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METHODOLOGY FOR ESCA ANALYSIS OF ELECTROMIGRATION

SUNY Cortland

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This preliminary investigation examines the feasibility of using Electron Spectroscopy for Chemical Analysis (ESCA) to monitor electromigration in thin films. Because of the requirements of both the ESCA technique and the requisite in situ electromigration, the experimental methods are quite exacting. We report here designs and experimental methodology to permit the examination of electromigration with ESCA and suggest appropriate avenues for future research.

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Electromigration in thin films may be studied using Electron Spectroscopy for Chemical Analysis (ESCA). Because of the requirements of both the ESCA technique and the requisite *in situ* electromigration, the experimental methods are quite exacting. We report here designs and experimental methodology to permit the examination of electromigration with ESCA.

In the work reported here we have developed the methodology to examine electromigration voids using Electron Spectroscopy for Chemical Analysis (ESCA). It is known that electromigration in thin conductor line occurs primarily along grain boundaries[1]. In modeling electromigration, then, it would be useful to know the changes in the forces that a migrating atom experiences as it travels along the grain boundaries. The changes in the forces that a given atom experiences are reflected in the energy levels of that atom's electrons. Thus, by measuring shifts in the electronic energy levels of the atoms undergoing electromigration one can deduce changes in the local environment of the atom.

ESCA is a photoemission technique, wherein impinging x-rays (of energy hν) cause the emission of core-level or near-core-level electrons from the surface to be studied. Once the electron has left the surface it can be captured and its kinetic energy measured. From the kinetic energy (KE) of the electron (and a knowledge of the work function, Φ, of the surface) the electrons binding energy (BE) can be determined. Since energy must be conserved,

$$KE = h\nu - BE - \Phi$$

A change in the chemical environment of the atom may result in a shift in the binding energy of the electron. The binding energy of the electron, BE, then is equal to the original binding energy, BE_O, plus the shift, Δ. Hence the observed kinetic energy is

$$KE = h\nu - BE_O - \Phi - \Delta$$

Although the theory of such a measurement is quite straightforward, the application of this technique to electromigration in thin films is experimentally challenging. The specific difficulties involved, and the steps taken to solve them, will be discussed in detail in the following pages. These experimental challenges can be divided into two categories, although there is some overlap

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[1] P.S. Ho and T. Kwok; Rep. Prog. Phys. 52 (1989) 301 and references therein.

between the two. First, much of the equipment necessary is not commercially available, simply because photoemission analysis of electromigration is not commonplace (indeed, to our knowledge has not been attempted in any other lab). Modifications to the available ESCA system needed to be designed, built, and installed. Second, because of the characteristics of the ESCA technique, test stripes commercially available for electromigration or other reliability testing were inappropriate. Appropriate samples, therefore, also had to be designed.

MODIFICATIONS TO THE ESCA CHAMBER

To understand the importance of the various modifications to the test chamber it is necessary to understand the experimental requirements for the intended measurement. First, because photoemission is inherently a surface technique, the experiment must be performed on atomically clean surfaces under ultra-high vacuum (UHV -- generally pressures of 10^{-9} or 10^{-10} torr) conditions. This has several ramifications. For one, it means that the electromigration of the samples must be done *in situ*. To get meaningful data about electronic shifts that come about due to void formation it is necessary to take spectra of a cleaned sample both before and after the electromigration. Almost any material (and the aluminum from which the test stripes are made, in particular) becomes contaminated on exposure to air. The electromigration, therefore, must be done without exposure to air (that is, the sample must remain under UHV) to insure that the spectra taken before and after the electromigration are of exactly the same surface, except, of course, for any changes brought about by the electromigration.

A second result of the UHV requirement is that the choices for materials to be used for chamber modification are severely limited. Any material used must have an extremely low vapor pressure so that ultra-high vacuum may be maintained. The materials must be vacuum compatible over a large temperature range since the parts may be subjected to many heating and cooling cycles in the course of the experiment.

Most of the other experimental requirements are needed to accommodate the electromigration done in the UHV chamber. One obvious requirement is that the sample needs to be electrically isolated from the chamber. The chamber must also be outfitted with leads to carry the current to the sample. The leads to the sample must be shielded with a UHV compatible material.

To do a thorough analysis one would need to measure shifts due to void formation under a range of temperatures and applied currents. By changing these variables independently one could ascertain which changes are truly a result of the electromigration voiding alone and which are a result only of the inherent Joule heating. Thus, external heating and cooling of the sample must also be provided, as well as a means of accurate temperature measurement at the sample.

In order to facilitate measurement on a large number of samples, it was decided to try to meet all of the above requirements within the restrictions of the existing Perkin-Elmer (P-E) sample introduction system. This system allows for the exchange of samples within the UHV system without breaking vacuum. This requirement makes the design much more complex, since now all supports and connections must be make-or-break, but still reliable.

Figure 1a. Macor sample holder cap with power leads and thermocouple.

Figure 1b. Macor sample holder cap with position of the sample and the sample base.

The Perkin-Elmer sample holders designed to fit through the sample introduction system are molybdenum cylinders a half inch tall and an inch in diameter. In order to provide electrical isolation, Macor caps were designed to fit over the molybdenum holder as shown in figure 1 and schematic A. Macor is a machinable ceramic composite material with an extremely low vapor pressure and high electrical resistivity. This makes it an ideal material to provide electrical isolation in UHV applications. It is, however, a ceramic and therefore brittle, so machining must be done with great care.

The sides of the molybdenum holders must also be machined off (see figure 2 and schematic B) to accommodate the Macor cap. Both the molybdenum machining and the Macor machining must be done to very close tolerances (on the order of a few thousandths of an inch), so the Macor is in good thermal contact with the molybdenum (heating of the sample is done from below the sample holder) but no undue stress is applied to the Macor cap when it is fastened to the molybdenum. In fact, it is preferable if the tolerances are close enough so that only one screw needs to be used to fasten the Macor to the molybdenum -- there is a much smaller chance of cracking the Macor this way.

Electrical power is provided to the sample by means of tantalum clips which curl onto the top of the Macor cap and then run down vertical grooves on the sides as shown in the figure.

Tantalum was chosen because of the availability and ductility of the sheet material (we cut our strips from the tantalum cover pieces provided with the P-E sample holders), and also because the tantalum photoemission signal does not significantly overlap the aluminum photoemission signal (which is the signal to be measured).

Figure 2. Molybdenum sample holder from P-E, machined to accomodate Macor cap.

The Macor cap is also fitted with 0-80 screws on top which support a chromel/alumel (type K) thermocouple junction. Since we know that the temperature of the sample will rise during current passage, and since the Macor cap is not a good thermal conductor, it is important to bring the thermocouple to the top of the sample, as close to the aluminum stripe as possible. The commercial heating stage, which is used to support and heat the sample, originally had the thermocouple in the base of the stage, quite far from the sample.

Since there is only a pressure contact between the thermocouple and the surface of the sample, the thermocouple wire used on the sample holder should be much thicker than the wire used on the stage. We found that a junction made out of 0.015 inch diameter wire is sufficiently stiff to provide the necessary contact pressure. The thermocouple should be placed near, but not on, the aluminum stripe.

The heating stage, also, is fitted with a Macor collar to support and electrically isolate the power and thermocouple leads, as shown in figure 3 and schematic C. Power is supplied to the

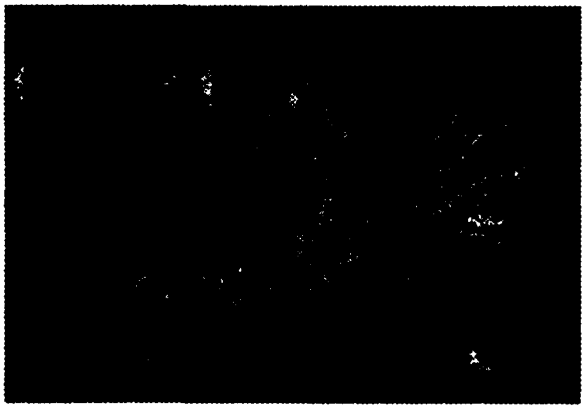


Figure 3a. Macor collar for heating stage, fitted with power clips and thermocouple connections.

Figure 3b. Macor collar with sample holder in place, showing thermocouple and power connections.

sample holder by means of a second set of tantalum clips, aligned to meet the tantalum power clips on the macor cap of the sample holder.

Two 0-80 screws wrapped with the chromel or alumel thermocouple wire provide thermocouple connection on the collar. The thicker thermocouple wires on the sample holder are bent over the sides of the holder to push against the thermocouple wrapped screws on the collar. The thermocouple connection from the stage to the Macor collar requires that the thermocouple junction be removed from the 'as shipped' position behind the set screw in the base of the stage. The junction must be separated, the alumel wire (from the pin closest to the front edge of the stage as it sits in the chamber) wound around the alumel wrapped screw on the collar, and the chromel wire (from the pin closest to the back edge of the stage) wound around the chromel wrapped screw on the collar. It is important to note that once the stage has been modified in this manner, the thermocouple on the sample holder completes the thermocouple circuit. *The heating stage circuitry, then, will not function properly without the modified sample holder in place.* To test the heating stage empty or to use the heating stage with unmodified sample holders, the thermocouple wires from the stage must be removed from the Macor collar and returned to the original configuration.

Both the power and the thermocouple connections from the heating stage to the sample holder are pressure connections only, to accommodate the make-or-break requirement of sample introduction. This means that alignment of the stage and the sample holder is crucial. With this alignment in mind, the horizontal grooves on the Macor cap are cut only part way around the cap. With the groove cut only part way around the cap, the sample holder is automatically oriented correctly when it is placed on the brass holding fork of the P-E sample introduction system. The only way that the sample holder will fit on the fork is in the precise orientation to align the power and thermocouple leads.

For unmodified sample holders, the P-E introduction system is designed so that the brass fork slips into the lower groove of the sample holder. For the modified sample holders the fork must slip into the upper groove. In order to keep the sample holder roughly centered vertically in the sample introduction tube (this is necessary in order to get the sample holder through the gate valve) the brass fork must be unscrewed from the supporting rod,

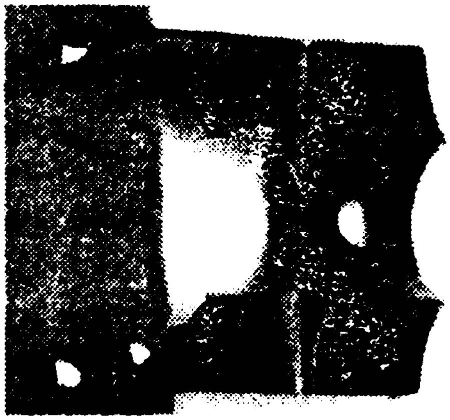


Figure 4. Brass fork from sample introduction system, modified to slip into upper groove of sample holder.

placed on top of the rod (rotating 180^{o} around the axis of the rod), and bolted. This way the fork sits on top of the rod rather than below. To accommodate this switch a groove must be cut in the brass fork from P-E (see figure 4 and schematic D).

The chamber is also fitted with two copper UHV current feedthroughs. Multistrand copper wire insulated with teflon tubing carries the power from the feedthrough to the heating stage while still allowing the full range of motion of the stage.

SAMPLE DESIGN

The requirements for sample design are, in many ways, even more stringent than the restrictions on chamber modifications. The directing goal for sample design, of course, is to get as much signal for measurement as possible. This puts restrictions on geometries, dimensions, and on the materials used on and around the test sample.

We chose to study aluminum lines on silicon dioxide, both because it is an industrial standard and also to permit comparison of results to earlier work done on similar samples here at Rome Laboratories. Even this basic choice had to be modified somewhat to accommodate some integral characteristics of the ESCA system, as shall be discussed later.

The inherent spot size of the ESCA system (i.e. that area which is bombarded with photons) is quite large, roughly 8 mm in diameter. This means that electrons are collected from every material within that 8 mm circle and some from further out, if one takes into account any slight x-ray source misalignment or other scattered photons. Since the cap, which is made out of Macor, has a large component of aluminum dioxide, the silicon wafer base of the sample was chosen to be as large as possible. The sample base, then, acts as a mask, so that the photoemission signal from the aluminum stripe is not contaminated with the photoemission signal from the aluminum in the aluminum dioxide of the Macor.

The shape of the sample base (square with one corner clipped off) was chosen for ease of manufacture. (Cutting circular sample bases out of single crystal silicon wafers, while theoretically possible, would not only be exorbitantly expensive but also a production nightmare.) The corner is clipped off so the sample base doesn't interfere with the thermocouple screws.

The size and shape of the aluminum stripe itself underwent many redesigning stages as we learned more about what would work and what would not work under bench-top trials and electron microscope examination. One important parameter to work around was the current density carried by the stripe. To keep our work comparable to other electromigration studies, we required that our samples be subjected to up to 5×10^{6} A/cm^{2} and chose a stripe thickness of

1000Å -- a typical thickness for thin lines in real applications. The silicon wafer base was also to be covered with a thin (350 Å) layer of silicon dioxide, on which the stripe would be grown.

The first sample design was a single wide (5 mm) aluminum stripe from one side of the silicon base to the other. The width was chosen as 5 mm rather than the 8 mm of the spot size in order to reduce the total current required. At 5 x 10^6 A/cm^2, an 8 mm wide stripe would require 30 A, whereas a 5 mm wide stripe would only require 19 A. Initially the restriction to 5 mm was arbitrary but there are other advantages. The reduction in total current also eased the requirements on cooling (to compensate for joule heating) and on the size or number of power leads to the sample. The 5 mm wide stripe also made it easier to accommodate the thermocouple on top of the sample base without interfering with the stripe but is wide enough so the majority of the x-ray beam is hitting the sample. A single wide stripe is also the easiest to manufacture.

Preliminary bench-top (in air, not UHV) studies were performed on samples cut from one inch diameter silicon wafers layered with aluminum. The samples were cut to be on the order of 5 mm wide and 15 mm long.

These preliminary experiments were motivated by a need for a number of approximate experimental parameters for further design development. For one, we needed to determine the amount of time necessary to observe void formation at various current densities and average times to failure. We also needed to know how much the sample temperature would increase due to joule heating under normal conditions. A more detailed description of the preliminary experiments is given in Appendix 1, only the overall results will be discussed here.

The samples were run under constant-current conditions, with different samples carrying from 0.25 A to 2.25 A. Of course, the higher the current density the hotter the sample became. The temperature of one sample, run at a current density on the order of 10^6 A/cm^2 went from ambient (25^{o}C) to 255^{o}C in roughly two minutes. Even for a sample exposed to only 0.25 A, it became immediately clear that at-air experiments would not be feasible without some cooling mechanism. Further samples were placed on silicon boride disks which acted as heat sinks. A fan was also used during many of the tests to help cool the sample. It is clear that even moderate temperature (30^o - 80^o C) electromigrations performed in the ESCA chamber will require the use of the cooling stage.

Experiments also showed that for our preliminary sample design, virtually all of the observed electromigration voids (and indeed those which caused sample failure) occurred at the power contact point. If the contact is a single clip, and the sample is the same thickness from contact to contact, the current density at the clip is by far greater than the current density along the center of the sample. However, in an ESCA experiment it is primarily the center of the sample (and obviously not *under* the contact clips) which is being observed. It is important, therefore, to modify the sample design so that the current density is lowest, not greatest, at the contact points.

This is done by changing the shape of the sample in two ways. First, we included a thicker aluminum contact pad at each end of the sample. From an experimental point of view, this contact area should be as thick as possible (even up to a few hundred microns). However, there are sample manufacturing considerations. The intended sample manufacturer suggested that the pads be no more than one micron thicker than the rest of the aluminum stripe.

The second modification was to shift from a single wide straight stripe to a much thinner (50 μ wide) stripe which meandered back and forth across the sample area (see schematic E). The outer dimensions of the stripe remained the same. The dotted diagonal lines on the schematic indicate the corners that must be cut off so the sample base does not interfere with the thermocouple screws.

There are both additional advantages and disadvantages to the modified sample shape. First, by reducing the stripe width from 5 mm to 50 μ, the cross-sectional area of the stripe is reduced by a factor of 100. The current then needed to realize the chosen current density is also reduced by a factor of 100. So instead of needing to having power cables in the chamber capable of carrying 19 A, we now need cables to carry only a few hundred milliamperes. As far as the demands on the sample holder and stage go, this modification to the sample design makes getting the necessary current to the sample considerably easier.

There is, however, a disadvantage. To the extent that there is bare silicon dioxide separating each lap of the meander, the actual aluminum sample area is reduced. Less aluminum sample area means less photoemission signal from the area of interest and a lower signal to noise ratio. Again, because of manufacturing limitations, the distance between each lap of the meander can be no less than 1 μ so than the 'lost' sample area is determined by the ratio of the meander stripe width to the distance between laps. For example, if the stripe is 50 μ wide and the distance between each lap is 5 μ, then 10% of the sample area is lost.

It is important to remember that maximizing the signal is crucial. We are not interested in the ESCA spectra of plain aluminum; we are interested in shifts that occur in that spectra with electromigration. It is reasonable to expect those shifts to occur because of changes in chemical states *near voids and at grain boundaries.* Our experiments showed that even if the current were spread out at the contact points (either by using multiple contact clips or by inserting an aluminum foil contact pad under the clip) there was still very little void formation in the central part of the sample, away from the power contacts. The fraction of the aluminum which fits the criteria of "near voids and at grain boundaries" will be extremely small. It is ironic that in order to study electromigration by ESCA we must maximize void formation, though the point of studying electromigration in the first place is to minimize or eliminate void formation.

The final modification to the sample design is a step toward getting as much signal as possible from the areas of greatest interest -- at voids and along grain boundaries. Instead of making the samples out of aluminum alone, the samples are to be made out of aluminum doped with palladium (5%). There are several advantages to adding a dopant, and to adding palladium specifically. First, the palladium will tend to diffuse out of the grains to the grain boundaries. By focussing not on the aluminum ESCA signal but rather on the palladium signal we are maximizing the fraction of the signal *from the region of interest* -- the voids and grain boundaries. The primary palladium ESCA lines (337 eV, 342 eV, and 534 eV) are far enough away from the aluminum (73 eV and 75 eV), silicon (103 eV and 153 eV), and oxygen (531 eV) lines to prevent any significant overlap. Furthermore, there is currently considerable industrial interest in using palladium as an aluminum dopant.

Although some refinement is advisable (see Appendix 2), these designs and results provide an experimental method and apparatus for ESCA studies of electromigration within the restrictions of ultra-high vaccuum, convenient sample introduction, and the beam characteristics of the Perkin-Elmer ESCA system.

The authors wish to acknowledge the support of the Reliability Physics Branch, Microelectronics Reliability Division of Rome Laboratory, Rome, New York. We would like specifically to express our appreciation to Dr. Lois Walsh and Dr. George Ramseyer for many helpful conversations.

Appendix 1: Bench-top Highlights

Aluminum stripe #6, run #1
dimensions: 700 Å x 4.5 mm x 15.5 mm
current: 1.00 A

Sample was run for one hour at constant current and the fan on high. The experiment showed that the previous failure of contact points (i.e. connections) were due to thermal effects at the failure point rather than the current density. There was no visible (SEM) electromigration on the sample, only an increase in voltage to maintain constant current.

Aluminum wedge (sample wafer edge), run #1
dimensions: 700Å x 22 mm x 7.5 mm at widest point
current: 1.00 A

The sample was run at one amp without a fan to determine the temperature rise that can be expected in the chamber if the cooling stage is not used. The sample ran for 25 minutes with a temperature increase (from ambient) of 45°C. After failure, the contact clips were moved and the current restarted. (See run #2.)

Aluminum wedge, run #2

The current was increased to 2.24 A but the contacts immediately failed at a voltage of 11.36 V and a surface temperature of 81°C.

Aluminum stripe #7, run #1
dimensions: 700Å x 5.0 mm x 18.0 mm
current: 1.50 A, 1.25 A

New clips were added for this run. Current was run with the fan on high to see if voiding would be visible at a higher current density. After the contact points failed twice at 1.5 A the current was reduced to 1.25 A. The sample ran at 1.25 A for roughly 30 minutes with a temperature increase of 26°C. The circuit failed at the left clip, yet there was no (other) electromigration voids visible under the SEM.

Aluminum stripe #8, runs #1, #2, and #3
dimensions: 700Å x 4.5 mm x 17.0 mm
current: 1.00 A

Two contact clips were added to lower the current densities and thermal gradients at the contact points, with the hope that the sample could be run longer and voids might be observable under the SEM.

The sample was run for roughly 18 hours and 45 minutes. SEM observation shows contact failure at both right clips, but no noticeable voiding outside of the holes under the contact clips. There seemed to be some faint shadows of something on the surface, but resolution was insufficient to see them on the photo. Current density was estimated to be 3×10^5 A/cm^2 in the stripe and 6×10^6 A/cm^2 under the clips.

Aluminum stripe #9, run #1
dimensions: 700Å x 4.5 mm x 12 mm

In an attempt to increase contact surface at the power input, indium had been placed under the clips. When the power supply was turned on the sample connection immediately failed. Under SEM it looked as if the indium had melted and the aluminum had boiled off on one side.

Appendix 2 Further Suggestions for Continuing ESCA Analysis

There are a number of tests which should be performed prior to electromigrating samples in the ESCA chamber. In many cases the experimental results may well suggest further refinements for the experimental design -- or indeed for sample design. In each case (unless otherwise noted) the temperature specified is the temperature at which the electromigration is done. All data should be taken at room temperature, to eliminate any Debye temperature effects from the data.

A. Use Auger analysis to refine the aluminum cleaning recipe. First pass recipe: sputter with Ar^+ at an ion current between 1μA and 2 μA and at 300 eV ion energy for 10 minutes. Initial sputtering should be done with the sample at room temperature. It may be necessary in later cycles to sputter with the aluminum slightly warm (200°C - 30 minutes) or to include an anneal (300°C -15 minutes) in the cycle. However, since part of the intent of the experiment is to separate thermal effects from electromigration effects it would be best (if at all possible) to not heat the sample prior to the electromigration. Nonetheless, it is obviously important to start with an exceptionally clean surface.

Since the entire sample should be cleaned evenly, it is important to focus the ion gun as tightly as possible and raster the beam as widely as possible. If the gun is not focussed, even rastering the beam will lead to an uneven or pitted surface after bombardment.

B. Use the profilometer to double check the aluminum stripe step height before and after the cleaning process (or, more easily, compare sputtered and unsputtered areas). Excessive or uneven cleaning will lead to uneven current densities in the sample. Several samples should be tested with the profilometer to check the reproducibility of the cleaning process. Recall that the intent of the cleaning is to just remove the native oxide layer -- not to sputter the aluminum line.

C. Put "blank" samples (i.e. with SiO_2 cover but no aluminum) in the ESCA to determine the signal contribution from the Alumel probe. This is a good idea but not quite as crucial if the aluminum is Pd doped.

D. Determine *in the ESCA chamber* the maximum temperature of the sample with the highest intended current density through the sample (5×10^6 A/cm^2). Does the heater current drop to compensate I^2R heating (i.e. maintain roughly constant temperature)? Is there a danger of melting the aluminum (melting point 660°C)? If so, at what current density?

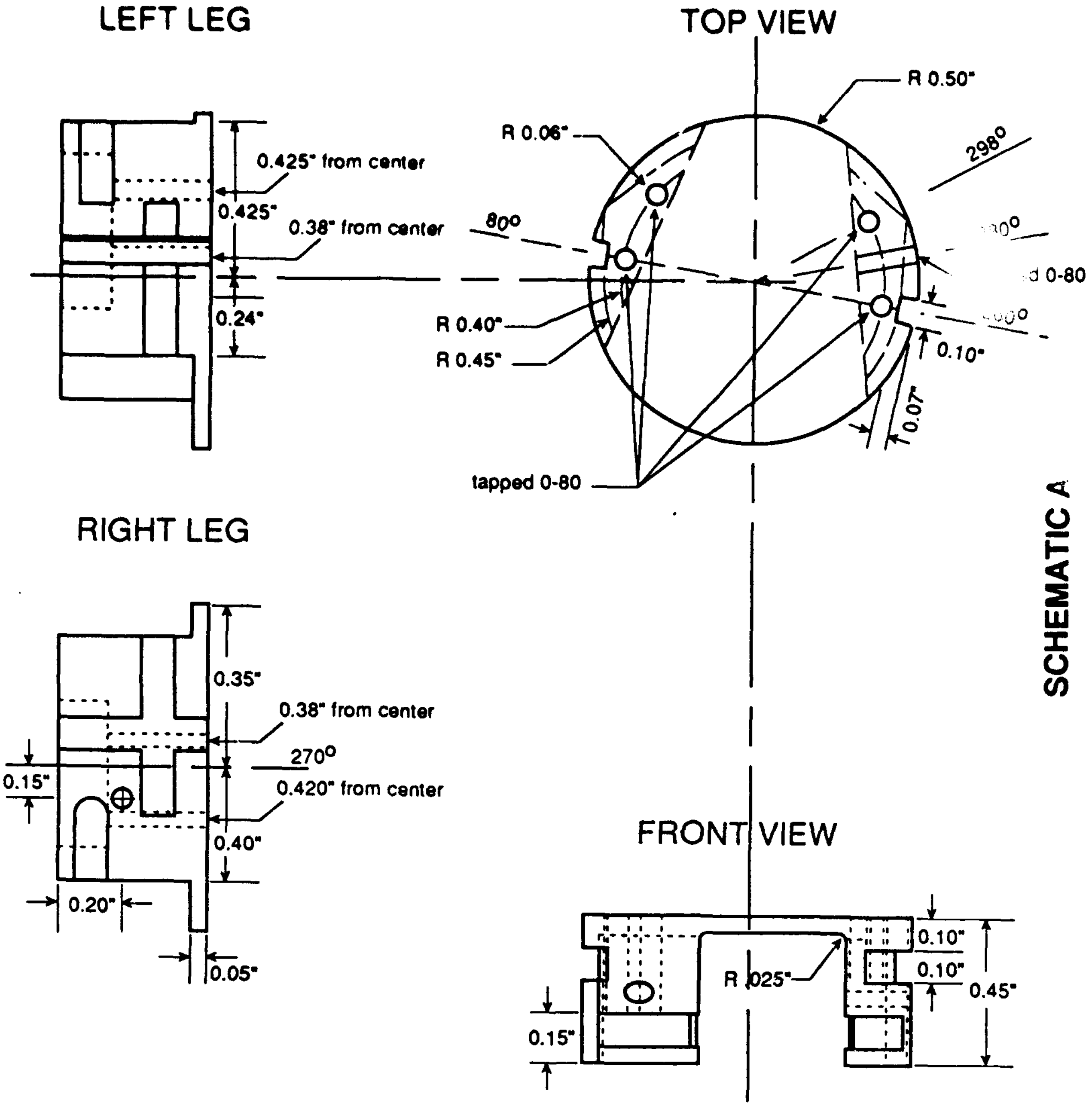
E. Determine changes in the photoemission spectra with increasing temperature (room temp to 600°C) without the electromigration current. This is particularly important with Pd doped samples as some of the palladium may diffuse out of the bulk to the surface with heating. Two experiments should be done:

1) Subject the sample to the same heating cycle as will be the norm for main experiments (i.e. maintain temperature for as long as the electromigration current would be applied) and cool the sample to room temp to take the spectra. Take data for heating cycles to 300°C, 350°C, 400°C, ..., 600°C.

2) Heat samples to the same temperature as in 1) but do photoemission with the sample hot.

F. Use Auger to determine the extent of impurity migration on samples which have been cleaned and heated but not electromigrated. The amount of time for heating (and/or electromigration cycles) for both E. and F. will be determined by G.

G. Use Auger or SEM to determine how long it takes to have obvious electromigration at the upper and lower limits of the intended experimental range (i.e. at 7×10^4 A/cm^2 at room temperature and at 5×10^6 A/cm^2 at 600°C). Check at 30 minutes, 1 hour, 3 hours, 6 hours, etc. as dictated by results.



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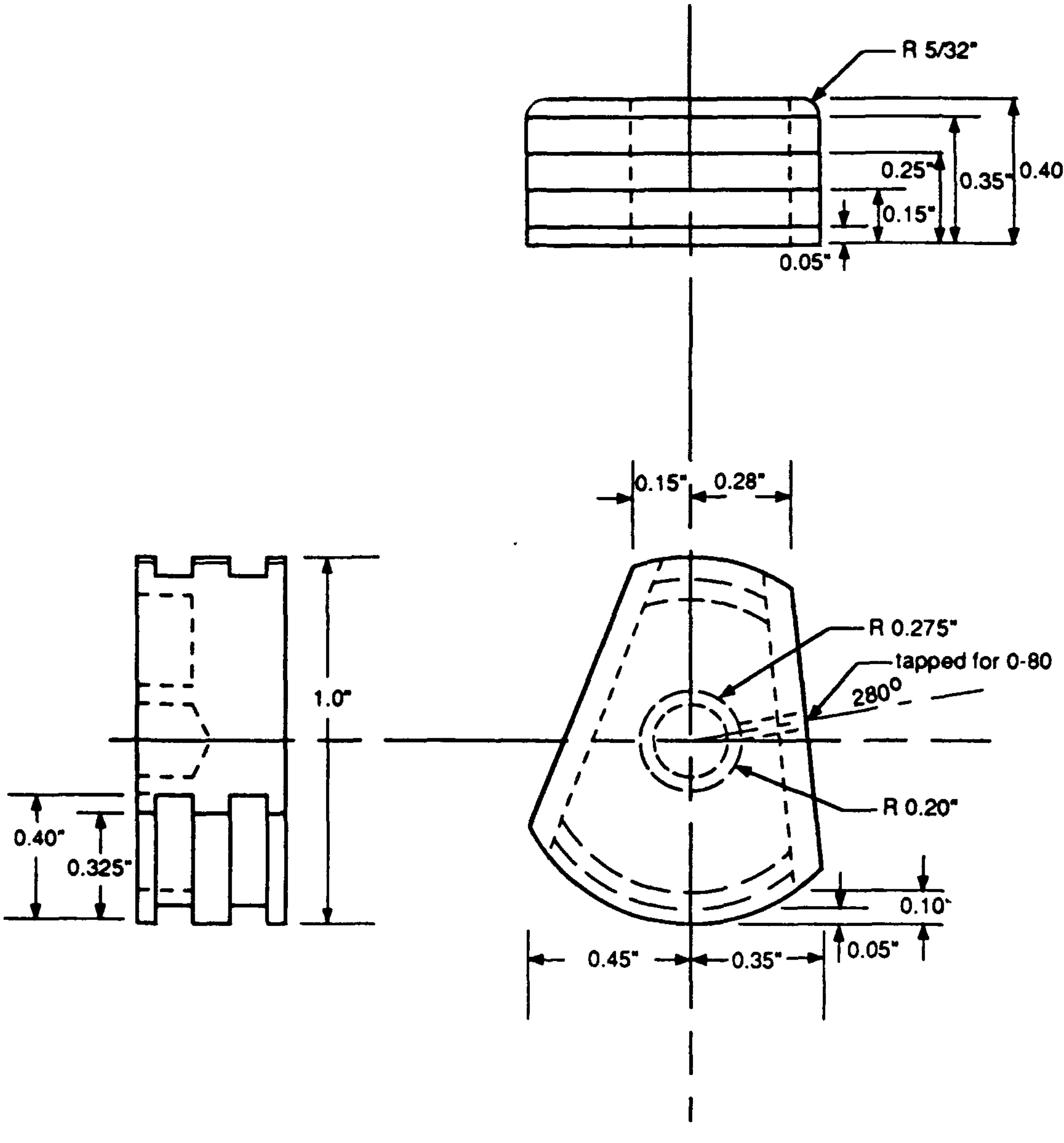
MACOR SAMPLE HOLDER
PETER GRACH



SCHEMATIC B

SCALE: 1" = 1/2"
MACOR SAMPLE HOLDER
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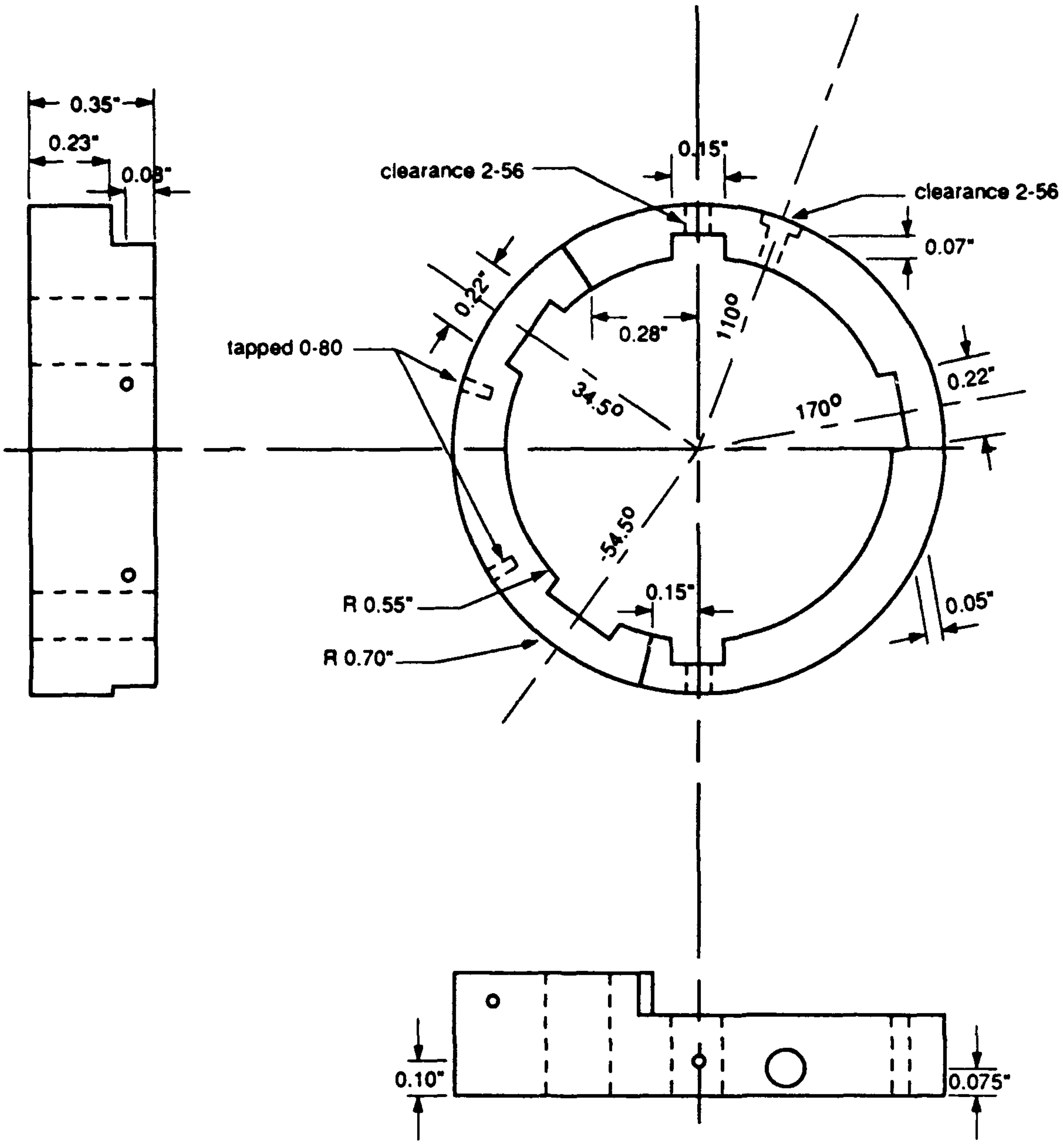
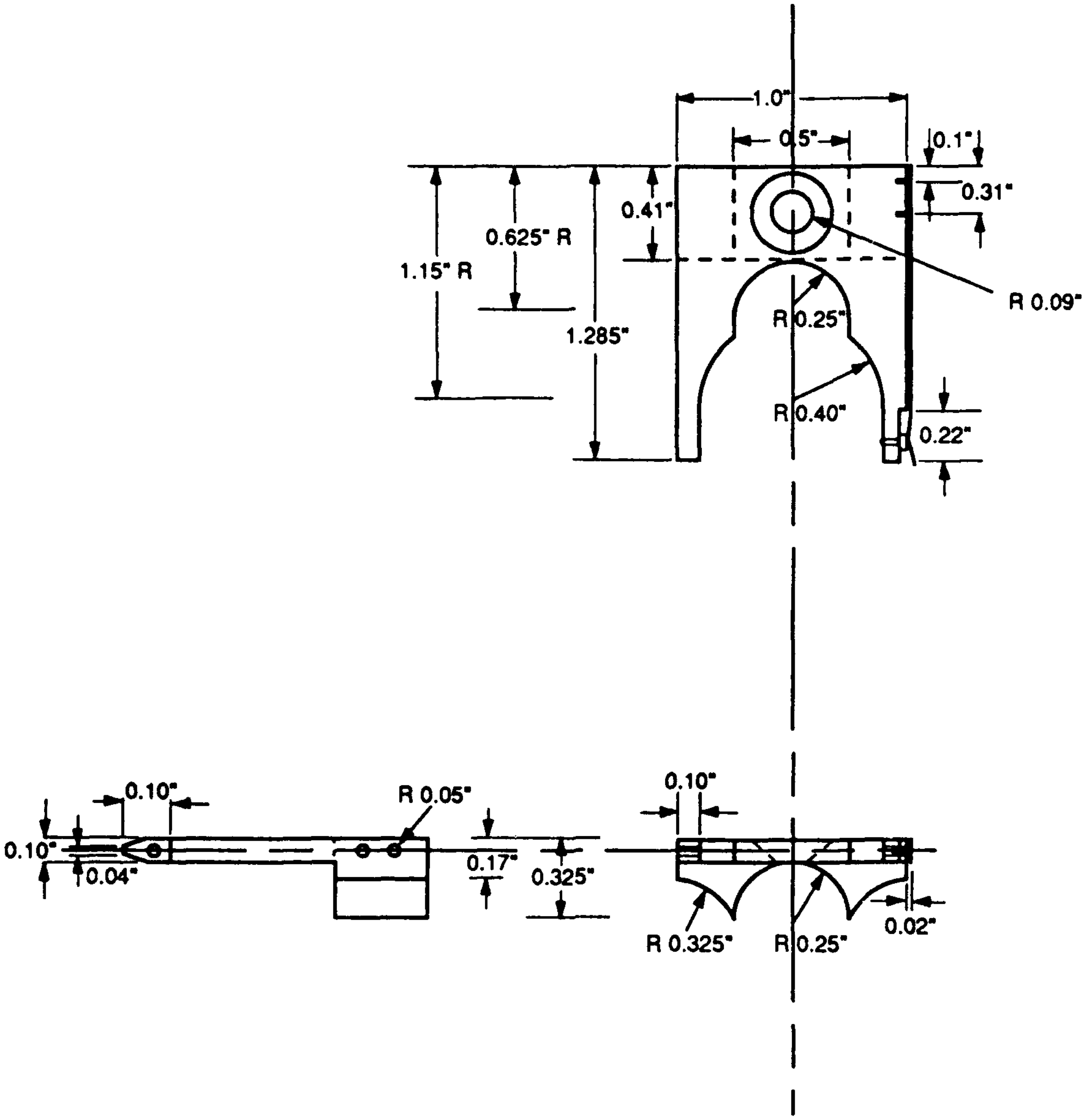


SCHEMATIC C

SCALE: 1" = 1/2"
MACOR STAGE PIECE



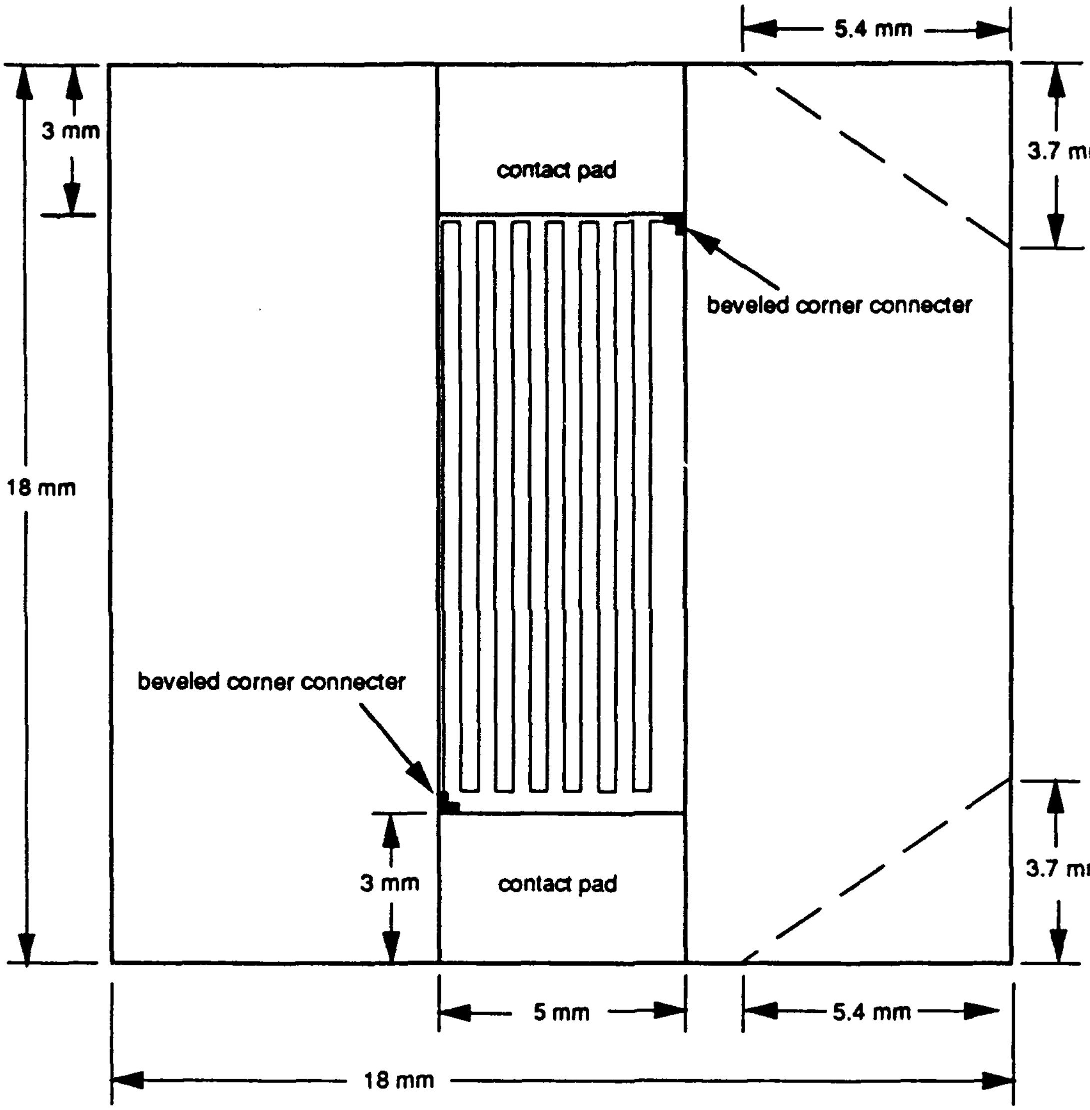
SCHEMATIC D

SCALE: 1/5" = 1/4"
BRASS FORK FOR
SAMPLE CHAMBER
PETER GRACH

Aluminum (with 5% Pd) meander stripe
on 350 Å SiO_2 on Si wafer

1 meander stripe;
98 laps of 50 µ wide across width
with 5 µ separation between each lap

stripe 1000 Å thick
contact pads 2 µ thick



SCHEMATIC E

MISSION
OF
ROME LABORATORY

Mission. The mission of Rome Laboratory is to advance the science and technologies of command, control, communications and intelligence and to transition them into systems to meet customer needs. To achieve this, Rome Lab:

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b. Transitions technology to current and future systems to improve operational capability, readiness, and supportability;

c. Provides a full range of technical support to Air Force Materiel Command product centers and other Air Force organizations;

d. Promotes transfer of technology to the private sector;

e. Maintains leading edge technological expertise in the areas of surveillance, communications, command and control, intelligence, reliability science, electro-magnetic technology, photonics, signal processing, and computational science.

The thrust areas of technical competence include: Surveillance, Communications, Command and Control, Intelligence, Signal Processing, Computer Science and Technology, Electromagnetic Technology, Photonics and Reliability Sciences.