to cool that part of the
substrate that was at the lower temperature. This procedure
is used to reduce or prevent any annealing from taking place
before data can be taken.

Cooling of the films continues until they reach
100K at which point the measurement routine is initiated.
Depending on the film conditions and the type of data required
the process of taking data may last up to two or more days
running continuously. With the films at 100K the first of
four temperature cycles over which data will be taken is
begun. As the temperature of the films begins to rise, a
measurement sequence is initiated every 10K.

The range over which the films will be measured
depends on their agglomeration temperatures. If the films
were evaporated at well below their agglomeration temperature
then they should at most be raised to just below this

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agglomeration temperature. But when they are deposited at temperatures above their agglomeration temperatures then the deposition temperature should be the upper limit of the temperature cycle.

The first temperature cycle is to anneal the films. For a film deposition with substrates at 450K on gold or silver the temperature is cycled over the range from 100K to 450K. The films are then allowed to remain at the highest temperature from one to three hours in order to allow rapid annealing to take place. During the second cycle, the temperature of the films is dropped to 100K and resistivity data is again taken up to 450K at 10K intervals. These two data runs correspond to unannealed and annealed data. For the third cycle the temperature of the films is dropped to 100K but this time a very thin film of from 10 to 50Å is deposited on all the films before the temperature is raised. As the temperature is raised the thin overlayer is annealed. The fourth data cycle obtains measurements on the annealed double layer.

This procedure is basically the same when there are two deposition temperatures, i.e. in the case of the split substrate holder. The only difference would be that the films evaporated at the lower temperatures (those on the non-crystalline substrates) would agglomerate before the films evaporated at the higher temperature. Therefore as the data sequence is carried past the deposition temperature of the films on the cold side (those evaporated at the lower

temperature), the annealing temperature of these films will have to be monitored. When their resistance begins to increase sharply, indicating agglomeration, the temperature cycling will stop at that point and that temperature will be the upper limit for the films during subsequent temperature cycles.

After the last data set has been taken, the films are allowed to reach room temperature before bringing the system to atmosphere. Usually any anomalous behavior of a film during the measurement phase can be understood by looking at the condition of the films and their metalizations after they are removed from the vacuum system. In many cases problems with the films are caused by cracking of the substrates, poor alignment of films with their metalizations, extreme shadowing and noncontinuous films.

Upon completion of the electrical measurement phase thickness measurements (the parameter with the largest experimental error) are taken on the films. Two methods for taking thicknesses are available, a multi-beam interferometer (Sloan Angstrometer) and a profiling device (Sloan Dektak) which uses a stylus. The Dektak measure the film in the region where electrical measurements are actually made. The interferometer cannot measure this thickness without destroying the film for TEM analysis by overcoating. The stylus pressure is set to less than 10 milligrams so that thinner films will not be scratched and to obtain the greatest sensitivity of the Dektak. In taking measurements across the

width of the film, usually three measurements are taken. Each measurement is across the width of the film but in a different location along the length. Thus one can see how uniform the thickness is along the film and obtain an idea of the error in thickness measurements.

In addition to measurements of film thicknesses the Dektak is used to measure substrate smoothness. This is especially critical in the case of NaCl in order to determine when the defects are very much smaller than the films to be measured.

EXPERIMENTAL RESULTS

The four probe resistivity data obtained along with film thicknesses is presented in a normalized form as sheet resistance vs. temperature (sheet resistance = resistivity / film thickness). Table I shows the major deposition parameters of the runs whose data are presented here.

Run Z

Run Z is the only run which utilized the split substrate holder shown in Figure 4. The deposition temperatures 380K and 340K were significantly below the agglomeration temperature 450K.

In this run the thin film added as an overlayer was approximately 10 to 50Å and was evaporated at 220K. Normally following the fourth cycle the run would be terminated but in this run the substrates were then raised to 450K on the mica side and 395K on the glass side of the substrate holder and approximately 150Å was evaporated onto all of the films. Two additional temperature cycles were made up to a temperature of 450K the first to anneal the films evaporated on glass slides and the second to obtain annealed data.

The first of the annealed runs (second temperature cycle) is shown in Figure 7. The data is sheet resistance vs. temperature with the resistance normalized by dividing by sheet resistance at 273K. Pictured separately is the data for the film on mica and those on glass.

TABLE I

LIST OF RUNS AND THEIR SIGNIFICANT PARAMETERS

Run	Metal	Sub-strates	Deposition Temperature	Deposition Rate	Films Thicknesses (Angstroms)
Z*	Gold	Mica	380K	10 $\overset{o}{A}$/sec	170,245,398 550,734,854
		Pyrex Glass	340K		145,270,415, 567,735
F_1	Gold	Mica	450K	5 $\overset{o}{A}$/sec	330,340,650 975
		Pyrex Glass	450K		270,400,650 950
G_1	Silver	Mica	450K	14 $\overset{o}{A}$/sec	316,423,761 714,982
		Pyrex Glass	450K		461,753,910
H_1	Lead	Mica	450K	28 $\overset{o}{A}$/sec	2086,3429 5276,7481
		Pyrex Glass	450K		4269,6510, 9030

*The thickness used are those for the initial evaporation before the additional overlayers.

Figure 7. Normalized sheet resistance (normalized at 273K) of gold films on mica and glass for Run Z.
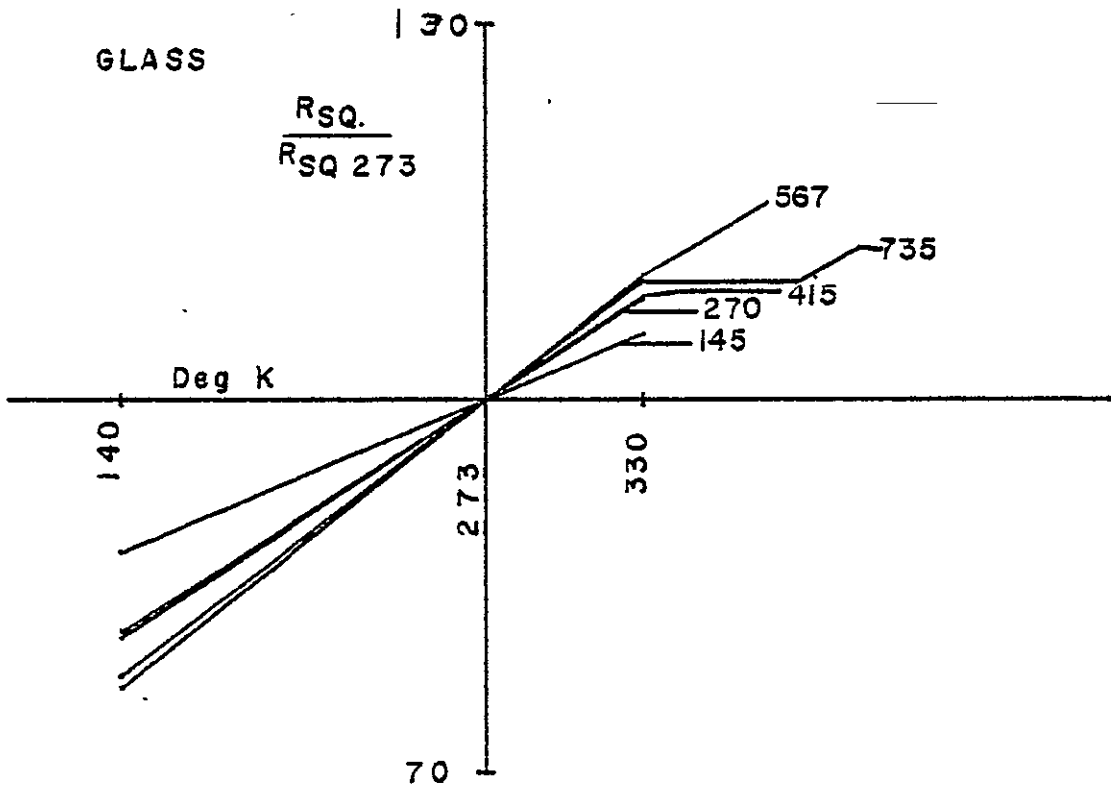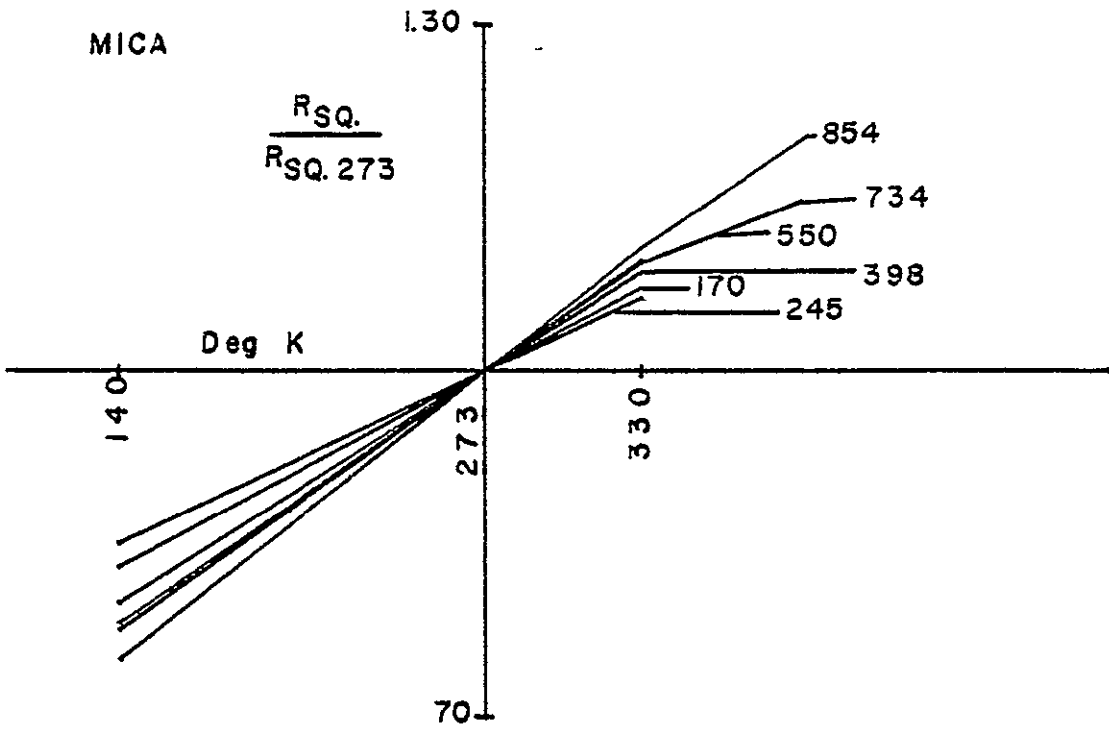
Run F$_1$
_____

      This run begins the series of evaporations where both glass and mica are held at 450K during deposition. Data for this run is taken in the range from 100K to 450K over four temperature cycles. Before the beginning of the third cycle a very thin film is deposited onto all films (approximately 10-50Å) while the substrates were at 100K. Figure 8 shows the second temperature cycle for mica and glass corresponding to annealed films.

Run G$_1$
_____

      In this run silver is evaporated at 450K well above the agglomeration temperature of all of the films. Figure 9 is a plot of this data. Again this is the data for the second temperature cycle.

Run H$_1$
_____

      Earlier runs were performed on lead using the same range of thicknesses as those of silver and gold but the resistances measured were orders of magnitude greater than those of the gold and silver data. Since the conductivity for lead is about an order of magnitude less than gold or silver the thicknesses of lead were made an order of magnitude greater than those on silver and gold. Looking at Figure 10 for the annealed data on mica and glass indicates very small or no size effect.

Figure 8. Normalized sheet resistance (normalized at 273K) of gold films on mica and glass for Run F_1.
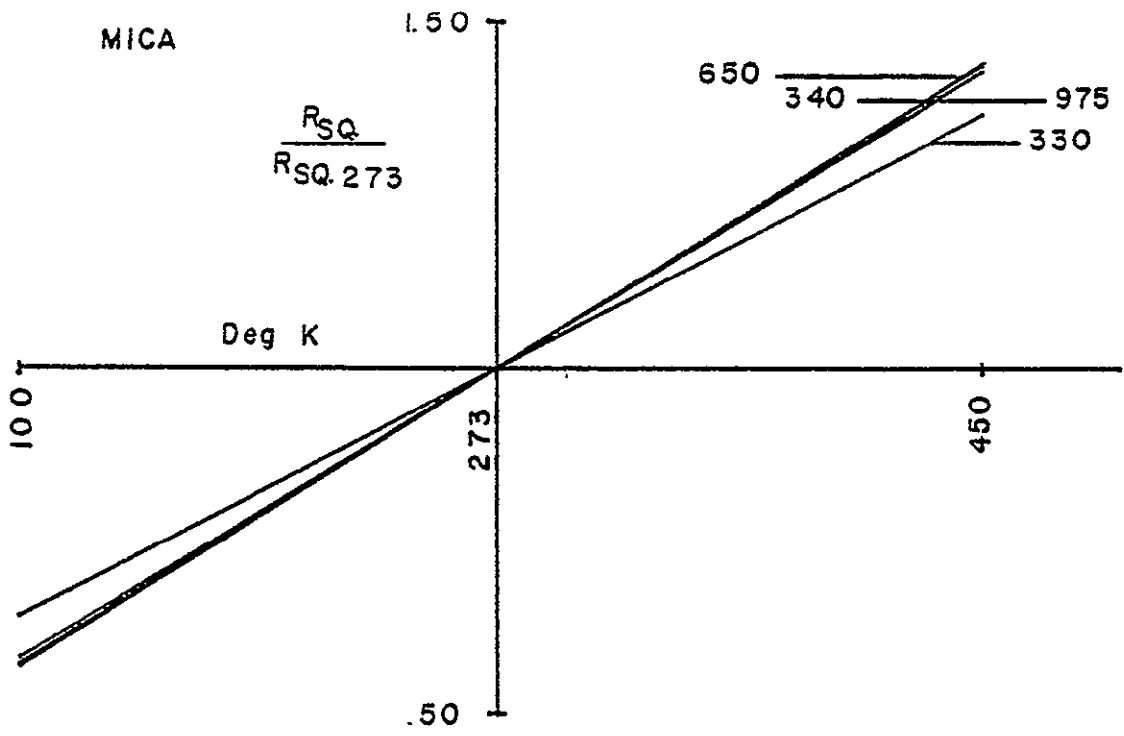
Figure 9. Normalized sheet resistance (normalized at 273K) of silver films on mica and glass for Run G_1.

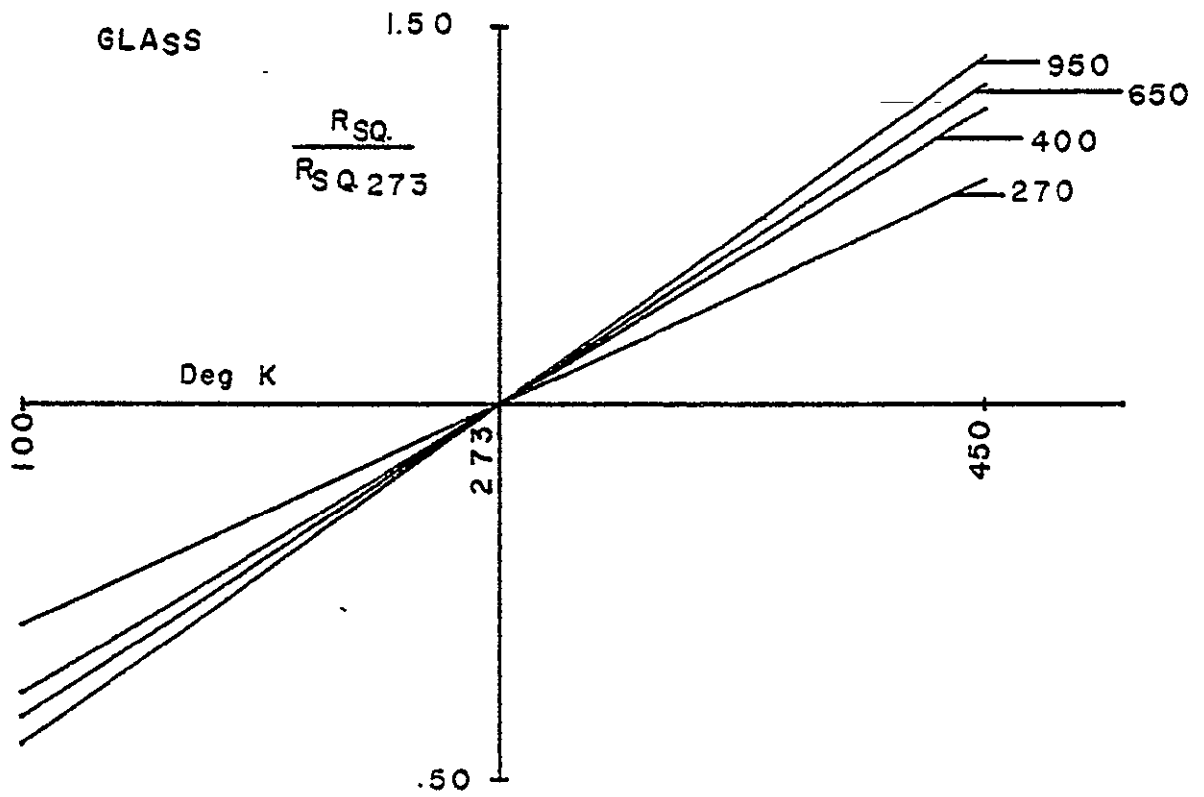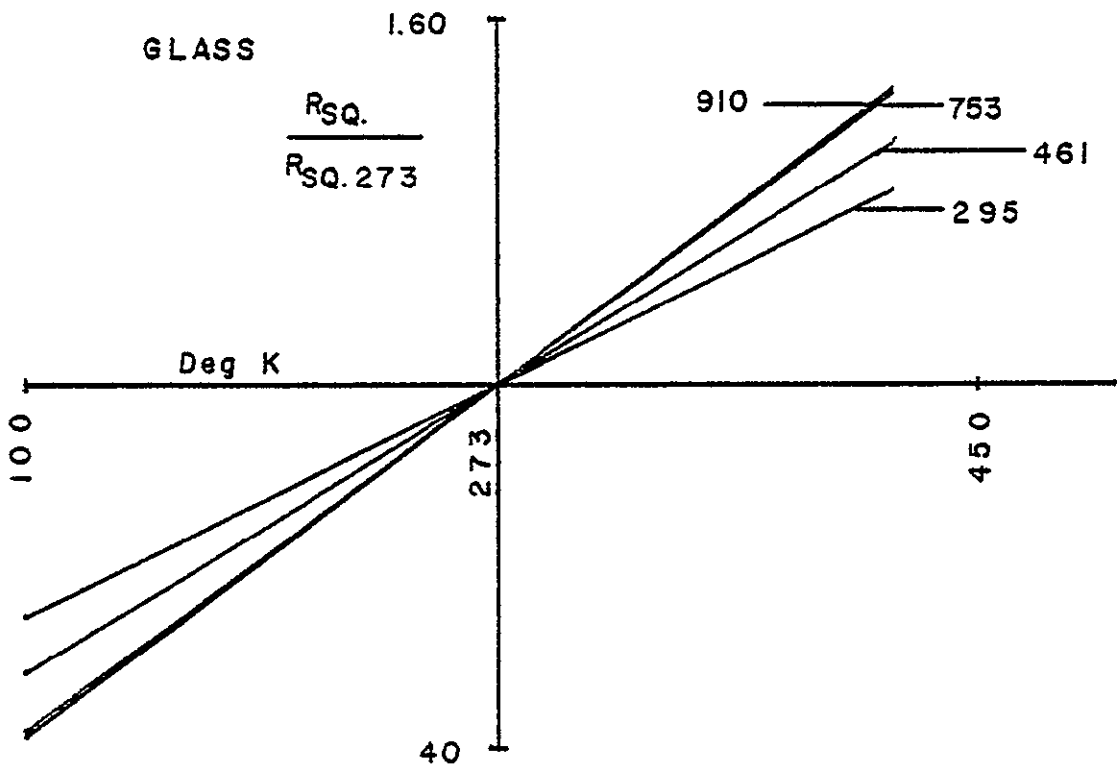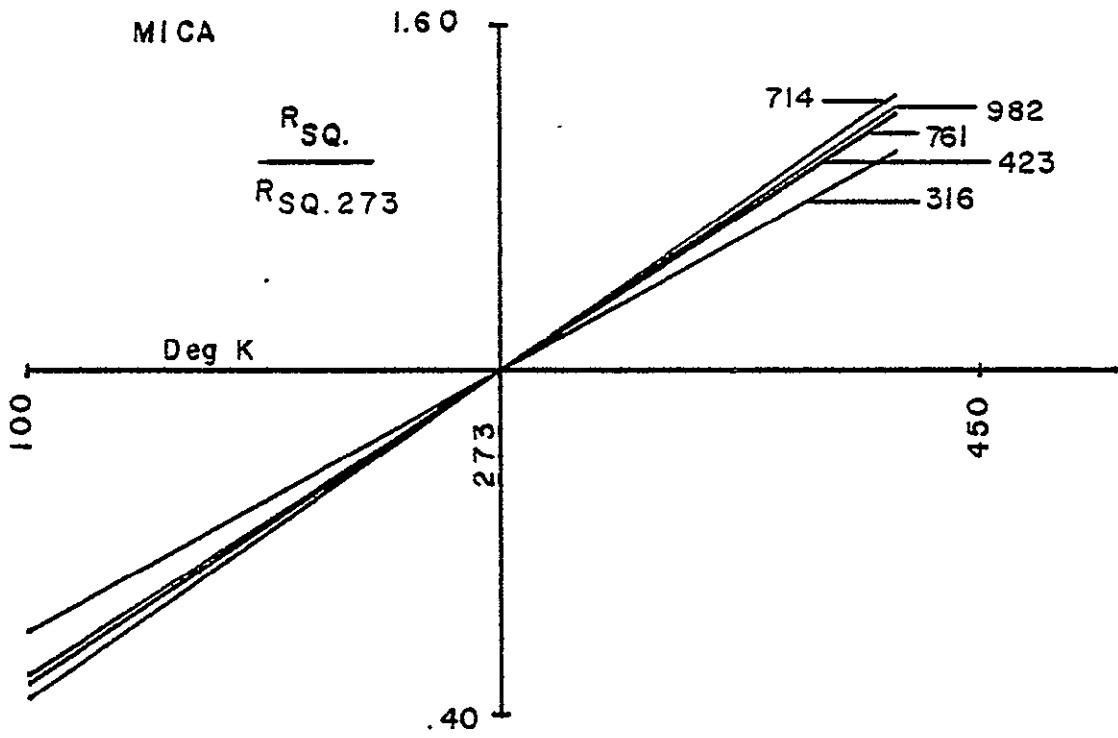Figure 10. Normalized sheet resistance (normalized at 273K) of lead films on mica and glass for Run H_1.

MICA

$$\frac{R_{SQ.}}{R_{SQ.273}}$$

1.70

Deg K

100

273

450

.30

7481 —— 5276

—— 3429

—— 1250

GLASS

$$\frac{R_{SQ.}}{R_{SQ.273}}$$

1.70

Deg K

100

273

450

.30

—— 9030

—— 6825

—— 6510

—— 4269
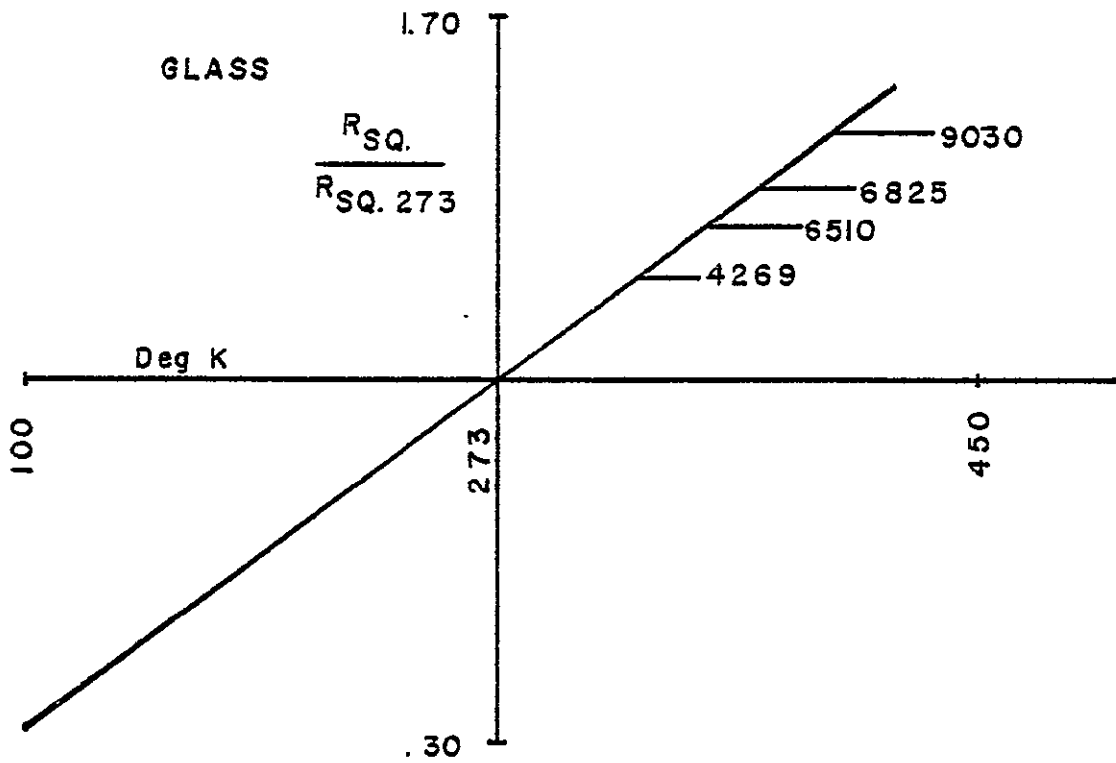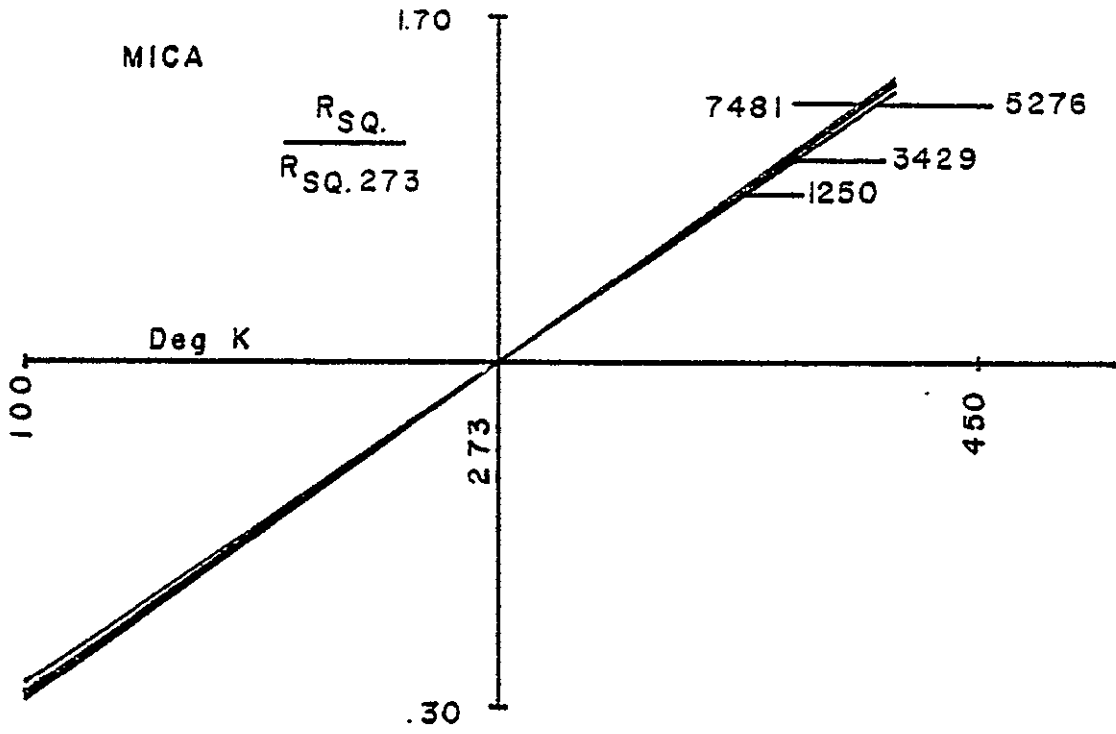
Table II is a list of the slopes and intercepts for the normalized data plotted.

TABLE II

SLOPES AND INTERCEPTS OF NORMALIZED DATA

Run	Thickness (Angstroms)	Slope ($\times 10^{-3}$)	Intercept
Z Mica	170	1.27	.652
	245	1.12	.695
	398	1.51	.589
	550	1.64	.551
	734	1.69	.539
	854	1.88	.487
Z Glass	145	.921	.749
	270	1.41	.616
	415	1.44	.606
	567	1.75	.523
	735	1.67	.544
F_1 Mica	330	2.07	.43
	340	2.47	.33
	480	2.26	.94
	650	2.49	.32
	975	2.42	.34
F_1 Glass	270	1.68	.54
	400	2.22	.39
	650	2.40	.34
	950	2.61	.29
G_1 Mica	316	2.62	.29
	423	3.06	.16
	761	3.06	.17
	714	3.30	.10
	982	3.15	.14
G_1 Glass	295	2.20	.40
	461	2.74	.25
	753	3.29	.10
	910	3.36	.08
H_1 Mica	1250	3.88	-.058
	2086	3.89	-.062
	3429	3.97	-.083
	5276	3.77	-.028
	7481	3.92	-.069
H_1 Glass	4269	3.88	-.060
	6510	3.92	-.069
	9030	3.88	-.060
	6825	3.87	-.057

ELECTRON MICROSCOPY STUDY

For the electron microscopy study a series of transmission electron·micrographs and diffractographs were taken of selected films from runs Z, F_1 (both gold) and G_1 (silver). A Zeiss EM10 electron microscope operated at 100kv was used. A 50 micron intermediate aperture was used for the selected area diffraction on all but one of the films (Figure 12e). The aperture size corresponds to a 1 micron specimen area. The micrographs allow the average grain size of a particular film to be determined. The lack of continuity, i.e., the amount of voids any particular film may have can also be determined from the micrographs. The degree of orientation, whether preferred or random, was achieved through the use of diffractographs. To some extent these diffractographs can be used to investigate the average grain size of a film. This is because the small aperture allows the diffraction spots to be resolved individually, such that the more dense the amount of diffraction spots the more grains in the area investigated.[11]

Run Z

The films from run Z are shown in Figure 11.

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[11]Gareth Thomas, Transmission Electron Microscopy of Metals (New York: John Wiley and Sons, Inc., 1962), pp. 28-29.

Figure 11. Run Z (a) film #1 295$\overset{\text{O}}{\text{A}}$, and (b) #4 773$\overset{\text{O}}{\text{A}}$ were deposited on mica at 380K, (c) film #5 270$\overset{\text{O}}{\text{A}}$, and (d) #8 790 $\overset{\text{O}}{\text{A}}$ were deposited on glass at 340K.
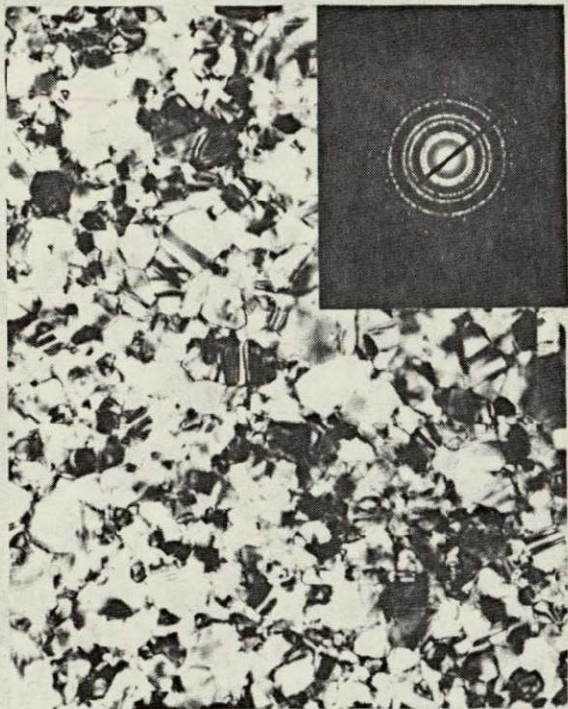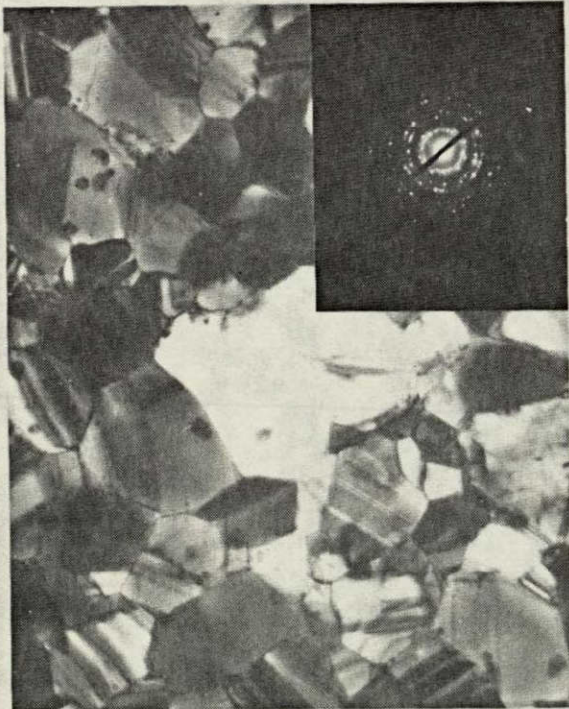
1700 Å

Figure 12.   Run F_1 (a) film #1 < 200Å, (b) #4 480Å, and (c) #12 975Å were deposited on mica at 450K, (d) film #5 < 200Å, (e) #7 400Å, and (f) #10 650Å were deposited on glass at 450K.

a

d

b

1700 A

e
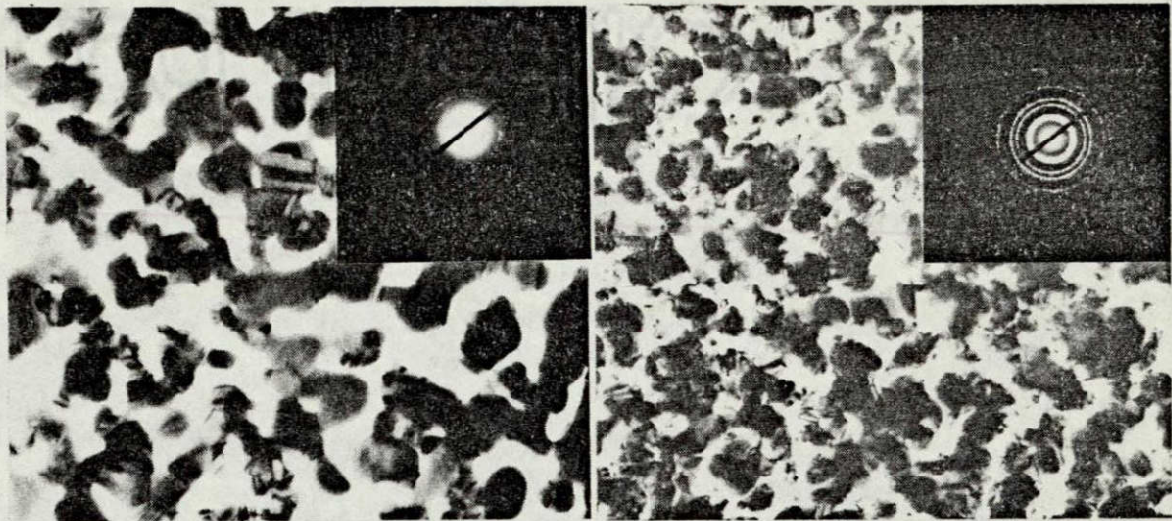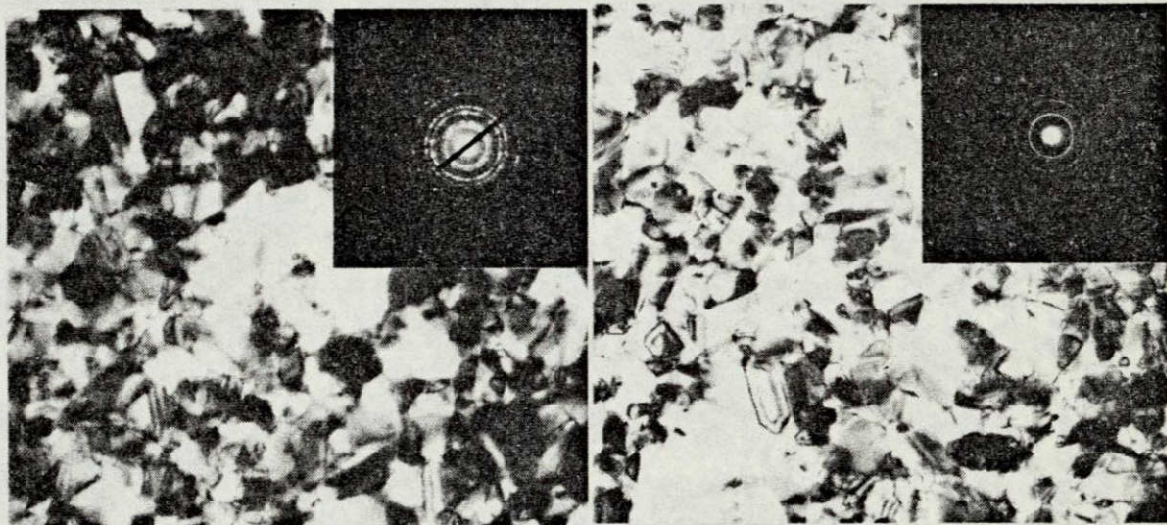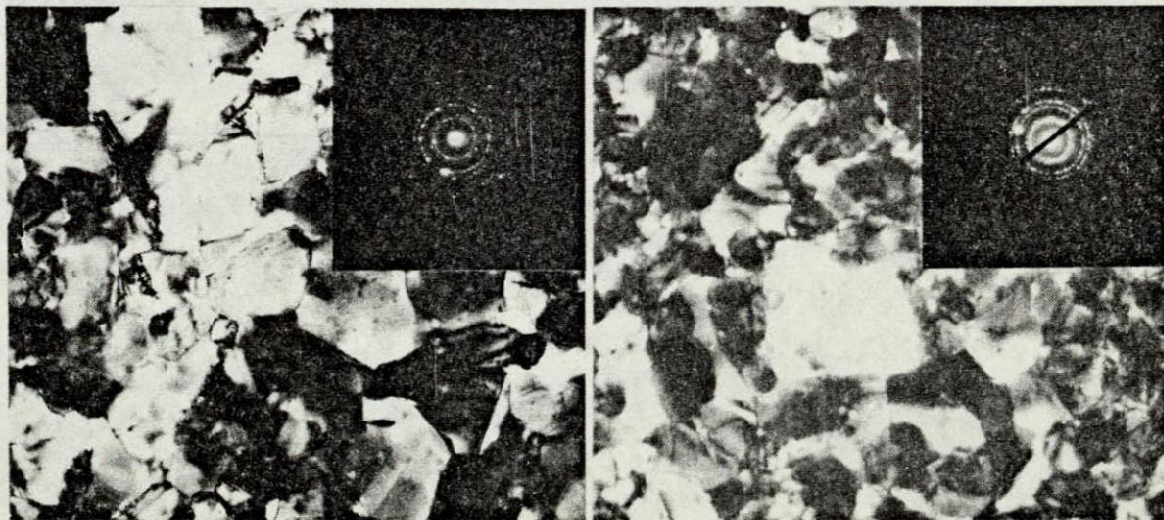
c

f

Figure 13. Run G_1 (a) film #3 423$\overset{o}{A}$, and (b) #4 761$\overset{o}{A}$ were deposited on mica at 450K; (c) film #6 295$\overset{o}{A}$ and (d) #8 753$\overset{o}{A}$ were deposited on glass at 450K.

├─── 1700 Å ───┤
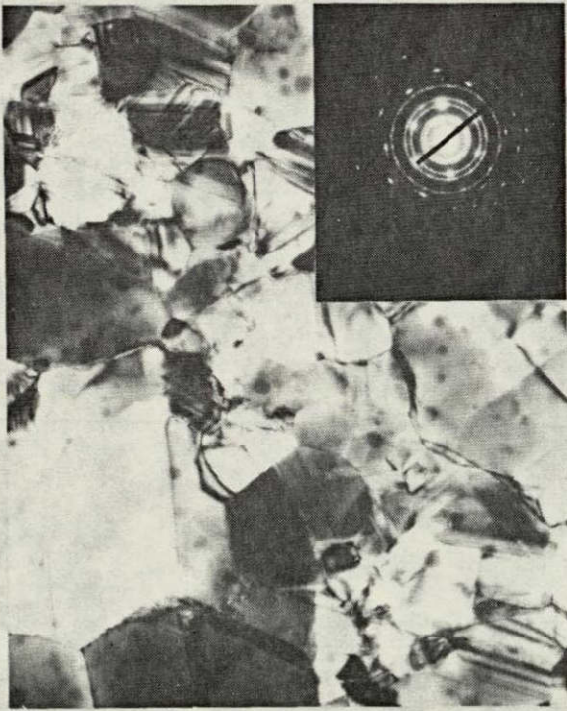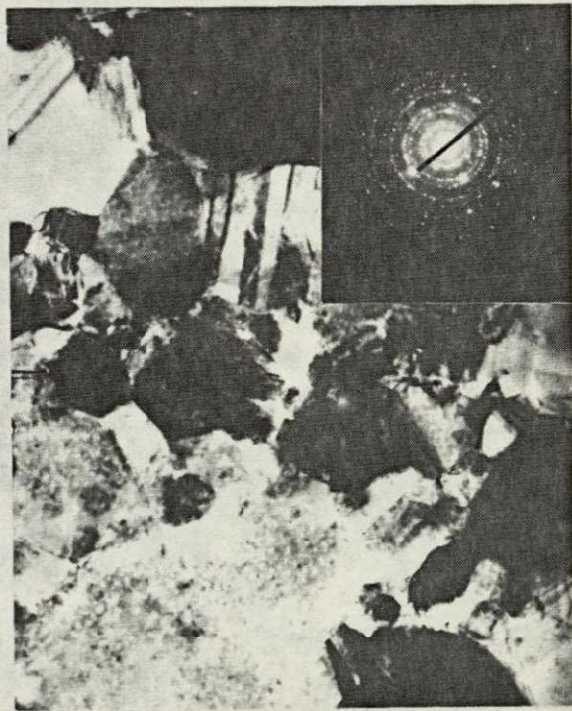
TABLE III

GRAIN SIZES OF FILMS

Run	Film #	Thickness	Grain Size
Z			
Mica	1	295Å	357Å
	4	773Å	880Å
Z			
Glass	5	270Å	473Å
	8	790Å	494Å
F_1			
Mica	1	200Å	305Å
	4	480Å	357Å
	12	975Å	678Å
F_1			
Glass	5	200Å	236Å
	7	400Å	446Å
	10	650Å	497Å
G_1			
Mica	3	423Å	883Å
	4	761Å	971Å
G_1			
Glass	6	295Å	572Å
	8	753Å	911Å

DATA ANALYSIS

For polycrystalline films where the grain size approaches or is less than that of the thicknesses, grain boundary scattering can become a significant factor in film resistivity. Mayadas and Shatzkes[13] (M-S) theory for incorporating grain boundary scattering is used for the data analysis along with the linear approximation to the M-S theory by Mola and Heras.[14]

The M-S theory is based on the assumption that there is an average grain diameter, D. The M-S theory is expressed by

$$\rho_f/\rho_g = [1-A/f(\alpha)]^{-1} \qquad (1)$$

where

$$A = \frac{6}{K_o \pi}(1-P)\int_0^{\pi/2} d\phi \int_1^{\infty} dt \frac{Cos^2\phi}{H^2(t,\phi)}\left(\frac{1}{t^3}-\frac{1}{t^5}\right)\left(\frac{1-e^{-K_o tH(t,\phi)}}{1-Pe^{-K_o tH(t,\phi)}}\right) \quad (2)$$

$$f(\alpha) = 1-(\frac{3}{2})\alpha+3\alpha^2-3\alpha^3 \ln(1-1/\alpha) = \rho_o/\rho_g \qquad (3)$$

$$\alpha/l_o = \frac{1}{D}\frac{R}{1-R} \qquad (4)$$

---

[13]A. F. Mayadas and M. Shatzkes, op. cit.

[14]E. Mola and J. Heras, "Numerical Evaluation of the Resistivity of Poly-Crystalline Metal Films with the Mayadas-Shatzkes Model," Electrocomponent Science and Technology, 1 (September, 1974), pp. 77-78.

$$H(t,\phi) = 1 + \alpha/Cos\phi(1 - \frac{1}{t^2})^{1/2}$$

ρ_f = Film resistivity

ρ_g = Resistivity from grain boundary scattering

ρ_o = Film resistivity without size effects

a = Film thickness

l_o = Mean free path of electron

D = Grain size

R = % of electrons scattered at grain boundary

P = Specularity parameter

K_o = a/l_o

The value of R can vary from 0 for no reflection of the electrons from the grain boundaries to 1 for every electron reflected and P varies from 0 for diffuse scattering from the surfaces to 1 for completely specular scattering.

One of the linearized approximations of M-S due to Mola and Heras which assumes D = a is:

$$\rho_f/\rho_o = 1 + M(P,R)l_o/a \qquad (5)$$

A functional form for M(P,R) was fitted to Mola and Heras' results by Longbrake.[15]

$$M(P,R) = [1.3049\ R/1-R + .457(1-P)] \qquad (6)$$

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[15] Ibid.

Therefore from a linear least squares fit of ρ_f vs. 1/a the slope is used to obtain the value of R over a range of P when the value of the product is known. The intercept yields ρ_o. If the assumption D = a is replaced by D = Ca then resistivity data where grain diameter is not equal to film thickness may be analyzed if the constant C can be determined.

An approximate form of M-S theory which makes use of the M(P,R) functional form is given by:[16]

$$a = \rho_g/R_{sq} + 6(1-P)(l_o/[\pi f(\alpha)])\int\int \qquad (7)$$

where

$R_{sq} = \rho/a$ and $\int\int$ is the double integral in eq. (2)

In rewriting the expression the largest error, thickness measurement, is approximately removed from the independent variable. A weighted least squares analysis of a vs. $1/R_{sq}$ yields the value of ρ_g if the $\int\int$ is assumed to be a slowly varying function of K_o and is calculated using an average value of K_o for a given temperature. From the intercept value an iterative calculation yields the value of P. The bulk values of resistivities for the material, ρ_o, are obtained from the published data of Meaden.[17] Utilizing the

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[16]J. Brient, Private communication.

[17]George Terence Meaden, Electrical Resistance of Metals (New York: Plenum Press, 1965), pp. 15-16.

ratio ρ_o/ρ_g and equation (3) the value of α was obtained.

The value of α/l_o was calculated for the gold and silver

film using the values of l_o determined from the values of

ρ_o and the product $\rho_o l_o$ from Hubin and Gouault.[18] Values

for R were determined, once the average grain size D was

available, from equation (4).

Results

Examination of the micrographs reveals that for

some of the films the grain size is relatively constant with

thickness and for others the grain size is proportional to

film thickness. Thus a separate analysis was used for each

of these groups.

Run Z. Since the final data cycle for this run

included an additional evaporation of 150$\overset{o}{A}$ and annealed to

a much higher temperature (450K) than the previous data cycles

(350K), the micrographs for this run correspond to the higher

annealing temperature as well as a multiple layer film.

From the analysis of the films on mica the micro-

graphs showed a grain size dependence on thickness for the

two thicknesses examined. Assuming a linear relation between

D and a, measurements on the micrographs yield the value of

the constant in the expression D = Ca and the linearized

form of M-S theory (equation 5) is used to analyze the data.

---

[18]M. Hubin and J. Gouault, "Resistivity and Thermo-
electric Power Between -100°C and +100°C of Gold and Silver
Thin Films Formed and Studied in Ultrahigh Vacuum," Thin
Solid Films, 24 (1974), pp. 311-331.

From Table IVa the values for R are shown under the linear-
ized M-S theory. This analysis indicates the reflection of
electrons at the boundaries is close to 100% (i.e. R = 1).
The analysis is independent of the value of P any value of P
from 0 to 1 yielding the same value of R. The data was also
analyzed by using the approximate theory of M-S (equation 7)
where an average of the two grain sizes was used for the
value of D. In this analysis the R values are of the order
of 60%.

For the films deposited on glass the grain size is
relatively constant. The approximate M-S theory (equation 7)
is used to analyze the data. An average grain size of $484\overset{o}{A}$
was used in calculating the reflection coefficients. Table
IVa lists the values of R and P for the range of tempera-
tures measured. The reflection coefficient is 60 to 70%, of
the same order as that of the mica films analyzed in the
same fashion. In addition the surface scattering is specu-
lar, P = 1.

For the initial data cycles there are no corres-
ponding micrographs. The M-S analysis for the approximate
form is applied to this data (equation 7). Results show
that the values of $\alpha/l_o [=1/D \ R/1-R]$ are of the same order
although smaller than those for the films on glass and mica
for the final annealed data. R values using the same $484\overset{o}{A}$
average grain diameters are indicated. It should be noted
that although micrographs are not available for the initial
deposition or for the thin overlayer deposition the structure

TABLE IVa

DATA ANALYSIS OF RUN Z — Au

APPROXIMATE M-S ANALYSIS

Substrate	Temperature (Deg. K)	$\alpha/l_0 \,(\times 10^{-3} \mathring{A}^{-1})$	P	R
Initial Deposition of Films				
Mica:	170$\mathring{A}$, 245$\mathring{A}$, 398$\mathring{A}$, 550$\mathring{A}$, 734$\mathring{A}$, 854$\mathring{A}$			
Glass:	145$\mathring{A}$, 270$\mathring{A}$, 415$\mathring{A}$, 567$\mathring{A}$, 735$\mathring{A}$			
Mica	144	2.26	1	.58
	295	2.38	0	.60
	337	3.37	0	.68
Glass	144	2.56	1	.55
	295	2.76	0	.57
	337	2.80	0	.58
Deposition of 10-50$\mathring{A}$ Onto All Films				
Mica:	195$\mathring{A}$, 270$\mathring{A}$, 423$\mathring{A}$, 575$\mathring{A}$, 759$\mathring{A}$, 876$\mathring{A}$			
Glass:	170$\mathring{A}$, 295$\mathring{A}$, 440$\mathring{A}$, 592$\mathring{A}$, 760$\mathring{A}$			
Mica	140	2.23	1	.58
	295	2.35	0	.59
Glass	140	2.53	1	.55
	295	2.83	0	.58

TABLE IVa (continued)

Substrate	Temperature (Deg. K)	α/l_o ($\times 10^{-3}$ $\overset{o}{A}^{-1}$)	P	R

Deposition of $\simeq 150\overset{o}{A}$ Onto All Films

Mica: $295\overset{o}{A}$, $490\overset{o}{A}$, $630\overset{o}{A}$, $773\overset{o}{A}$, $893\overset{o}{A}$, $1135\overset{o}{A}$

Mica D = $619\overset{o}{A}$

Glass: $270\overset{o}{A}$, $505\overset{o}{A}$, $647\overset{o}{A}$, $790\overset{o}{A}$, $894\overset{o}{A}$

Glass D = $484\overset{o}{A}$

Mica	151	2.58	1	.62
	294	2.78	1	.63
	455	2.79	0	.63
Glass	151	2.66	1	.56
	294	3.37	1	.62
	455	3.45	1	.63

Linearized M-S Theory

This analysis is for mica films after deposition of $\simeq 150\overset{o}{A}$.

Mica: $295\overset{o}{A}$, $490\overset{o}{A}$, $630\overset{o}{A}$, $773\overset{o}{A}$, $893\overset{o}{A}$, $1135\overset{o}{A}$

C = 1.09

Temperature (Deg. K)	$M\rho_o l_o$ ($\mu\Omega$-M-$\overset{o}{A}$)	ρ_o ($\mu\Omega$-M)	P	R
151	1116	3.4	1-0	.98
294	1100	5.o	1-0	.98
455	760	6.8	1-0	.98

factor α/l_o does not change significantly from that for the
final films.

Run F_1. The analysis of gold films on mica using
the linearized approximation to the M-S theory and D = Ca is
indicated in Table IVb. The first annealed data cycle shows
a reflection of approximately 90%. The reflection coeffic-
ient was not sensitive to the values of the specularity
parameter. After the addition of the 10-50Å deposit to all
the films the reflection coefficients' range was about 86%
irregardless of the value of P.

The approximate M-S theory was also performed on
these same mica films. The value of D used was an average of
the grain size of the three films examined. The values for
the reflection coefficients were approximately 35% for both
annealed data runs.

Those gold films deposited on glass were only ana-
lyzed by utilizing the approximate form of the M-S theory
expressed in equation (7). Significantly lower values of R
were obtained for the films on glass ($\simeq$29% compared with $\simeq$
36% on mica). Also the values of P indicate completely dif-
fuse scattering from films on glass substrates compared with
specular scattering from films on mica substrates.

Run G_1. Although these silver films, both those
evaporated on glass and those on mica, appeared to have grain
size that were not constant with thickness the linearized M-S
analysis resulted in ρ_o less than the Meaden values. When

TABLE IVb

DATA ANALYSIS OF RUN F_1 - Au

APPROXIMATE M-S ANALYSIS

Substrate	Temperature (Deg. K)	$\alpha/l_o (\times10^{-3})$	P	R

Initial Deposition of Films

Grain size used in analysis: Mica, 446Å. Glass, 437Å.

Mica: 330Å, 340Å, 650Å, 975Å

Glass: 270Å, 400Å, 650Å, 950Å

Substrate	Temperature (Deg. K)	$\alpha/l_o (\times10^{-3})$	P	R
Mica	117	1.14	1	.34
	293	1.26	1	.36
	446	1.37	1	.38
Glass	117	.874	—0	.28
	293	.968	0	.30
	446	.970	0	.30

Deposition of 10-50Å Onto All Films

Substrate	Temperature (Deg. K)	$\alpha/l_o (\times10^{-3})$	P	R
Mica	117	1.13	1	.34
	292	1.26	1	.36
	446	1.39	.7	.38
Glass	117	.850	.1	.27
	292	.930	0	.29
	446	.920	0	.29

TABLE IVb (continued)

Linearized M-S Theory

This analysis applied only to mica films.

Mica: 330$\overset{\circ}{A}$, 340$\overset{\circ}{A}$, 650$\overset{\circ}{A}$

C = .65

Temperature (Deg. K)	$M\rho_o l_o$ ($\mu\Omega$-M-$\overset{\circ}{A}$)	ρ_o ($\mu\Omega$-M)	P	R
117	443	1.27	1-0	.89
293	506	2.63	1-0	.90
446	614	3.69	1-0	.92

Deposition of 10-50$\overset{\circ}{A}$ Onto All Films

117	322	1.39	1-0	.85
292	342	2.76	1-0	.86
446	391	3.84	1-0	.87

the approximate M-S analysis of equation (7) was applied to these films, both the films on glass and mica resulted in reflections in the order of 10 to 20% and large P values indicating specular services as shown in Table IVc.

Run H_1. Since the thicknesses of these films were so large in comparison with the three previous runs micrographs of these could not be obtained. The approximate form of M-S analysis, equation (7), is indicated in Table IVd.

For the mica in the first annealed data run the range of α/l_0 $(x10^{-3})$ is from .53 to 2.2 while for the glass the values are .88 to 5.1. After a $\simeq 150\overset{o}{A}$ film is deposited on all the films and annealed the values of α/l_0 $(x10^{-3})$ for mica are .58 to 2.5 and .94 to 5.4 for glass. The variation of α/l_0, the structure factor, with temperature indicates the weak dependence of this data on size effect as previously noted when discussing the data for this run in Figure 10.

TABLE IVc

DATA ANALYSIS OF RUN G_1 - Ag

APPROXIMATE M-S ANALYSIS

Substrate	Temperature (Deg. K)	α/l_o (x$10^{-3}$$\overset{\circ}{A}^{-1}$)	P	R

Initial Deposition of Films

Grain size used in analysis: Mica, 827$\overset{\circ}{A}$. Glass, 742$\overset{\circ}{A}$.

Mica: 316$\overset{\circ}{A}$, 423$\overset{\circ}{A}$, 761$\overset{\circ}{A}$, 714$\overset{\circ}{A}$, 982$\overset{\circ}{A}$

Glass: 461$\overset{\circ}{A}$, 753$\overset{\circ}{A}$, 910$\overset{\circ}{A}$

Substrate	Temperature	α/l_o	P	R
Mica	105	.245	.84	.17
	292	.306	.74	.20
	456	.345	.65	.22
Glass	105	.150	.68	.10
	292	.290	.94	.18
	456	.434	.73	.24

Deposition of 10-50$\overset{\circ}{A}$ Onto All Films

Substrate	Temperature	α/l_o	P	R
Mica	105	.236	.83	.16
	295	.287	.84	.19
	397	.319	.67	.21
Glass	105	.137	.67	.09
	295	.237	.97	.15
	397	.380	.70	.22

TABLE IVd

DATA ANALYSIS OF RUN H_1 - Pb

APPROXIMATE M-S ANALYSIS

Substrate	Temperature (Deg. K)	$\alpha/l_o (x10^{-3} \overset{o}{A}{}^{-1})$	P
	Initial Deposition of Films		
Mica:	$2086\overset{o}{A}$, $3429\overset{o}{A}$, $5276\overset{o}{A}$, $6379\overset{o}{A}$, $7481\overset{o}{A}$		
Glass:	$4269\overset{o}{A}$, $6510\overset{o}{A}$, $9030\overset{o}{A}$		
Mica	103	.534	1
	298	.922	1
	430	2.24	1
Glass	103	.881	1
	298	3.13	1
	430	5.09	1
	Deposition of $\simeq 150\overset{o}{A}$ Onto All Films		
Mica	102	.576	1
	295	.916	1
	451	2.53	1
Glass	102	.94	1
	295	3.03	1
	451	5.38	1

DISCUSSION

The analysis of size effect of the films using M-S
Theory is very sensitive to the values of a. A 5% variation
of a, well within the error of thickness measurements, is the
difference between analyzable data and that which is not ana-
lyzable. This emphasizes the need for accurate and high
resolution thickness measurements. There is also a question
about whether the thermocouples are measuring the changing
temperature of the films when they are located on the front
face of a substrate on the substrate holder. This can be
resolved if thin film thermocouples are deposited adjacent
to the films and used to monitor the temperature.

APPENDIX

QUARTZ CRYSTAL CALIBRATION

During the film deposition the thickness growth of
the films is monitored through a quartz crystal oscillator.
The thickness is determined from the frequency of the quartz
crystal by using the relation $T = 2F/D$, where T is the thick-
ness, F is the frequency change, and D is the density of the
metal. Calibration is achieved by determining an equivalent
density.

Initially the bulk density is used in the evapora-
tion. Once the data runs have been completed and film thick-
nesses are measured the oscillator is calibrated. From the
above relation it can be seen that a plot of $2F$ vs. T gives
the density of the metal.

Of course this calibration only holds when the same
geometry is repeated and the same deposition parameters are
used. In order for this calibration to hold true in addi-
tion to maintaining the same system configuration the quartz
crystal oscillator must be maintained at the same tempera-
ture throughout the evaporation. The reason for this added
control is that the quartz crystal frequency is very sensi-
tive to temperatures. Figure 14 is a graph of the frequency
response with temperature.[19] From the figure the most
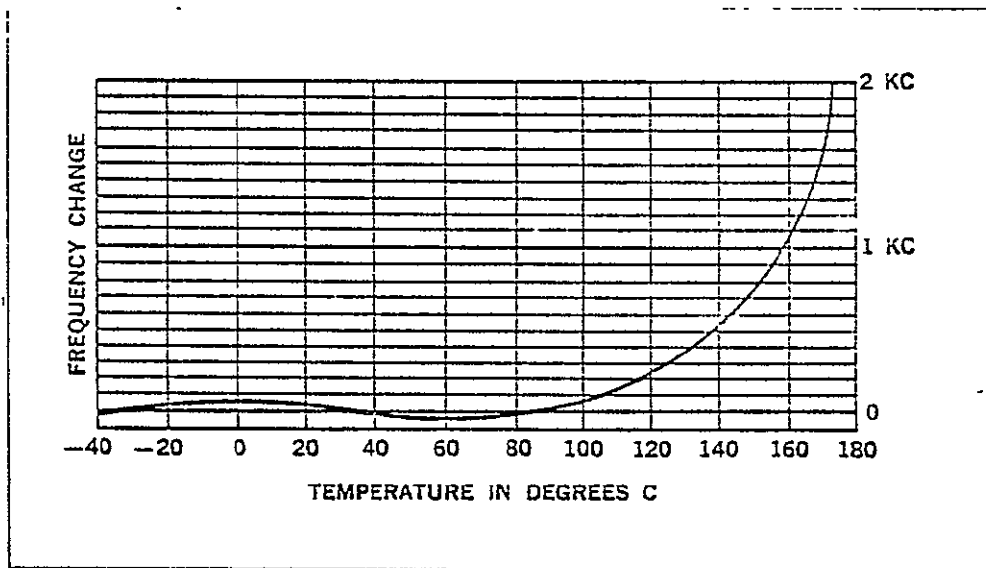desirable range to be in is that from 20° C to 80° C since a

[19]Sloan Technology Corporation, "Sloan OMNI IIA
Deposition Control Master Manual," p. 4-3.

68

Figure 14. Temperature Coefficient of Oscillation of Quartz
Crystals.

FREQUENCY CHANGE

2 KC

1 KC

0

-40 -20 0 20 40 60 80 100 120 140 160 180

TEMPERATURE IN DEGREES C

temperature change in this range results in a less than 10%
frequency change.

In order to monitor this temperature change a ther-
mocouple was attached to the oscillator housing. It was
placed in such a way as to be in contact with the quartz
crystal but not interfere with its oscillations. A reservoir
capable of heating water was attached to the copper tube that
is attached to the housing. This copper tube is used as a
conduction cooling line. By controlling the temperature of
the water through it the temperature of the oscillator can be
maintained within a desired range. Since the stainless steel
housing is relatively massive the temperature effect of the
heated water reservoir was not as large as anticipated.
Another crystal oscillator holder was obtained which has only
the housing for the crystal inside the vacuum chamber. This
crystal holder being much less massive will be more suscept-
ible to temperature control. Table V contains calculated
values for the effective density for several runs.

TABLE V

QUARTZ CRYSTAL OSCILLATOR CALIBRATION

Run	Rate	Substrate Temperature	Density (gm/cm^3)	Calibrated Density	Range of Thickness	Temperature Change of Oscillator
Z-Au	10$\overset{o}{A}$/s	340K	19.3	18.7	300- 1,100$\overset{o}{A}$	320-322K
F_1-Au	5$\overset{o}{A}$/s	380K 450K	19.3	18.75	200- 1,000$\overset{o}{A}$	401-403K
G_1-Ag	14$\overset{o}{A}$/s	450K	10.5	10.96	300- 1,000$\overset{o}{A}$	399-400K
H_1-Pb	28$\overset{o}{A}$/s	450K	11.3	11.0*	4,000-15,000$\overset{o}{A}$	392-395K

*Only thicknesses of films on glass used.

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