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~~INTERIM TECHNICAL REPORT~~

~~on the~~

⑥ RESEARCH, DEVELOPMENT, AND FABRICATION
OF TUNNEL EMISSION CATHODES

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⑨ ~~THIRD~~ QUARTERLY REPORT *no 3.*

Covering the period January through March, 1963

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HARRY DIAMOND LABORATORIES
WASHINGTON, D. C.

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ABSTRACT

Research was continued on tunnel emission cathodes with ^{microscope} ~~microscope~~ ^{analyzer} ~~analyzer~~.
~~During the third quarter,~~ emission current densities up to 740 ~~ma/cm~~ ^{microamp/cm} square cm were obtained. The transmission coefficients (emission current/film current) ranged between ~~1 x 10⁻⁴~~ and 1 x 10⁻⁸.
^{Some times ten to the minus four and ten to the minus eight powers.}

The mechanism by which electrons are transferred from the emitter layer to the accelerator layer is not clear from the data. In some cases, it appears as though tunnel emission occurred, while in other cases it appeared as though thermionic or Schottky emission occurred.

In all cases, the units either shorted or opened after several testing cycles. The failure is probably due to the accumulation of heat at the junction due to the poor heat sink conditions. Several approaches to provide a heat sink are being studied.

Transmission electron microscopy is being used to study the emitter and accelerator films, and X-ray fluorescence is being used to determine film thickness.

Testing has been conducted under d-c and pulse conditions. ↗

MEETINGS AND CONFERENCES

C. E. Horton and J. W. Hall, II made the following trips:

1. General Electric Research Laboratory, Schenectady, New York, January 7-10, 1963. Discussed vacuum systems, thin film techniques, electron microscopy, tunnel emission testing devices, insulation, etc.
2. Electronics Laboratory, Electronics Park, Syracuse, New York, January 11, 1963. Discussed advantages and disadvantages of doing theoretical work on tunnel emission cathodes with P. R. Emtage.

Electrical Test Results

A. Film Current Measurements

The first electrical tests during this quarter involved the use of X-Y recorders to study the I-V characteristics of the Al-Al₂O₃-Au sandwich and the emission current as a function of film current. Four-terminal measurements were made using the d-c circuit shown in Figure 1. Since the exact mechanism of electron transfer from the emitter to the accelerator is not known, the current through the insulator is referred to as the film current.

To test for emission, the sandwiches were encapsulated in metal tubes with an octal stem. Most of the sandwiches gave I-V characteristics, but the emission was negligible for these particular tests. In some cases, a negative resistance region was exhibited in the characteristic as shown in Figure 2. The particular unit represented here was tested eleven times. The voltage was applied manually and the time for a single sweep was 2-3 seconds. The retrace was not recorded. A definite negative resistance region is exhibited in each curve except curve 1.

During each sweep the power rises to at least 0.3 watts. The unit was allowed ten to fifteen minutes to cool between consecutive sweeps so as to lessen the temperature effect on the next sweep. The negative resistance effect increases in each consecutive sweep and the change appears to be irreversible.

It is emphasized that the negative resistance effect, which rarely occurs, is undesirable from the point of view that the I-V characteristic is erratic and the sandwich burns out more rapidly than sandwiches which do not have negative resistance effect in their characteristics. Also, as will be shown in the next section, erratic emission is sometimes associated with the negative resistance effect.

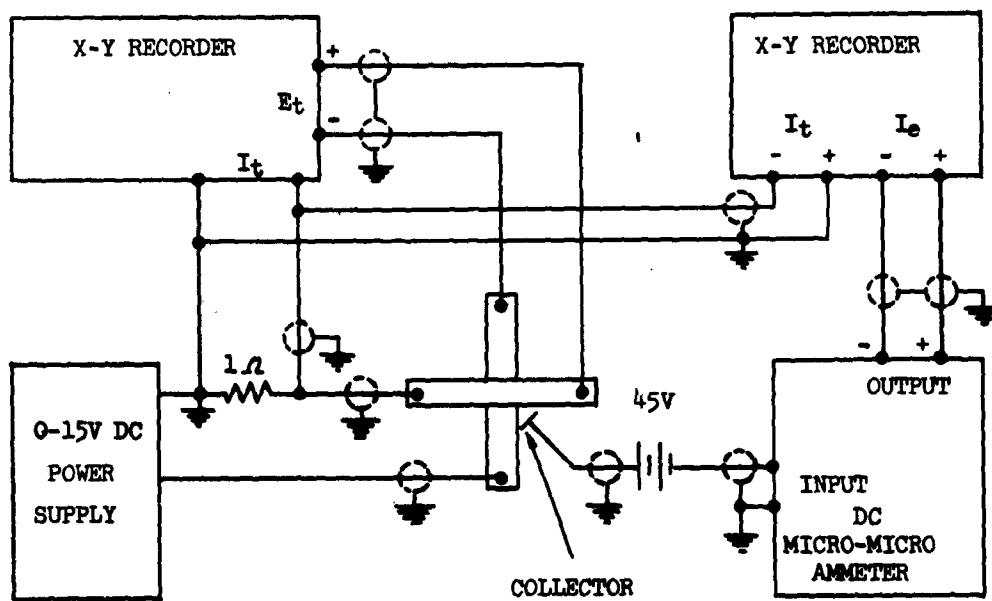
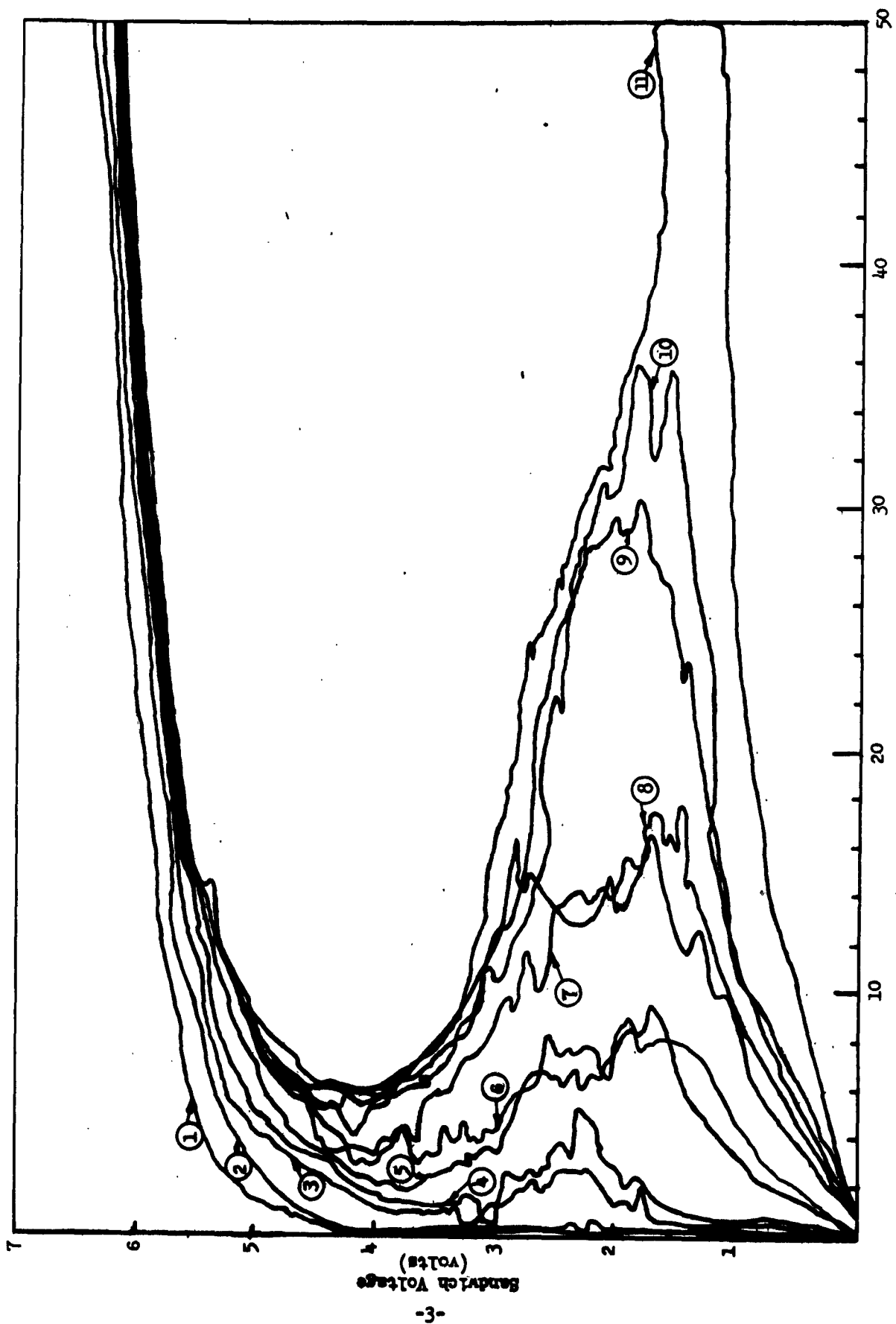


Figure 1. Test Circuit Using X-Y Recorders



Film Current (ma)

Figure 2. Negative Resistance Effect

Several workers in the field have observed the negative effect, but thus far no satisfactory explanation has been given (1,2).

Since the encapsulation of the sandwiches in metal tubes is cumbersome and time-consuming, it was decided to conduct the emission testing in the 400 l/s ion system. This change allowed us to install six units within ten minutes and, since the system is cycled twice a day, twelve units per day can be tested.

In this setup, two adjacent octal instrumentation feedthroughs are used. Gold wires connected to the feedthroughs are indium soldered to the emitter and accelerator films, and a coiled tungsten wire is used as the plate.

Some attempts have been made to flash barium onto the surface of the accelerator to reduce the work function. As a result, shorts have occurred indicating an excessive amount of barium was evaporated.

At the present time we are not too concerned with barium as a means to increase the emission current. It is our feeling that we should first learn to fabricate a sandwich which operates reliably over a long period of time. When this is accomplished, efforts can then be made to increase the emission by the use of barium or other elements, such as thorium.

At the present time we are able to consistently fabricate sandwiches which will withstand 100 ma or more for several minutes under d-c conditions in vacuum. Within any lot, the yield ranges from 80-100%. In some cases the films are damaged by scratches due to handling or by heat due to soldering connections to the films.

B. Emission Testing

During the change from testing in metal tubes to the ion system, it was decided to use a dual beam oscilloscope with a polaroid camera attachment

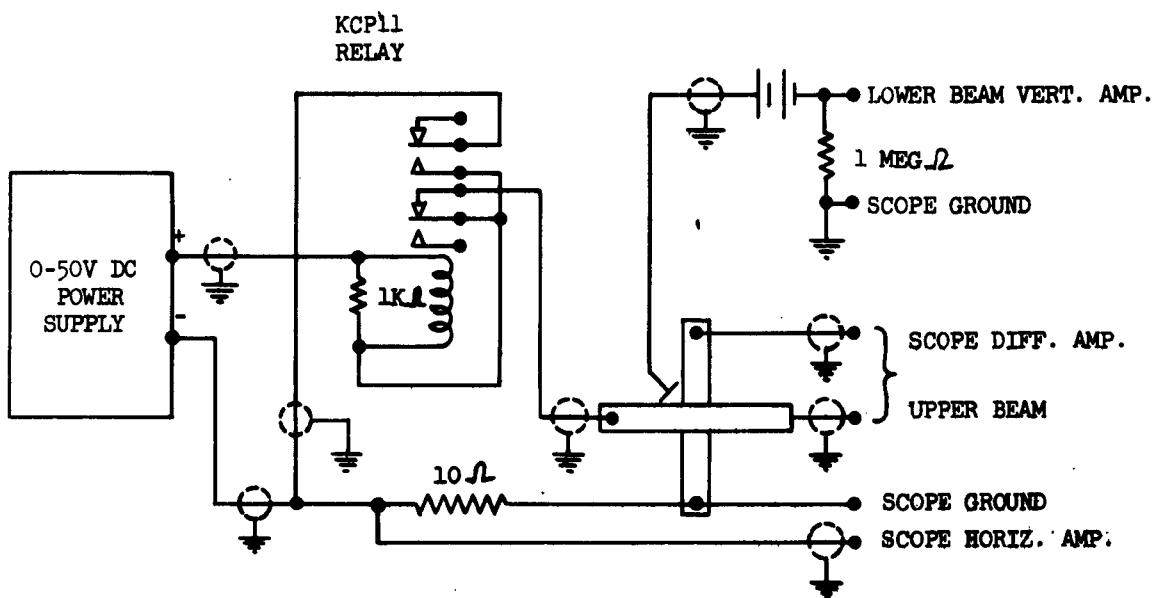


Figure 3. Test Circuit Using Dual Beam Scope

rather than the X-Y recorders. The modified circuit is shown in Figure 3.

It is important that a four-terminal measurement be made in order to obtain accurate values of the voltage. If two or three terminal measurements are made, the voltage cannot be accurately measured since part of the voltage is dropped across the film.

Emission currents up to $7.4 \mu\text{a}$ from $\text{Al-Al}_2\text{O}_3\text{-Au}$ sandwiches have been recorded with a transmission coefficient of 2.5×10^{-4} at 6.4 volts across the sandwich. The junction area is 1 mm^2 .

Care must be taken in calculating the current transmission ratio of a tunnel cathode. The greatest possibility for misinterpretation occurs near the burnout point of the cathode. In performing a series of tests on a tunnel cathode, it is common practice to draw increasingly high currents so that the early tests show clearly the low-current properties and the last test shows the maximum current that can be drawn before the cathode fails. The cathodes often show a high burst of emission current just before burnout, and the ratio of this emission current to the film current might be used to infer a high current transmission ratio. However, this emission is probably thermionic, associated with the high local heating that occurs at burnout. We do not include these transients in the emission currents and current transfer ratios that we report.

From the data collected thus far, there is no indication of a single mechanism by which electrons are transferred from the emitter to the accelerator layer. In some cases, the emission current versus film current is a straight line, while in other cases it is curved.

In Figure 4 is shown the film current versus the sandwich voltage (Curve 1) and the emission current versus the film current (Curve 2). The retrace is shown and does not exactly coincide with the forward trace.

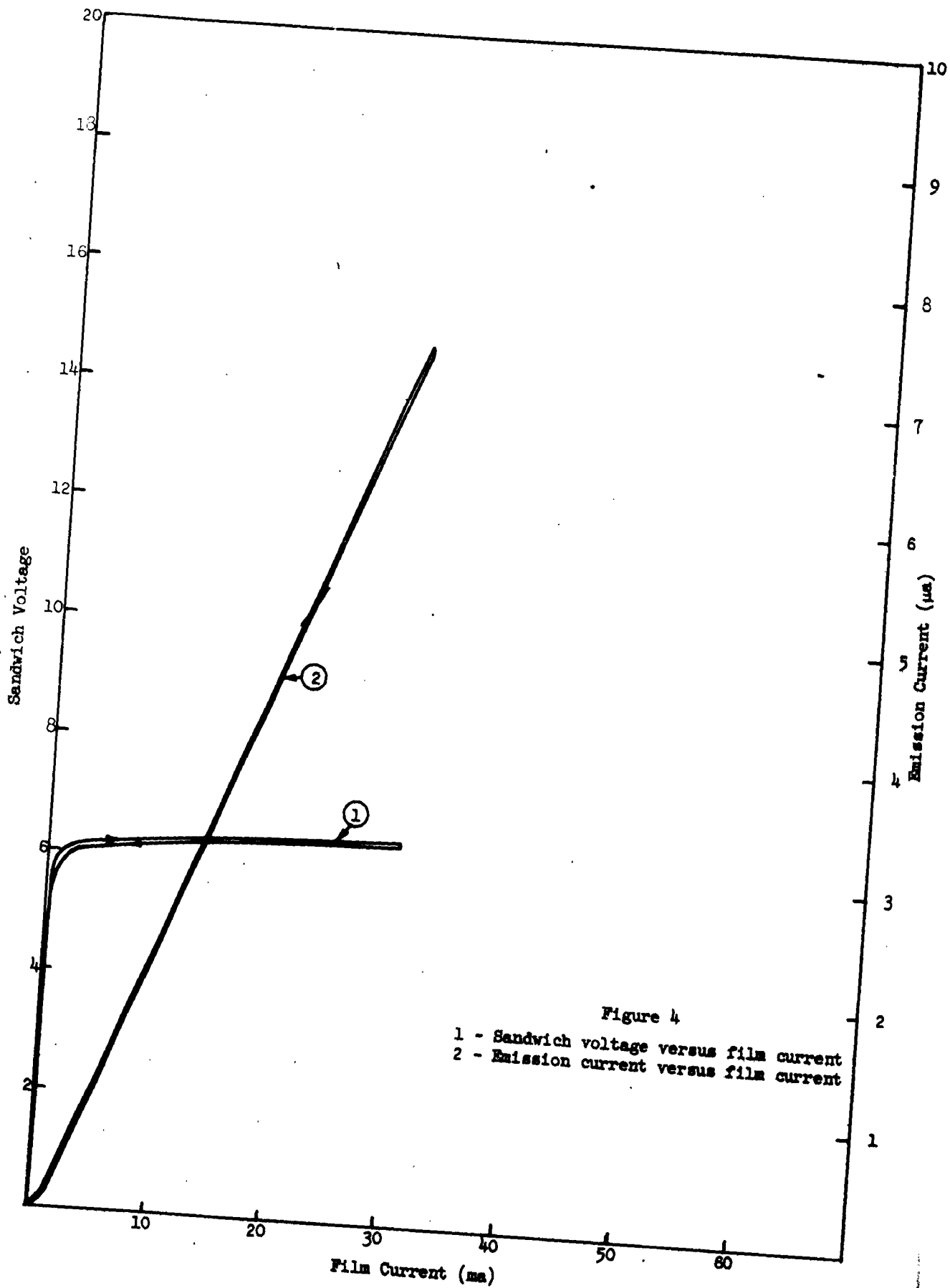


Figure 4
1 - Sandwich voltage versus film current
2 - Emission current versus film current

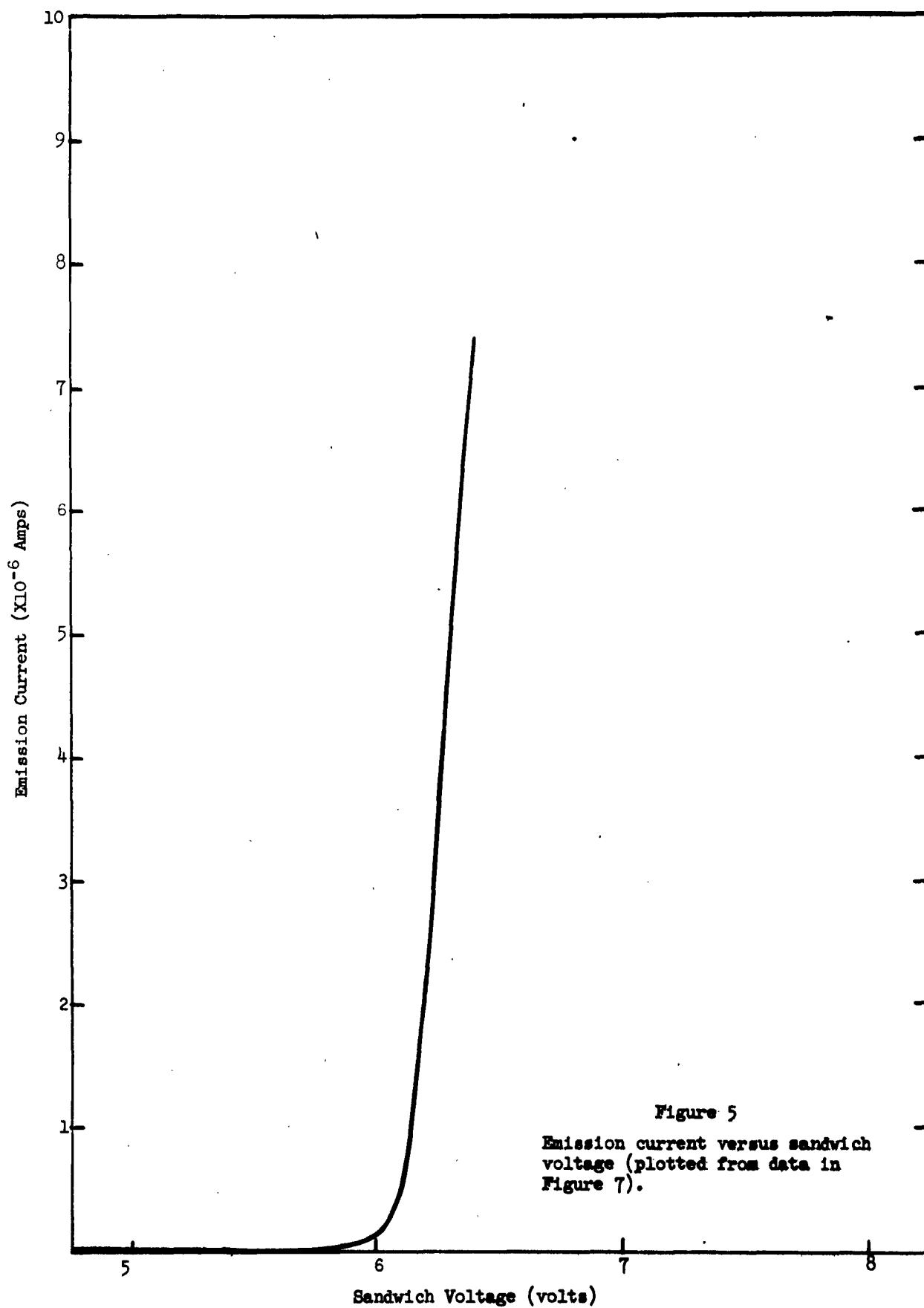


Figure 5
Emission current versus sandwich
voltage (plotted from data in
Figure 7).

It is pointed out that the emission current (I_e) does not become appreciable (1×10^{-9} amps) until the film current (I_f) becomes appreciable ($1/2$ ma) which occurs at about 5.8 volts for this particular unit. The I_e (emission current) versus V_f (film voltage) is shown in Figure 5, which was plotted from the information given in Figure 4. The I_e - I_f curve in Figure 4 has a slight curvature between 0 and $0.1 \mu\text{a}$. Above this point the I_e - I_f curve is linear, which suggests the tunneling mechanism as the means by which the electrons are transferred from the emitter film to the accelerator film. At low voltages, the film current is very low and varies about linearly with voltage.

There are several reasons why emission is not directly proportional to film current for sandwich voltages approaching the work function voltage. The electrons in the film will have an energy distribution. The electrons will also have a momentum distribution; that is, for two electrons of equal magnitude momentum, one may pass from the film to the vacuum and one may be reflected at the boundary because its incident angle is too large. Further, even the work function is subject to a distribution or "patch" effect at the film surface.

In light of the fact that energy, momentum and work function variations are present, it is obvious that until the sandwich voltage is significant (in respect to the work function of the accelerator layer), the emitted electrons will not be proportional to film electrons to the extent that they will be at voltages lower than the work function. That is, higher voltages will "erase" distribution effects found at marginal conditions.

In some cases, the curvature at low emission currents is not exhibited, as shown in Figure 6 by Curve 2, which is the emission current versus the film current. Curve 1 is the sandwich voltage versus the film current. The sandwich was tested five times and five different emission characteristics were recorded

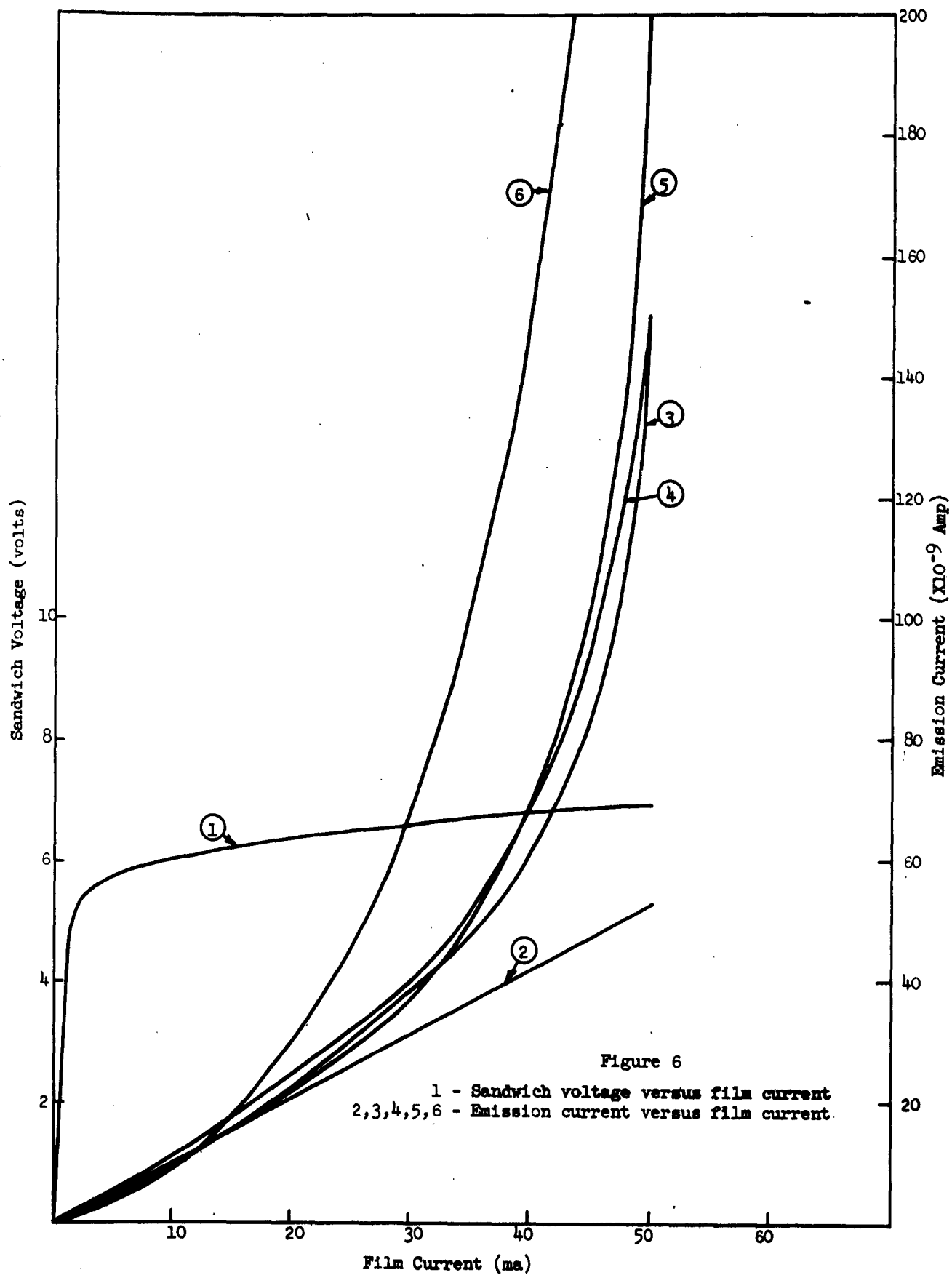


Figure 6
 1 - Sandwich voltage versus film current
 2,3,4,5,6 - Emission current versus film current.

as shown. During each consecutive test the emission current increased with the same applied voltage which was a triangular pulse of 1-2 seconds duration. The retrace was not recorded.

Since a power dissipation of at least 0.3 watts is reached during each sweep, and poor heat sink conditions are present, the sandwich was allowed several minutes to cool between consecutive sweeps. It was thought that the characteristic might revert back to the straight line after the cooling period, but evidently an irreversible change occurred.

It is noted that the film voltage characteristic did not change, which indicates that the irreversible change is occurring in the accelerator film. The gold film is thin ($\sim 300 \text{ \AA}$) and when heated it has a tendency to form globules. This tendency to form globules could also cause pores to be formed. The formation of the globules could cause the gold film to become non-conducting, in which case the circuit would be open as is the usual case after burnout.

In the case of the irreversible change with the negative resistance effect (Figure 2), the change appears to be taking place in the insulator.

The presence of impurity ions in the oxide layer due to exposure to the atmosphere and liquid anodization could also be a possible cause of instability. Smith and Miles (3) have shown that the formation of an oxide film by gaseous anodization using a glow discharge yields oxide films which will withstand film currents up to 1000 A/cm^2 .

Formation of the oxide layer by gaseous anodization markedly reduces the ion impurity density and also could cause the structure to be different from that formed by liquid anodization or atmospheric oxidation. In other words, the presence of impurity ions could affect the manner in which the oxide is formed. In both cases, the irreversible changes are probably associated with a structural

change, such as crystallization, pore formation, cracking, or the formation of globules of the accelerator film which result due to the heating effect. An increase in temperature can also increase the rates of diffusion of the atoms between the films.

In discussing the negative resistance effect shown in Figure 2, it was stated that the emission from a sandwich in which the negative resistance effect is present is unstable and accompanied by high noise. This is exemplified in Figure 7 by Curve 2 which is the emission versus film current. The film current-voltage characteristic (Curve 1) is shaped differently from that shown in Figure 2, but the negative resistance effect is pronounced in both cases. The vertical lines in Curve 2 indicate the noise level. Usually there is very little if any noise in either of the characteristics in the absence of the negative resistance effect.

We seldom see the negative resistance effect, and less frequently are able to obtain emission from the sandwiches exhibiting the effect. For lack of data, very little can be said except that burnout occurs very rapidly (ten of seconds) at moderate film currents (50-100 ma).

Films

X-ray fluorescence is being used to determine the thickness of the gold accelerator films. The unit used is a General Electric XRD5 with a Hammer #N305 amplifier. Included are a platinum target X-ray tube, an LF crystal set at L_{Al} line (36.96°) with an X-ray plate power input of 50 KVP at 20 ma.

Both sample and glass background are counted for 1000 seconds. Thirty-five micrograms of gold (as gold chloride) on a glass slide gave 80.1 counts per second. The same glass slide without gold gave 24 counts per second. This 56.1 counts per second is equivalent to 35 micrograms of gold, and 0.625

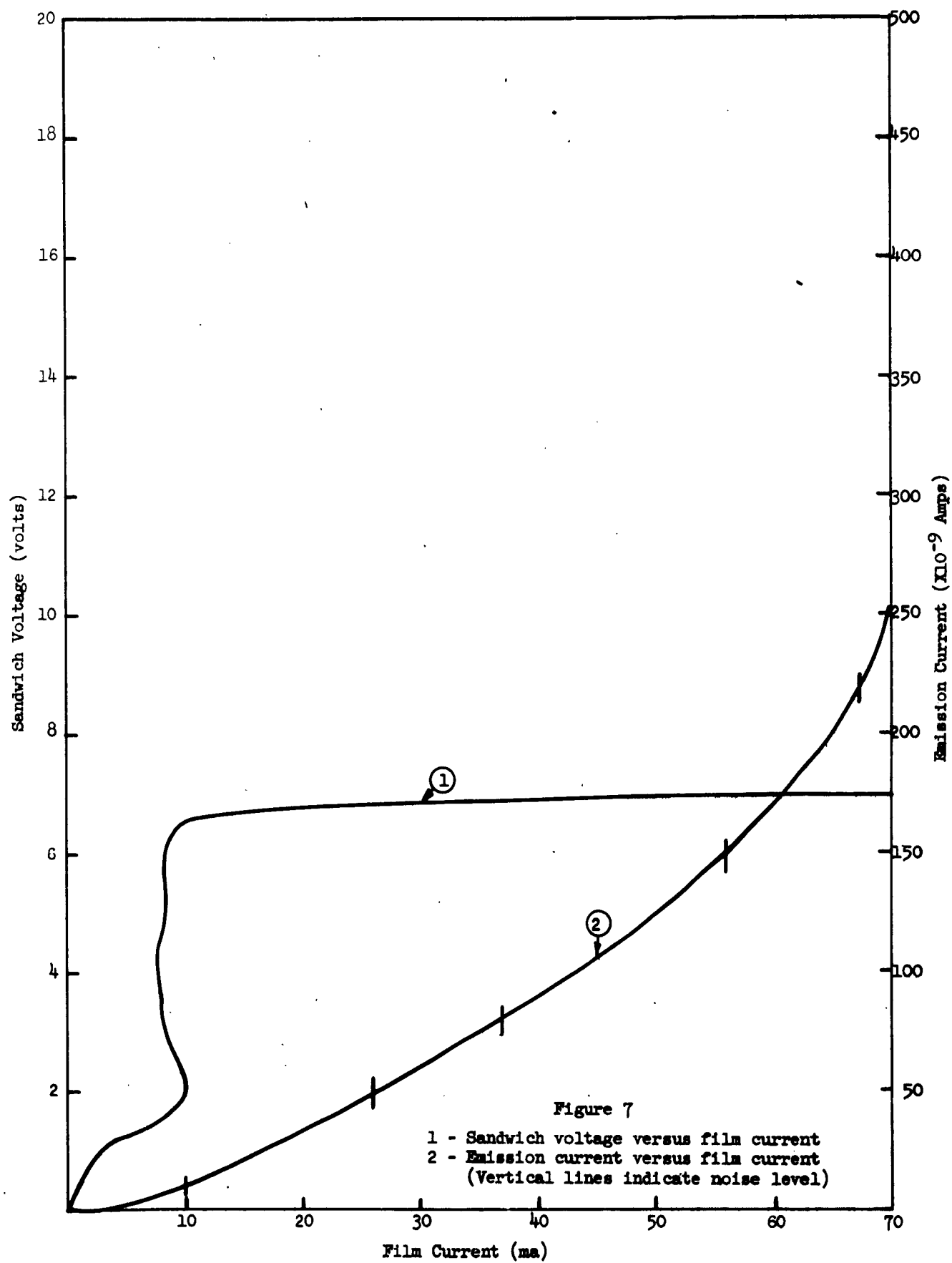


Figure 7

- 1 - Sandwich voltage versus film current
 - 2 - Emission current versus film current
- (Vertical lines indicate noise level)

Film Current (ma)

micrograms of gold is equal to 1 count per second. Since the accuracy of the counting process is approximately 0.5 counts per second, the error in measuring this amount of gold is $\pm 0.9\%$.

To study the film surface in detail, an RCA EML-1B electron microscope is used. To study the surface structure of various substrates, the indirect carbon replica method is employed. This method is outlined in Appendix I. A modified method of replicating the surfaces of evaporated thin films had to be used because the final carbon film exhibited an increasing tendency to disintegrate into minute pieces when placed in the acetone bath. The modified method is given in Appendix II. Sometimes the film adheres to the replicating surface, in which case one sees a combination of a surface replica and a transmission microphotograph.

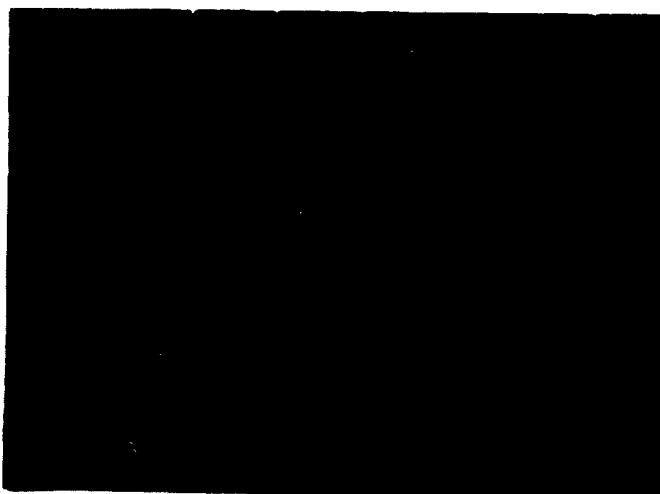
For gold films that have been evaporated on a glass slide at room temperature, it has been found that conduction begins at an average thickness of 50-70 $\text{\AA}$. For gold accelerator films evaporated at room temperature on glass slides, this value is a lower limit of the thickness of the accelerator. A transmission microphotograph of a gold film with an average thickness of 40-50 $\text{\AA}$ is shown in Figure 8. The island structure (dark portions are gold) is clearly indicated. The larger structures shown are either defects in the glass substrate surface or contamination.

In Figure 9 is shown a transmission photomicrograph of a gold film of an average thickness $\sim 700 \text{\AA}$. The photograph indicates that the gold film is not completely continuous at this thickness by the presence of pin holes (light areas). Thus far we have not been able to obtain emission from sandwiches with an average accelerator gold film thickness greater than $\sim 500 \text{\AA}$. Indications are that the gold films from which emission has been obtained had a high pin-hole density.



1 μm

Figure 8. Non-conducting Gold Film 40-50 Å
Average Thickness (x 27,000)



1 μm

Figure 9. Conducting Gold Film ~ 700 Å
(x 16,000)

The effect of pinholes on emission will be studied and an effort will be made to determine what correlation, if any, exists between the emission current and pinhole density.

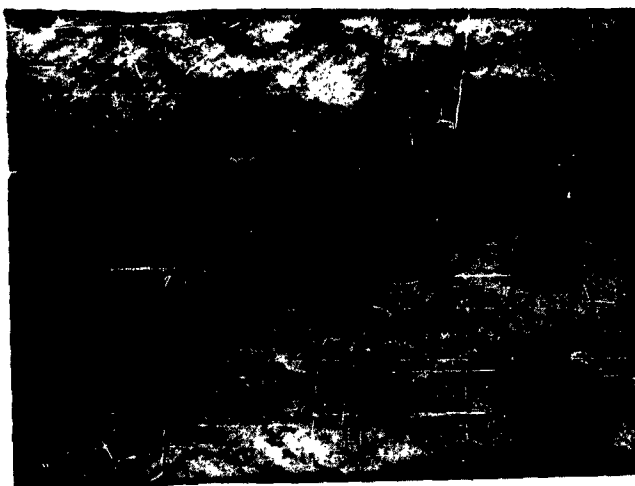
In Figure 10, the aluminum-gold border line at the junction of a sandwich is shown. The dark line from left to right is the border and the lower portion is the aluminum film. In the gold film, shown in the upper portion of the photograph, small pits or globules are present. The aluminum film appears to be much smoother than the gold film.

Burnout Phenomena

After several testing cycles, the sandwich is destroyed and an open circuit is generally observed indicating that the accelerator and/or emitter films have become non-conducting. In some cases, a short circuit is observed indicating that the accelerator and emitter films are in contact. In the former case, the formation of globules, as previously discussed, could possibly be the cause of non-conduction. In the latter case, the short circuit could be caused by the migration of Al ions through the oxide to the accelerator layer.

In any case, these effects appear to be caused by the accumulation of heat that is generated at the junction due to poor heat sink condition.

The relatively poor heat sink conditions are suspected since the glass substrate has a very low thermal conductivity. Also, the films are too thin to conduct away an appreciable amount of heat. If the accelerator and emitter films are increased in thickness to provide higher conductivity, other problems result. The emitter film, which presently is aluminum, has appreciable grain size in the 100 Å range which serves to roughen the surface. If the accelerator is increased in thickness beyond several hundred angstroms, emission cannot be obtained due to the attenuation of the electrons.



1 μm

Figure 10. Aluminum-Gold Border Line at Junction. Gold-Top Portion, Al-Lower Portion (x 27,000).



1 μm

Figure 11. Smooth Strate Unfired (x 16,000).

Two approaches are being taken to solve the problems: 1) to provide a high conductivity substrate such as copper or aluminum, and 2) to evaporate the accelerator layer through a grid so that alternately thick and thin areas are obtained. In the latter case, the thick areas should serve to conduct the heat and current away while the thin areas act as the centers of emission.

It should be mentioned here that there are other means by which the amount of heat generated at the junction can be reduced. One may reduce the junction area and/or perform the testing under pulse conditions at a low duty cycle (10% or less). By using either or both of these approaches, the problem of heat sinking the cathode can be by-passed until it can be solved by direct means. Measurements of other cathode properties can therefore proceed independently of the work on heat sinking. For some applications, small areas and pulse methods will be legitimate solutions to the problem, compatible with the specific requirements the cathode must meet in these instances.

A. Substrates

The requirements for what appears to be a suitable substrate are 1) smoothness, 2) high coefficient of thermal conductivity, 3) low vapor pressure, and 4) adherence between the evaporated film and the substrate. As pointed out in the first quarterly report, the smoothness of the films is dependent on the smoothness of the substrate. The way in which the film grows depends partly on the binding forces between the substrate molecules and the atoms of the evaporant. Assuming that the heat generated during the operation leads to the destruction of the sandwich, a high conductivity substrate is required. Also, since the evaporations are carried out in an ultra-high vacuum system that must be kept clean, the substrate material should have a low vapor pressure.

In this discussion, a surface is considered smooth or not smooth relative to the surface of a glass microscope slide. This criterion is chosen since we have been able to form thin film sandwiches on glass microscope slides which operate from several seconds to several minutes under d-c conditions and several hours under rectified 60 cycle a-c testing. We are assuming that the smoothness of the glass slide is sufficient and that other factors, such as low thermal conductivity, are responsible for the destruction of the unit. Another possible factor which can lead to the destruction of the sandwich is breakdown due to the presence of impurity ions in the oxide layer as discussed in the previous section.

The materials which have been chosen for evaluation as substrate materials are capacitor grade aluminum, OFHC copper, MgAl silicate ceramic, and Smooth Strate. The surfaces of the Al, Cu, and MgAl ceramic have been prepared by electropolishing, chemical and mechanical polishing. The Smooth Strate is a glazed ceramic, commercially available, that has a thermal conductivity coefficient about thirty times that of glass.

The metal substrates are cleaned in the same manner as glass slides. The ceramic and Smooth Strate were cleaned by firing in air at 700 C for an hour. The first tests were made using the Smooth Strate and polished MgAl silicate ceramic. The results of the test showed all sandwiches formed on the MgAl ceramic to be shorted and those formed on Smooth Strate gave about the same results as the sandwiches formed on glass. Electron transmission microscopy was used to study the surfaces of the various substrates. From these studies it was learned that considerable thermal etching occurred whenever the Smooth Strate was fired in air at 700 C. In Figure 11 a transmission microphotograph

of the Smooth Strate surface is shown before it was fired and in Figure 12 the surface is shown after firing in air at 700 C for an hour. In both cases, the magnification is x 16000.

In Figure 13 is shown the surface configuration of a glass microscope slide (x 15,000). The surface of unfired Smooth Strate, shown in Figure 11 appears to be smoother than that of the glass (Figure 13).

A surface replica (x 15,000) of the MgAl silicate ceramic is shown in Figure 14 and appears similar to the fired Smooth Strate shown in Figure 12.

Due to the etching effects of firing, the next Smooth Strate substrates to be used will be cleaned in the same manner as glass slides.

The aluminum substrate surfaces were prepared by mechanical polishing and electropolishing. In Figure 15 is shown an electropolished surface x 27,000. This surface appears smooth relative to glass (Figure 13) or unfired Smooth Strate (Figure 11).

After the aluminum substrates have been prepared, they are anodized to $\sim 200 \text{ \AA}^{\circ}$. An aluminum film is then evaporated and subsequently anodized to $\sim 65 \text{ \AA}^{\circ}$ after which a gold film is evaporated. The initial anodization to $\sim 200 \text{ \AA}^{\circ}$ forms an oxide layer which serves as an insulator between the gold layer and the substrate. The aluminum substrate is not used as the emitter layer since an evaporated film is believed to be of higher quality in that the impurities are not precipitated out.

To date, all sandwiches formed on Al substrates are initially shorted. By probing the substrate and films, it was found that the aluminum emitter film and gold accelerator film were both shorted to the aluminum substrate. During the evaporations of these films, the $200 \text{ \AA}^{\circ}$ oxide layer probably cracks due to the source temperature and possibly the bakeout. The occurrence of cracks



Figure 12. Smooth Strata Fired in Air at 700 C
For 1 Hour (x 16,000).

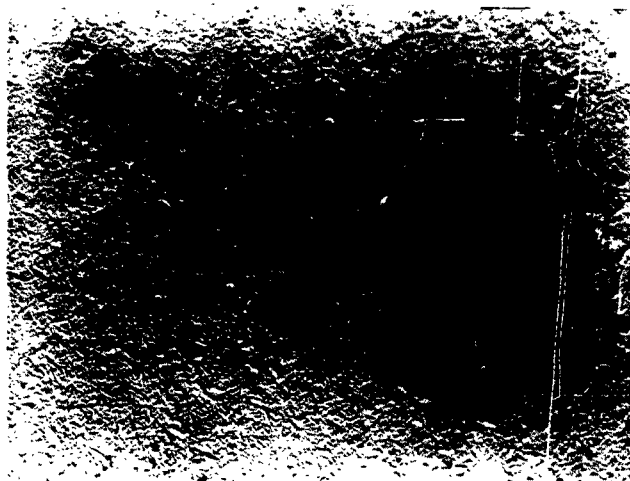
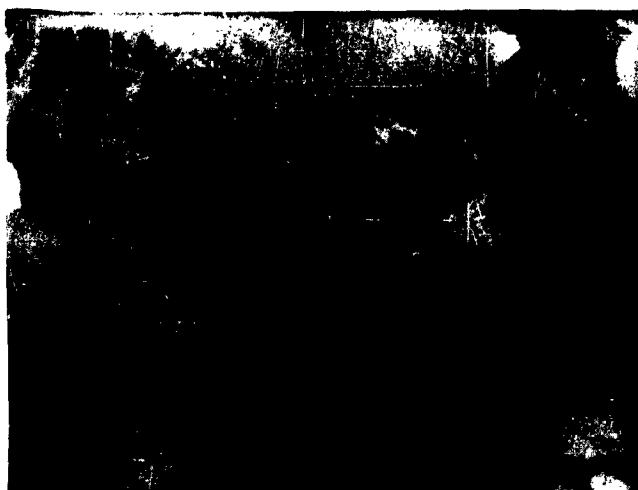


Figure 13. Clean Glass Surface (x 15,000).



1 μ

Figure 14. Surface of Unfired MgAl Silicate Ceramic (x 15,000).



1 μ

Figure 15. Electropolished Aluminum Surface (x 27,000).

could cause the short circuits observed. In future work, the aluminum substrate will be anodized several hundred angstroms thicker or SiO_2 masking will be used.

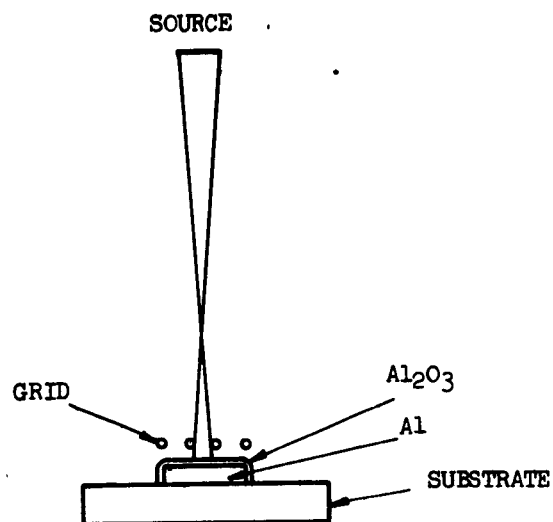
Very little has yet been done with copper since this metal does not anodize. The aluminum film evaporated onto the copper substrate has to be air grown to form the oxide film with a maximum thickness of $20\text{-}30 \text{ \AA}$. This area is masked down using SiO_2 to form an insulator between the gold accelerator film and the copper substrate. The sandwiches made in this way have all been initially shorted. It is possible that the SiO_2 film cracks upon exposure to the source causing the gold to become shorted to the copper substrate.

B. Evaporation Through Grid

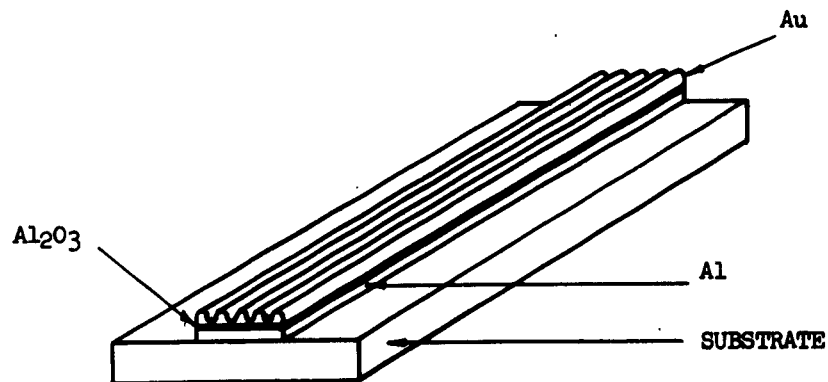
The second approach to provide a heat sink lies in the formation of alternately thick and thin areas of the gold accelerator film. This structure, shown in part B of Figure 16, is obtained by evaporating through a grid as shown in part A. The grid is positioned about 5 mils from the surface of the oxide layer and a defocussing effect is obtained since the source is an extended one. The thickness at the valleys is thin ($< 500 \text{ \AA}$) and should serve as the emission centers, while the thicker ($> 20,000 \text{ \AA}$) rows should serve to conduct an appreciable amount of heat and electrical current.

The grid is of 0.4 mil tungsten wire wound at 716 turns per inch. Gold was evaporated through the grid at a pressure of 1×10^{-4} torr. In three out of three cases, the diodes were initially shorted. Three other evaporations were conducted at 1×10^{-3} torr, in which cases the sandwiches gave I-V characteristics.

The sandwiches with this type of geometry will be tested for emission in the Fourth Quarter.



A. Geometry for evaporating through a grid to obtain the surface shown below.



B. Surface geometry obtained by evaporating through a grid.

Figure 16

It is not exactly known why the evaporations at 1×10^{-3} gave better results than those at 1×10^{-4} . These particular evaporations were conducted in an oil diffusion system and a shutter was not used so that the temperature of the Al-Al₂O₃ began to rise as the source was being heated for the evaporation. This relatively long exposure to the source radiation could have caused the Al₂O₃ to crack in which case the gold would be deposited in the cracks forming the shorts. During the higher pressure evaporations (1×10^{-3} torr) the cracks could very possibly have healed since there was 10 times as much oxygen present.

APPENDIX I

The method originally used for the surface replication of substrates consists of the following steps: (1) Dip the substrate in a bath of petroleum ether so that the desired area of inspection is degreased, (2) Cover and allow to thoroughly dry, (3) Cut small strips of replicating material (Triafol BN, from Naftone, Inc., New York, New York) and degrease in petroleum ether, allowing to dry, (4) Prepare beaker of doubly distilled acetone, (5) Immerse strip of replicating material in acetone until it becomes soft and pliable, while at the same time applying acetone to the substrate from a medicine dropper, (6) Lay softened strip evenly on area of substrate desired and allow to dry for minimum of ten minutes, (7) Strip dried replicating material from specimen, (8) Mount strip conveniently on glass microslide with side that was next to specimen facing outward, (9) Inspect strip under light microscope for breaks, air bubbles, etc., that would produce effects of untrue replication, (10) Attach glass slide to rotary shadow apparatus in vacuum system, (11) Set up apparatus necessary for evaporation of chromium and carbon, (12) Allow vacuum system to pump down to .1 microns of Hg, (13) Evaporate chromium at appropriate angle of incidence for shadowing (25° for ceramic, 30° for glass), (14) Evaporate carbon at 90° angle of incidence onto positive face of replicating material, (15) Allow system to return to atmospheric pressure and remove glass slide, (16) Return strip to bath of acetone and leave until replicating material has completely dissolved, (17) Retrieve floating carbon film on copper specimen screen, (18) Allow to dry thoroughly, (19) Mount copper screen with carbon film in specimen chamber of electron microscope and inspect as desired.

The above described technique is commonly known as the "indirect carbon replica" method using cellulose acetate or polyvinyl alcohol (P.V.A.) primary replica. It yields a true replica of most surfaces and is fairly simple and straight forward to use especially with ceramic substrates.

APPENDIX II

The method of replicating the surface of evaporated thin films (glass substrate) was changed from that described in Appendix I because the final carbon film exhibited increasing tendency to disintegrate into minute pieces in the acetone bath. The general procedure of replication was adhered to with the following substitutions and additions: (1) Nitrocellulose in amyl acetate - concentrations of 1% and 10% - was employed as the replicating material. The solution is dripped onto the specimen and allowed to flow over the desired area. When the amyl acetate evaporates, a very thin film is left that has faithfully replicated the overflowed surface. This film is then stripped off the specimen by first lightly breathing on it to loosen it and then peeling off scotch tape that has been pressed firmly on. (2) When replicating an evaporated gold film, the gold sometimes is pulled off with the nitrocellulose. If this happens, the replica is allowed to float in a solution of aqua regent until the particles of gold have disappeared or both the actual film and replica are examined. (3) After chromium and carbon have been evaporated onto the positive side of the replicating film, the entire accumulation is first immersed in a bath of amyl acetate to dissolve the nitrocellulose. The carbon film is then transferred to a bath of acetone for additional washing and finally inserted into a bath of distilled water to uncurl the film.

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