THE PRODUCTION OF THIN METALLIC FILMS
OF
CONTROLLED TRANSMISSION

By

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and

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The assistance and suggestions given by Mr. E. S. Bishop and Professor J. L. Ryman are held in esteem. Gratitude is felt for Mrs. Thelma Pruett, who patiently and ably typed the paper, and for the many other persons whose contributions aided materially to the successful completion of the research.

Introduction

It is a well known fact that a thin film coated on any surface may change the properties of that surface completely. Mechanical structure, resistance to corrosion or contamination, optical constants, electric and magnetic behavior, thermal emissivity, photoelectric and thermionic emission, etc., of a surface depend very much on that film. It is also generally observed that the characteristics of thin films are radically different from that of the same substance in massive form and vary with the thickness. There is no general agreement on the limits of thickness of thin films. Usually, it covers the range from a monomolecular layer to a thickness of several microns.

The enormous controlling effect on a surface and the anomaly in character of a thin film aroused profound interest among theoretical and practical fields in both chemistry and physics. A great deal of research work has been done with thin films and numerous applications have been found. First surfaced mirrors, non reflecting lens and beam splitters, widely applied in photography, interferometry, spectroscopy and other optical fields; coated quartz fibers extensively used in sensitive electric instruments such as quadrant electrometers, string galvanometers, and electrocardiographs; electronic tube components coated for better thermionic or secondary emission; interferometric filters recently developed for band or monochromatic transmission are some of the well known examples of their uses. Furthermore, the technique of thin film coating is found very essential and widely used in crystallography, metallurgy and electron microscopy

The production of thin metallic films may be accomplished by any of the following methods:

1. Chemical and Electro-chemical Methods:
   a. Electro-deposition
   b. Reduction
   c. Thermal decomposition

2. Physical Methods
   a. Thermal evaporation
   b. Cathodic sputtering[1]

Of the two physical methods, thermal evaporation is the better in most cases. It requires simpler apparatus, is more flexible, and consumes less time for the complete operation. It will be the method considered and adopted in this work.

Thermal evaporation is the process of heating a metal or non-metal in a high vacuum, to near its boiling point, whereupon it emits molecular rays in all directions.[2] The distribution of the molecular rays depends only upon the degree of vacuum. If the vacuum is sufficiently low they are emitted linearly, similarly to light rays, and if the vacuum is not low enough, the distribution of the metal behaves in accordance with the laws of diffusion.[3] The degree of vacuum affects the chemical nature and physical properties of the thin films, as does, also, the temperature of the source and the distance from the source to the substrate. The degree of vacuum should be such that the mean free path of the molecules from the source should be greater than the distance from

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[1] Lewis, W., Thin Films and Surfaces, London: English Universities Press, Ltd., p.19.

[2] Strong, J., Procedures in Experimental Physics, New York: Prentice-Hall, Inc., (1939) p.168.

[3] Lewis, op. cit., p.20.

the source to the substrate.[4] If the temperature of the source is excessively higher than necessary to evaporate the metal, the deposits are pulverulent and non-adherent, due to the vapor density of the gaseous metal becoming too great.[5] This process was known as early as 1890, at which time Edison obtained patents in connection with it, however, it was not widely used until after the developement of the bare tungsten heater technique by R. Ritschl and the development of high speed vacuum pumps.

The type of apparatus most frequently mentioned in the literature as having been used for evaporation chambers have been glass bell jars mounted on flat glass or metal bases. The substrate was placed either above or below the source if the metal was evaporated from some variety of filament coil and above the sourc if the metal was evaporated from a crucible. Crucibles were used when the material did not adhere to a filament coil or when the material was in powder form. It was recommended that small systems be made of glass, since there is less danger of leaks, and large systems that employ large flat surfaces be made of metal, since glass will break under the high pressure.[6] Strong[7] describes a vertically mounted steel bell jar that was used at the California Institute of Technology for making mirrors up to 40"

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[4]Lewis, op. cit., p.20.

[5]Ibid., p.21.

[6]Yarwood, J., High Vacuum Technique, New York: Wiley & Sons, Inc.,(1946) p.28.

[7]Strong, op. cit., p.173.

in diameter. A horizontal system of metal was suggested by Tolansky.[8] Yarwood[9] suggested that the chamber could be made from metal and listed copper, brass, gun-metal, drawn and cast steel, and aluminum as the ones most commonly used and suggested tin-plating, painting with shellac, or even coating with "W" wax as useful techniques for closing pores of metal.

Since the thickness of a thin film affects greatly its characteristics, it is, therefore, extremely important to production as well as research work, to make thin films of desired thickness However there is very little that has been published on thickness control. Only two references to any method of thickness control were found. Tolansky[10] and Yarwood[11] mentioned that films of desired thickness could be made by use of a photoelectric cell but no details were given by either of them. It is, therefore, the object of the present investigation to construct an evaporation apparatus by means of which thin films of predetermined thickness can be produced.

The method of thickness control employed in this research consisted of shining a light, placed inside the chamber, on a photo-multiplier tube, placed adjacent to a window in one end of the chamber, and letting the evaporating metal being deposited on a glass plate between the light and the tube cause a decrease

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[8]Tolansky, S., Multiple-Beam Interferometry, Oxford: Clarendon Press, (1948) p.26.

[9]Yarwood, op. cit., p.29.

[10]Tolansky, op. cit., p.27.

[11]Yarwood, op. cit., p.87.

in the photo-electric current sufficient to open a sensitive relay and thereby actuate a shutter mechanism.

Aluminum was chosen from the evaporating material because it adheres well to glass, is readily available, is easily evaporated from tungsten coils, and has high reflecting power at practically all wavelengths. Aluminum mirrors are particularily suited for reflecting ultra-violet light.

A survey of the literature revealed that a large amount of research had been done in finding suitable filaments from which metal could be evaporated and in finding suitable metals to evaporate since success depends on "the thermal stability and vapor pressure of the material and the practicality of bringing material to the evaporation temperature in a vacuum".[12] It showed in some cases a complete reversal of earlier opinion as to the suitability of using some particular filaments with the commonly evaporated metals. Strong[13] mentioned difficulty in evaporating aluminum from tungsten, because of reaction between tungsten and aluminum and the failure of aluminum to adhere or wet the tungsten The same thing was noted by Countryman[14] a few years later. However, in an article which described the results of evaporating 26 metals from 9 different filaments, Caldwell[15] reported that aluminum adhered well to tungsten, evaporation took place readily and the reaction between aluminum and tungsten took place slowly

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[12] Strong, op. cit., p.168.

[13] Strong, J., Phys. Rev., 43, 498 (1933).

[14] Countryman, M. A., Jour. of Applied Physics, 8, 432 (1937).

[15] Caldwell, W. C., Jour. of Applied Physics, 12, 779 (1941).

with little effect on evaporation. Strong[16] later noted that the solubility of tungsten in aluminum was about 3% by volumn and since its spectrum could be observed during evaporation, the tungsten might be boiling away but chemical analysis of metal films gave no positive indication of the presence of tungsten, a concentration of 0.03% by weight being detected. Olsen, Smith, and Crittenden[17] found that aluminum would be successfully evaporated provided the weight of the aluminum did not exceed 35% of the weight of the tungsten coil. Strong[18] suggested that the dissolved tungsten appears to be almost completely precipitated back onto the coil as the evaporation proceeds. It was illustrated by Strong that the aluminum does not evaporate directly from the fused metal but from the tungsten wire. He made a "self-photograph" of the molecular rays of aluminum passing through a pinhole and depositing on a glass substrate. The "self-photograph" showed the outline of the whole coil and consequently the aluminum must have come from all points on the filament. An improved technique used later by Strong[19] consisted of three or four small diameter tungsten wires twisted together and then the filament shaped into a helical coil. The molten metal flowed into the space between the wires and completely covered the tungsten wire giving what he called a "minimum

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[16] Strong, J., Procedures in Experimental Physics, New York: Prentice-Hall, Inc., (1939) p.173.

[17] Olsen, L. O., Smith, C. S., and Crittenden, C. R., Jour. of Applied Physics, 16, 425 (1945).

[18] Strong, op. cit., p.173.

[19] Strong, loc. cit.

'ratio' of heat radiation to molecular radiation of aluminum". Confronted with the problem of the filaments burning out, Edwards[20] experimented with various types of filaments including straight wires, two parallel wires, troughs of graphite, etc. Success in using tubes of aluminum wrapped around tungsten coil which had been insulated with aluminum oxide was noted by DeVore.[21]

All of the publications seemed to use a fairly standard evaporation technique with some minor variations. The procedure followed was to pump the system down to about 0.1 mm. of mercury and prefuse the coil, that is, heat the filament and melt the material to be evaporated. At this pressure none of the contaminated metal would reach the substrate. After waiting for a period of 15 minutes, a diffusion pump was cut on and at a sufficiently low pressure the filament heated until evaporation began to take place and then a shutter mechanism opened and the evaporated metal was allowed to reach the substrate.

The pumping speed of the pumps chosen should be such that the desired vacuum will be maintained at all times during the evaporation process. By Gaede's definition of pumping speed[22], it is the speed as measured at a given pressure, and equals the volume of gas pumped from a vessel at that pressure. To determine the pumping speed desired, the formula

$$S = \frac{V}{t_2 - t_1} \ln \frac{p_1}{p_2}$$

where V is volume of container, p_1 and p_2 are pressures at times

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[20] Edwards, H. W., Rev. Sci. Instruments, 4, 449 (1933).

[21] DeVore, H. B., Rev. Sci. Instruments, 9, 202 (1938).

[22] Yarwood, op. cit., p.55.

t_1 and t_2.

In order to obtain a uniform film, Strong[23] suggested the use of several tungsten filaments suitably arranged, rather than one movable source. The condensation on a plane surface is proportional to the inverse square of the distance from the source, and to the cosine of the angle between the normal to the surface and line connecting the point to the source[24].

To obtain a satisfactory deposit cleanliness and a low vacuum is essential, therefore, the substrate should be cleaned with great care. The cleaning of the substrate was found to follow a generalized pattern. It was first scrubbed with an ink eraser to free surfaces and edges from rouge and other contaminations. The eraser is suitable for this because of the abrasive action it has due to the pumice or ground glass in it. The mirror is next washed with soap and water or Aerosol[25] and water. Aerosol is preferred to soap since it can be washed off the face of the mirror without leaving any residue. The substrate was rinsed with distilled water and dried with a clean cotton towel. Finally the glass is exposed to a glow discharge during evacuation.

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[23] Strong, op. cit., p.177.

[24] Tolansky, op. cit., p.28.

[25] The compound Aerosol OT is manufactured by the Selden Division of the American Cynamid and Chemical Corporation, Bridgeville, Pa.

Construction of Apparatus

For convenience we shall consider the apparatus to be divided into three major components, the chamber assembly, the control system, and the pumping system.

Chamber Assembly

The chamber assembly (Plate 1) included the evaporation chamber, evaporation plate, control plate, and wire cage.

In the construction of the evaporation chamber, a section of 10" steel pipe was used. The outside of the ends were beveled approximately 1/4" and two steel rings, 1" square in cross section, were sweated on and a bead of fluxless weld laid around the chamber between the bevel and the ring. The chamber was sandblasted inside and out to remove the rust and then was placed in a lathe and the rings and ends faced off together. A cylindrical steel lug, 2" long and 2" in diameter was welded on the outside of the chamber at the midpoint from the ends, and a hole was drilled through the lug and the wall of the chamber and tapped with a 1/2" pipe tap. A brass nipple (Fig.1) was screwed into the lug until it seated against the machined surface of the lug. An effort was made to use a gasket between the lug and the nipple but no success was achieved with either rubber or lead because of the small surface area involved. The joint between the lug and the nipple was then sealed satisfactorily with Apiezon wax "W".

The inside of the chamber was given a coat of Apiezon wax

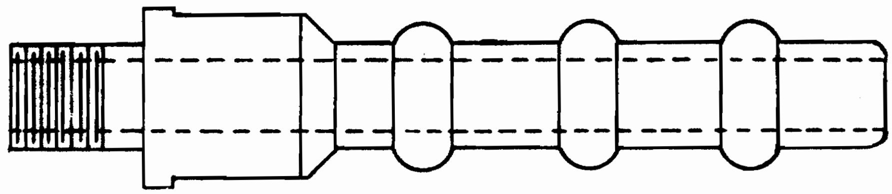

Fig. I Brass Nipple

"W" and the outside was given three coats of glyptal.[1] The Apiezon wax was applied by heating the outside of the chamber with an oxygen and gas burner until the inside was not enough to melt the wax, and the wax in stick form was rubbed against the walls until a layer was deposited on them. This was done to prevent rusting, to stop possible leaks, and to give a surface with a low vapor pressure[2] and one that did not absorb gases.[3] The glyptal was applied also to prevent rusting and to stop any possible leaks. It was recommended in applying glyptal that different colors be used alternately to insure complete coverage of the surface to which it was being applied.[4] The end surfaces of the chamber were hand ground with carborundum

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[1]Strong, J., Procedures in Experimental Physics, New York: Prentice-Hall, Inc., (1939) p.176.

[2]ibid., p.557.

[3]ibid., p.126.

[4]ibid., p.134.

and water, using a circular disk of plate glass as a grinding surface. A ring of synthetic rubber 1/16" thick was used as a gasket between each end of the chamber and the end plates. Celvacene, a silicone grease, was applied to each side of the gasket to insure a vacuum seal. It was found necessary to renew the grease occasionally as the continued pressure seemed to cause the grease to harden and the heat from the evaporation filaments, to cause the grease to become powdery.

The right hand end plate, the evaporation plate, was constructed of 1/4" brass. It was secured to the end of the chamber by eight 3/8" by 1" studs equally spaced about a circle 5 7/8" in radius on the end face of the chamber. The weight of the plate was supported by two 1/8" guide pins placed at the two intersections of the vertical center line and the circle upon which the studs were placed. This plate contained the four filament electrodes which were constructed from a brass rod and fixed on a horizontal line through the center of the plate, insulated and a grounded post on each side of the center so that two filaments could be operated simultaneously or separately. The two grounded posts consisted of a rod 3" long and 5/8" in diameter, silver soldered to the plate 2 5/8" apart. The insulated posts were 4" long with one end turned down to 1/4" for a distance of 1" and threaded. The posts were insulated from the plate with mica and bolted to the plate by means of the threaded end. They were placed 8 5/8" apart leaving a distance of 3" between each pair of filament post for the filament wires. To fasten the filament to the post and insure a good electrical contact, the end of the filament posts (Fig. 2) were made in the form of a clamp. The top surface of the filament post was milled 3/16" for a distance of 1" from the end of the post.

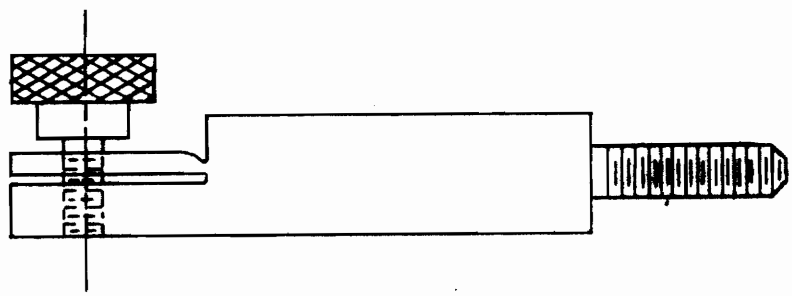

Fig. 2 Filament Electrode

One-eighth inch below the milled surface and parallel to it a slot was sawed into which the ends of the filament wires were placed and held firm by pressure from a knurled nut. The clamping action was aided by filing a notch on the top of the clamp where the milled surface met the shank of the filament post.

The shutter assembly consisted of a shutter, brass rod, "O" ring vacuum gasket and housing, and a trigger mechanism. The shutter was a two quadrant sector disk of 1/8" aluminum, 9 1/2" in diameter. It was fastened rigidly to the shaft by means of a collar and set screw. The shaft was a standard 1/4" brass rod 5" long fitted through the "O" ring vacuum gasket and housing. This housing was the type recommended by Linear Inc.,[5] (Fig. 3). It consisted of a cylinder of brass 1" in diameter and

[5]For description of this and other vacuum gaskets, see brochure from Linear Incorporated, Philadelphia, Pa.

2" long. A hole was drilled through the longitudal center of the housing and reamed with a 1/4" standard reamer. This hole counterbored with a 23/64 drill for a distance of 11/16". A bushing of 18/32" length, 23/64" outside diameter and 1/4" inside diameter was made and pressed into counterbored portion of hole until flush with end of housing, thus leaving the desired

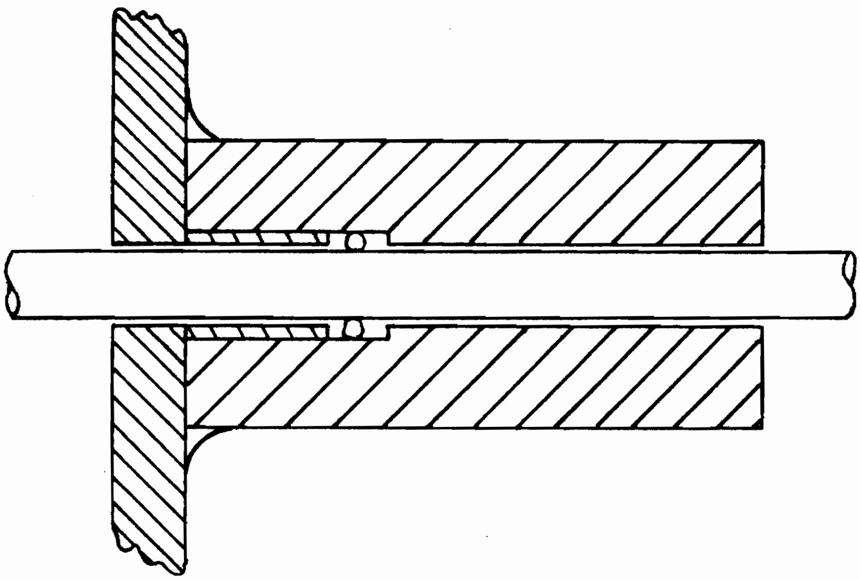

Fig. 3 "O" Ring & Housing

recess in the housing for the "O" ring gasket. A 1/4" hole was drilled in the center of the plate and the housing was then silver soldered to the plate, being aligned with the hole in the plate by a 1/4" rod inserted through housing and hole in plate before soldering. A 1/4" "O" ring vacuum gasket was placed in the housing and the shaft inserted through it.

The trigger mechanism consisted of a spring loaded arm 4 1/2" long fastened to the shaft by means of a set screw and acted as a lever arm to rotate the shaft, the spring pulling the shutter closed. The shutter was held in the open position by locking the lever arm with a sliding bar which was connected to the core of a solenoid. When the solenoid was energized, it drew the bar from under the lever arm and thus allowed the spring to close the shutter.

The left hand end plate, the control plate, which contained a window and the jacks for bringing current to the control light, was constructed of 1/4" steel. The control plate was originally constructed of 1/2" glass but it broke as a result of the atmospheric pressure. Above the center of the plate on the vertical center line, a hole 3" in diameter was cut, its location such that its center was on the same horizontal line as that of the control light. The inside of the plate, where it made contact with the rubber gasket, and the area on the outside of the plate adjacent to the window were hand ground in the same manner as the ends of the chamber. The hole in the plate was covered with a circular disk of glass 1/2" thick and 4 1/2" in diameter, which was sealed to the plate with Apiezon wax "W". Two jacks (Fig. 4) were constructed and bolted on the plate on a horizontal line below the window and symmetric with the vertical center line. Both jacks were insulated from the

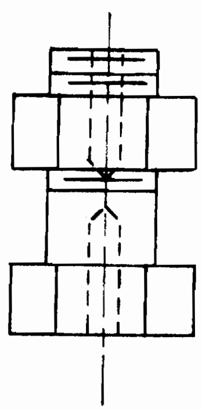

Fig. 4 Control Plate Jack

plate with 1/32" mica and joints sealed with Apiezon wax "W" on both sides of the plate. The plate was given three coats of glyptal on the outside and greased on the inside with Celvacene to prevent rusting.

The removable wire cage was built up from 1/4" steel rods. Two of these rods were bent into a circle of 8 1/2" outside diameter and ends welded together. These two end pieces were connected together by six 1/4" steel rods, 23" long, symmetrically placed around the circles and welded. Across the two top rods a strip of 1/4" bakelite, 1" by 5", was fastened flat by means of two hook bolts, and wing nuts making it possible to vary its position along the length of the cage. A 6-8 volt automobile head light bulb was mounted in a sheet iron housing, which was fastened to the above strip at its center. One electrical lead was soldered to the housing and the other to the contact inside the socket and insulated from the housing. These two leads were brought forward along the base of the cage and connected to the two jacks in the left hand plate. Across the two bottom rods, two strips of 1/4" bakelite, 1" by 6", were connected to the cages. These strips acted as supports for the cage and insulated it from the chamber.

The substrate holder consisted of an adjustable circular plate (Fig. 6) with a clamp located in the center. The clamp was an adjustable plate of 1/8" aluminum, 2" in diameter, and secured to the holder by a bolt and winged nut. A rectangular section was cut out of the holder and covered with a plate of 1/8" glass. This was used as the control window, since it and

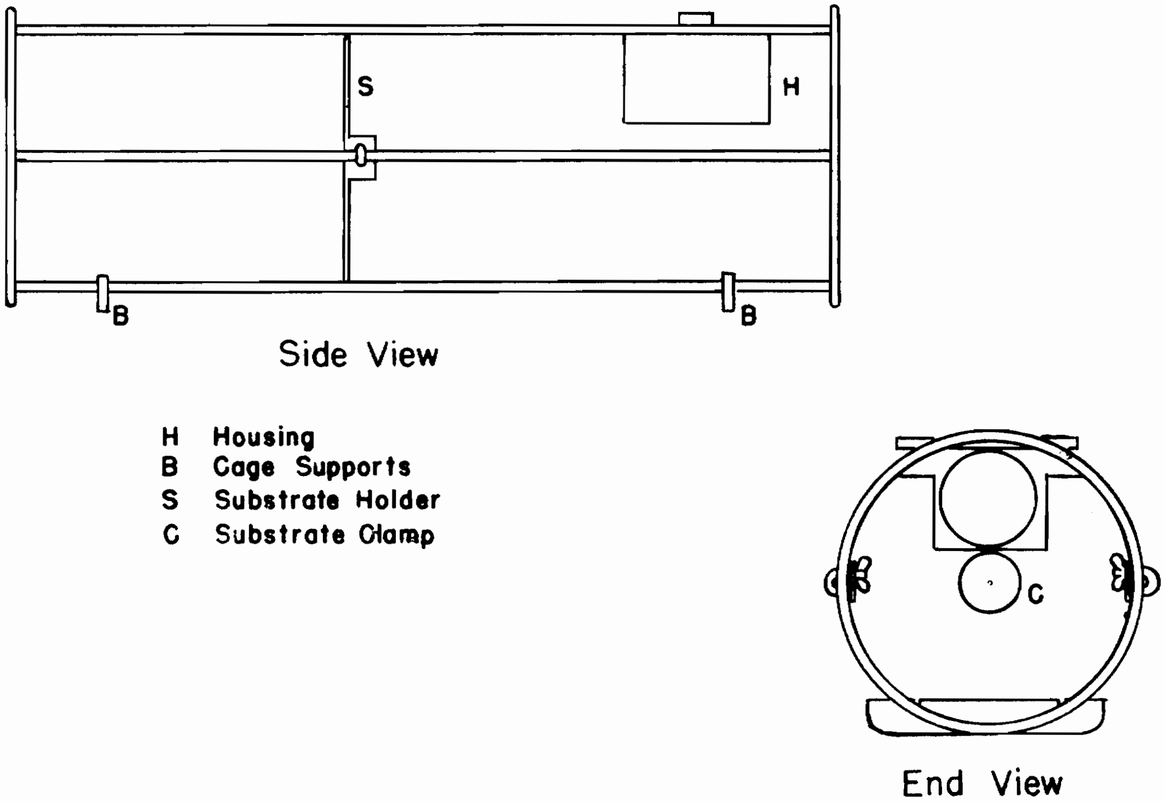

Fig. 5 Wire Cage

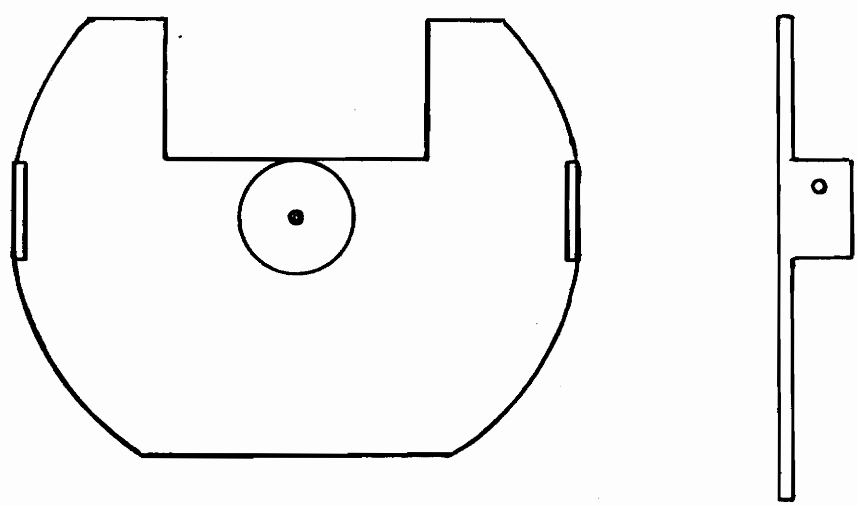

Fig. 6 Substrate Holder

the substrate received metal evaporated from the filament under the same conditions. The control window and the test substrate were clamped to the holder by the same clamp.

Control System

The control system included a power supply, a photomultiplier tube, a control light circuit, a solenoid, and an evaporation filament circuit.

The direct current power supply was a 1000 volt full-wave rectifier[6] employing two 2X2/879 high voltage diodes. The output of the rectifier was smoothed with a one stage condenser-input filter and then placed across ten 22000 ohm resistors connected in series. The resistors acted as a voltage divider with 100 volts across each resistor. Each of these 100 volt steps were placed across a dynode of a 931-A photo-multiplier tube (Fig. 7). The tube received light from the control lamp located inside the evaporation chamber. The electrical energy for this light was obtained from a 110 to 7 volt step-down transformer. A Variac was placed in the primary of the transformer for controlling the voltage. Thus, it was possible to control the intensity of the control light by varying the potential across the primary of the transformer. Therefore, the intensity of the light was recorded in terms of voltage. With this light source held at constant intensity, the light reaching

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[6]Radio Corporation of America, RCA Victor Division, RCA Tube Handbook, HB-3. Phototube section, 931-A. Typical Circuits, Dec. 1, 1943.

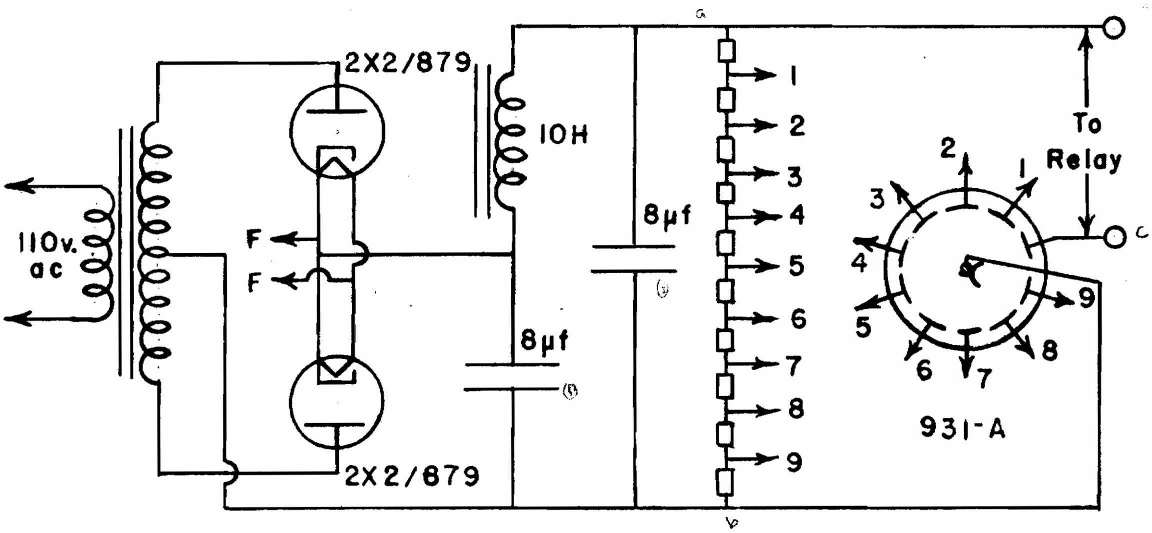


Fig. 7 Phototube Power Supply

the phototube was controlled by the thickness of the thin metallic film deposited on the control window, since the window was between the phototube and the light source. The variation of the intensity of the light reaching the phototube resulted in a variation of the photo-electric current. The photo-electric current held a relay closed, and when the current dropped to a certain value the relay opened. This

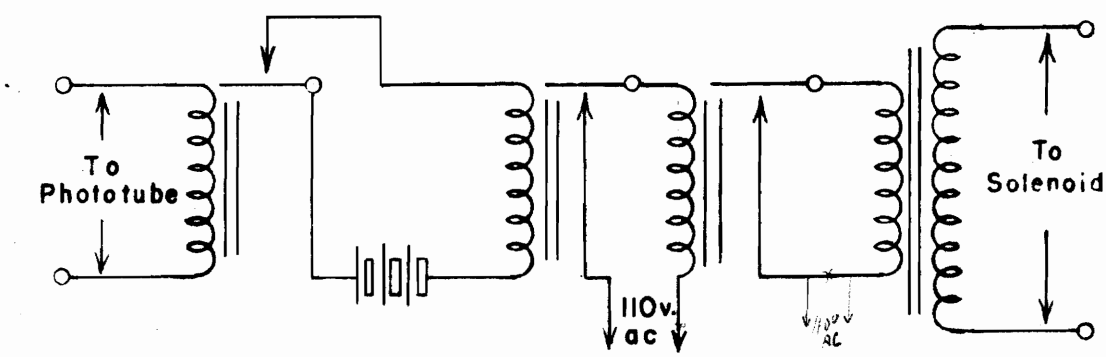


Fig.8 Relay Circuit

actuated a 6 volt direct current relay which, in turn, closed an alternating current relay (Fig. 8). The alternating current relay was connected in the primary side of the 110 to 220 volt step-up trnasformer supplying the solenoid and thereby served as a switch for the solenoid.

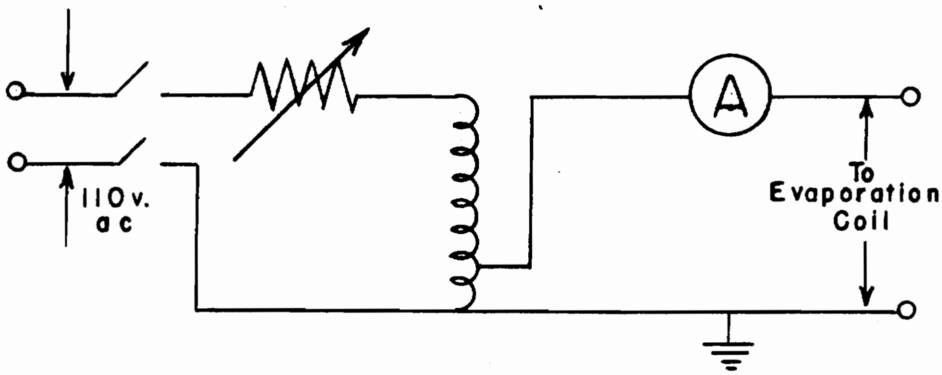


Fig.9 Evaporation Filament Circuit

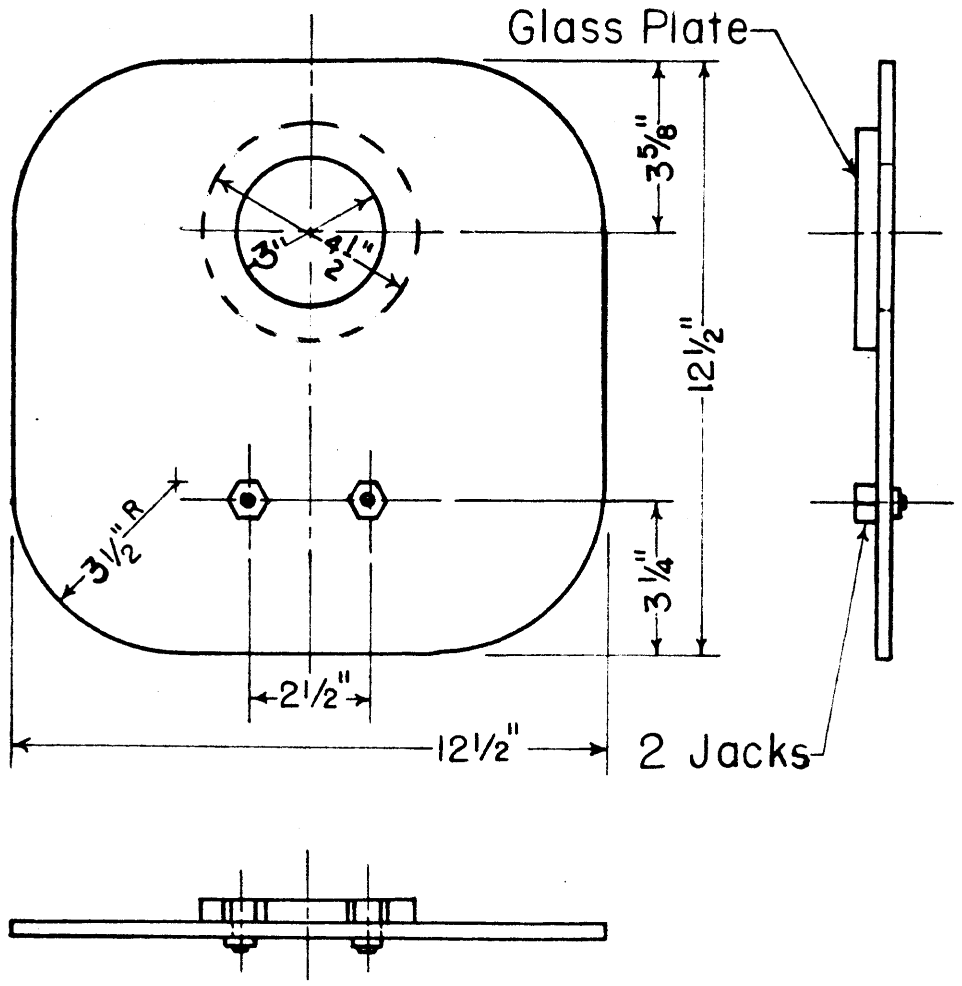
The evaporation filaments were supplied with electrical energy from an auto-transformer (Fig. 9) with 110 volts applied across the transformer's ten coils and the output taken from the first coil. This gave a potential of 10 volts and a current of approximately 35 amperes across the filaments.

Pumping System

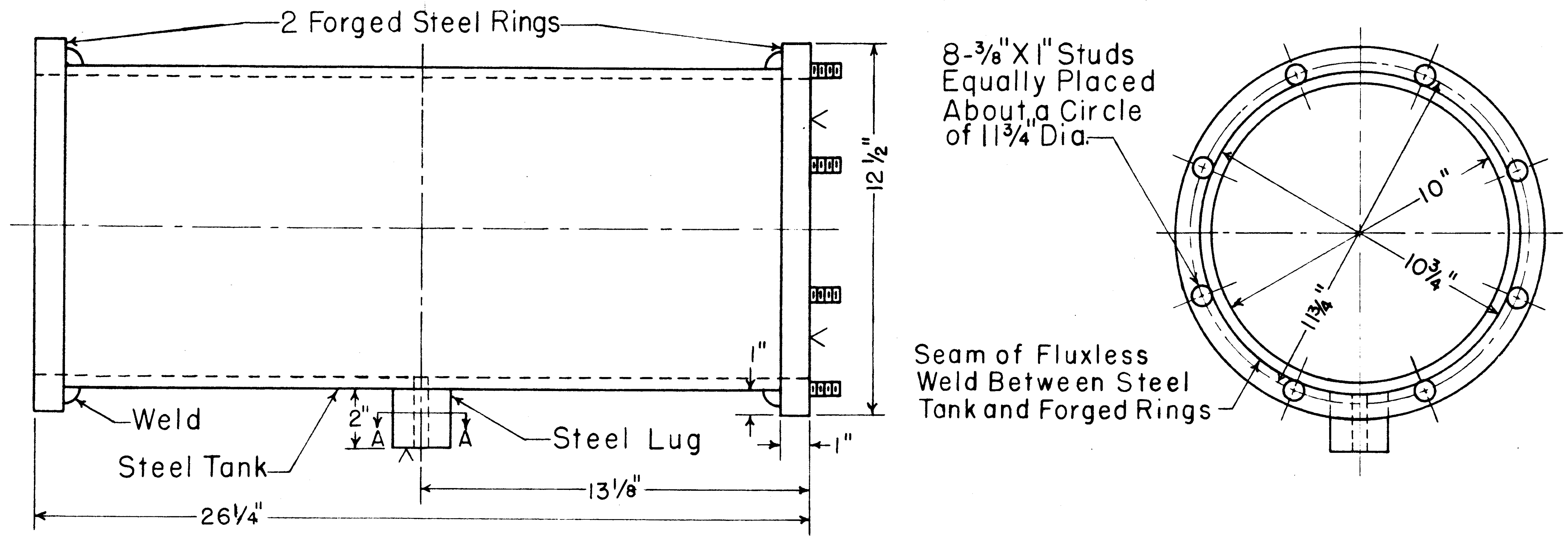
The pumping system included the mechanical pump, the oil diffusion pump, the pressure gauge, and the glass and rubber connections.

The mechanical pump was a Megavac, manufactured by the Central Scientific Company, Chicago, Illinois. It was rated to pump down a "suitable" system to a pressure of 0.1 micron of mercury, however, it pumped this system to about 3 microns and occasionally to as low a pressure as 2 microns. The mechanical pump was used as a backing pump for an oil diffusion pump, which was connected in series with it. The diffusion pump was a type 6FVC made by Distillation Products Incorporated. A 110 volt source was connected directly to the coils of the pump and the resistance of the coils was found to be such that the heat was just sufficient to vaporize the oil. With both pumps on, the system was pumped down to a pressure of 0.2 microns of mercury, which was sufficient for evaporating aluminum.

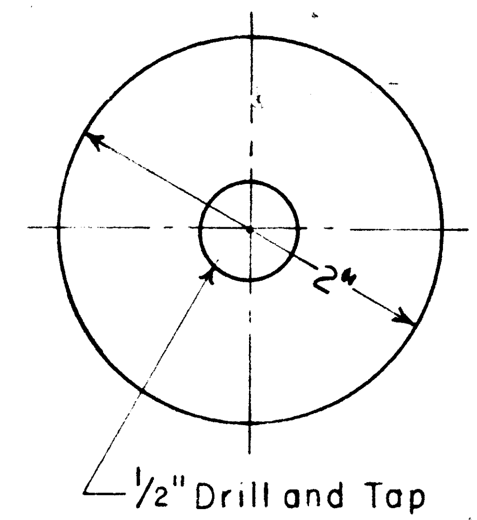
The pressure of the system was measured with a McLeod gauge. It was connected to the system with a glass tee, with one branch of the tee connected to the tubing between the system and the pumps, one branch connected to the McLeod gauge, and the third carrying a stopcock for admitting air to the system. Glass tubing was used to connect the McLeod gauge to the system after two futile attempts to use rubber tubing had been made. All glass to glass joints, except those to the diffusion pump, were made by inserting the ends of the glass tubing into a short piece of rubber tubing. The glass tubing was first coated with heavy Celvacene, and after the joint was made, the excess Celvacene was cleaned off with alcohol. The joint was then sealed with Apiezon wax "W" and given several coats of glyptal. Seven-eighths inch rubber tubing was used between the pumps to make the connections more flexible and eliminate the danger of damage to the glass system by movement of the mechanical pump.



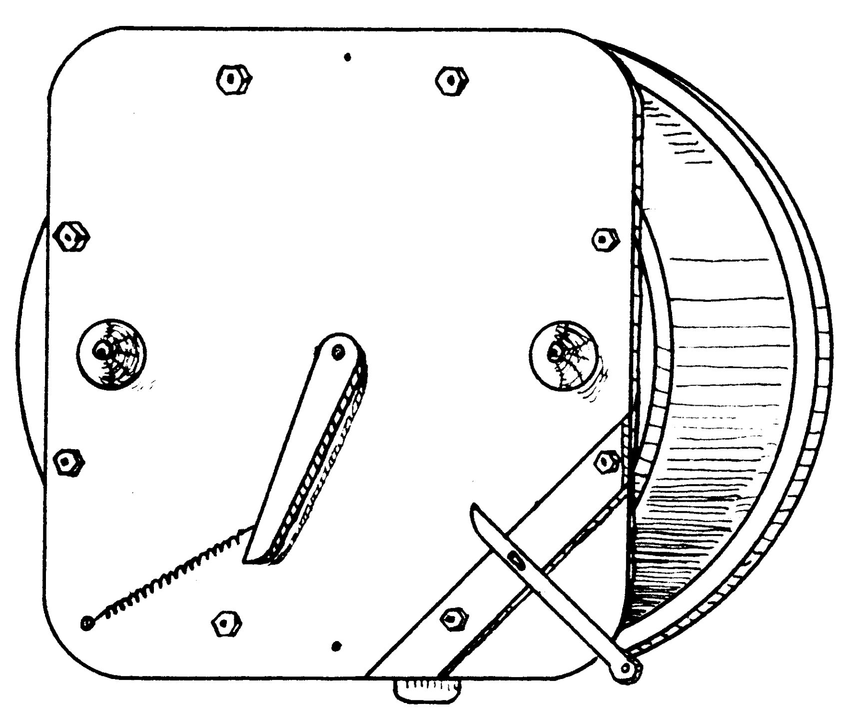
Control Plate



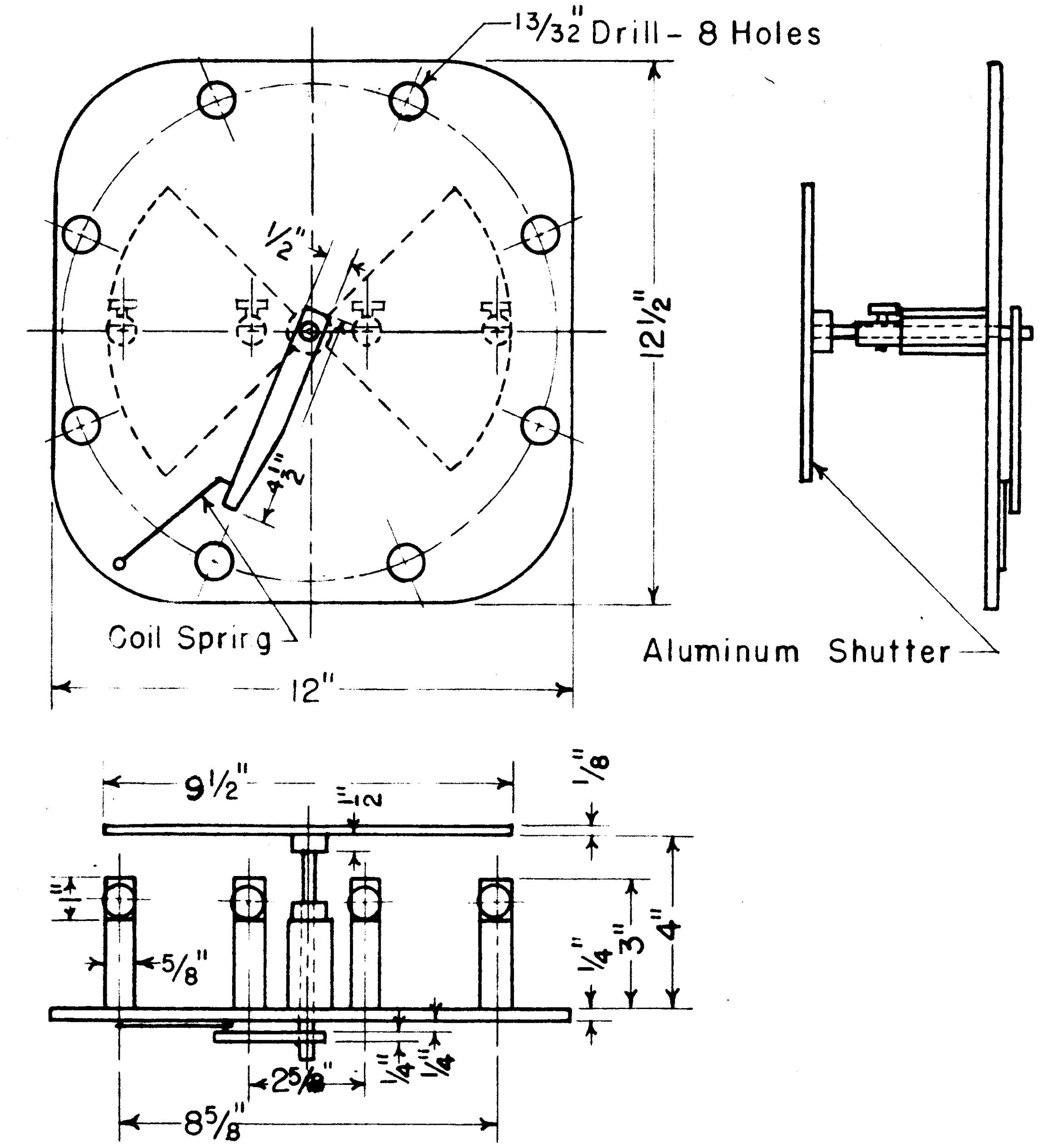
Evaporation Chamber



View A - Full Scale

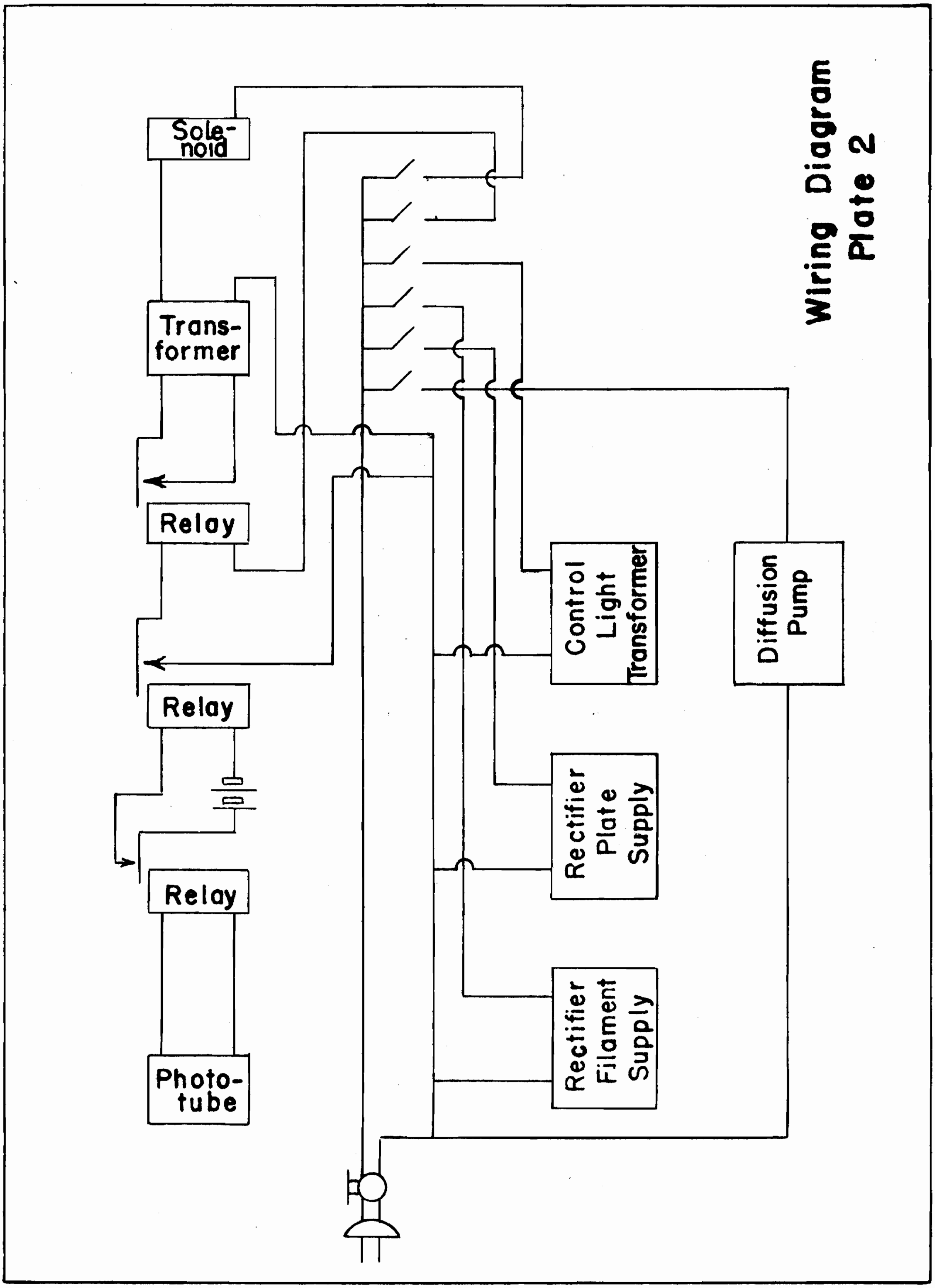


Perspective View



Evaporation Plate

VIRGINIA POLYTECHNIC INSTITUTE
PHYSICS DEPARTMENT
Evaporation Apparatus
Plate I
Lyde S. Pruett
Robert L. Smith
1950
Scale 1"=4"



Wiring Diagram
Plate 2

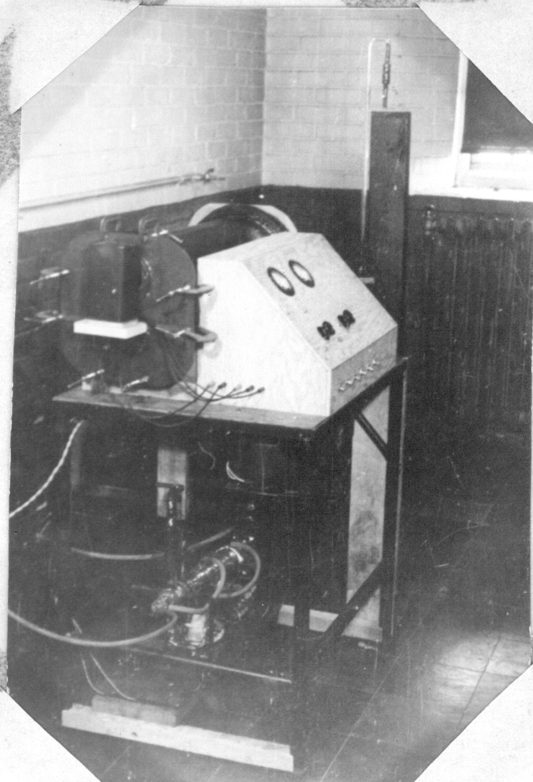


Fig. 10

View of apparatus showing control plate.

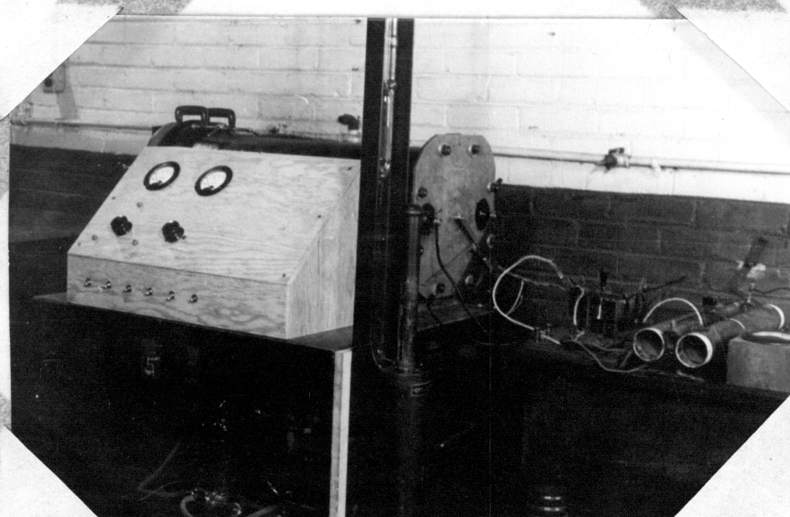


Fig. 11

View of apparatus showing evaporation plate.

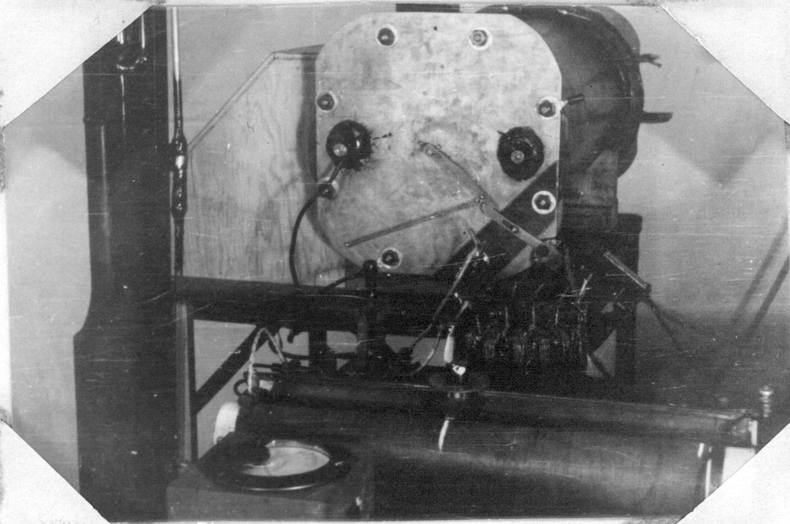


Fig. 12

View of chamber showing arrangement of trigger mechanism and solenoid.

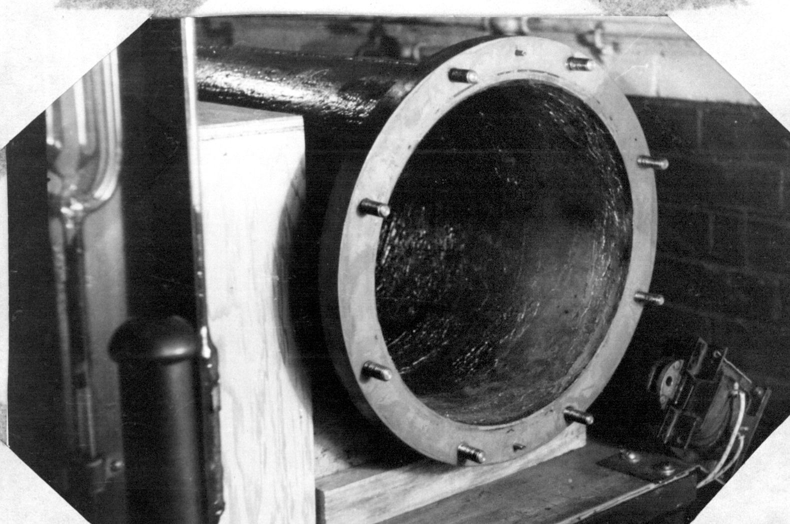


Fig. 13

View of chamber with evaporation plate removed.

Fig. 14

View of evaporation plate showing filament coils and shutter.

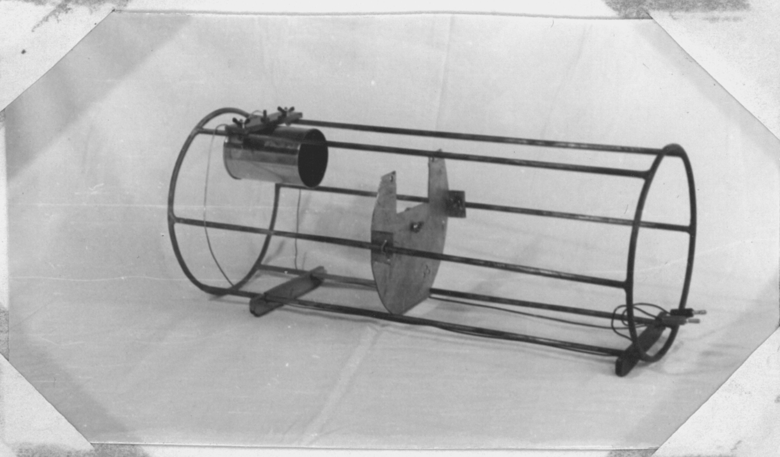


Fig. 15

View of wire cage, substrate holder, and lamp housing.

Evaporation Technique

The filaments were made of 1 mm. tungsten wire wound into helical coils 2 1/2" long with three loops to the inch. Clips of # 18 aluminum wire 1/2" long were bent into a U and fastened to the turns of the coil. The filaments were inserted into the filament posts from the control plate end.

The test window and sample slides, standard 1" by 3" biology slides, were cleaned with alcohol, washed in a solution of Aerosol, rinsed in tap water, and finally in distilled water. The slides were dried with a soft cotton towel leaving as little lint on the surfaces as possible. The test window and sample slides were clamped to substrate holder and the wire cage returned to the chamber. The control plate was then clamped to the end of the chamber with eight D-clamps.

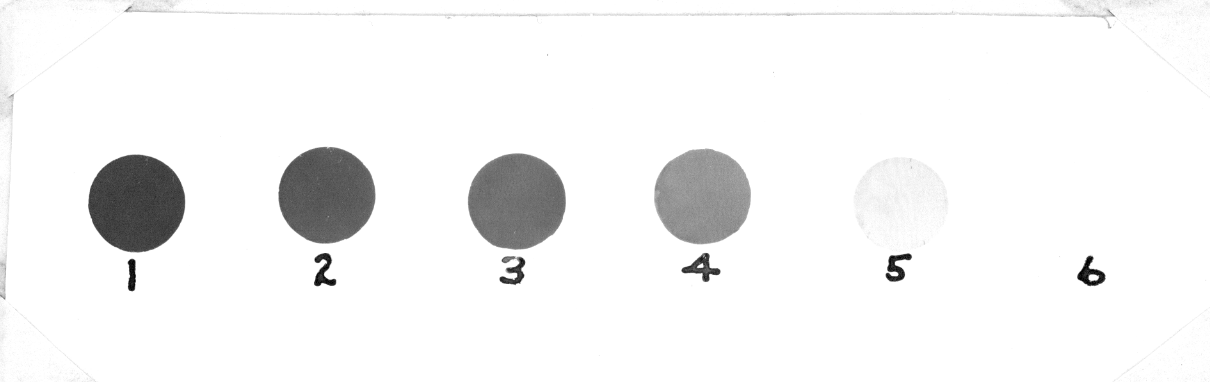
The Megavac pump was started and allowed to pump the system to 10 microns or below before the diffusion pump was turned on. Sometimes the Megavac pumped the system down to 20 or 30 microns and seemed to be unable to pump it lower. The best procedure found was to then cut the pump off and let the system stand for an hour or two, after which time the Megavac was started again and the system pumped to 10 microns or less. When the system reached 5 microns or less, the coils were heated and the metal fused. The pressure usually rose to about 9 or 10 microns during this phase and the evaporated process was stopped until the pressure was brought back to the desired value. The electrical lead from the ground side of the line was connected to the filament posts that were soldered to the evaporation plate and the chamber itself grounded, so that it was safe to touch the chamber while the filament coils were being heated. With the coils in readiness to be evaporated, the power supply for the phototube was cut on and the Variac to the rectifier transformer set at 84 volts which gave the proper voltage across the dynodes. The control light was then turned on and the Variac adjusted until the desired voltmeter reading and hence desired control light intensity reached. The trigger mechanism of the shutter was cocked and the filament current applied to the coils. When the desired film was deposited, the solenoid was energized and the shutter closed. The power to the filaments, the control light, the solenoid, the power supply, and the diffusion pump heater coils were then cut off by manually operated switches. When the diffusion pump had cooled sufficiently, the Megavac was cut off, air let into the system, and the sample slides bearing the films were removed from the chamber.

Calibration

The apparatus was calibrated by setting the intensity of the control light at a predetermined level and depositing the metal on a glass substrate until the electronic curcuit closed the shutter and stopped the evaporated metal from striking the substrate. The distance from the source to the substrate was 12" throughout the calibration. The mirror thus produced was removed from the evaporation chamber and its transmission for white light was determined by a spectrophotometer. A calibration curve of volts vs transmission was plotted for the ápparatus. The four points for this curve were obtained by setting the intensity of the control lamp such that it had 74, 78, 86, and 90 volts, respectively, in the primary of the control light transformer. The transmission of the four mirrors produced was determined by placing one, first in the upper part of the split beam of the spectrophotometer and obtaining a reading and then in the lower portion of this split beam and obtaining a reading and continuing this procedure until three sets of reading were produced for each mirror. The six readings obtained for each mirror were averaged and recorded as the transmission of the mirror (Table 1).

The calibration curve was checked for accuracy by taking from the curve the voltage necessary in the control light transformer to produce a mirror with 50 per cent transmission and a test run was made. The transmission of the mirror produced was determined by the procedure outlined above and found to be 49.68 per cent. This was considered as evidence of the satisfactory accuracy of the calibration.

The fact that the mirrors transmitted different amounts of light is illustrated in figure 16. The four calibration mirrors and the test mirror were placed in contact with a metal shield in which six, 1/2" holes had been drilled. The mirrors were so placed that each covered a hole, leaving one uncovered. Light from a source, placed above the shield and in such a position that the intensity was the same at each opening, was allowed to pass through the films and affect a sheet of photographic paper placed under the shield.



1. Opening not covered by mirror.
2. Opening covered by mirror of 55.63% transmission.
3. Opening covered by mirror of 49.68% transmission.
4. Opening covered by mirror of 43.64% transmission.
5. Opening covered by mirror of 21.17% transmission.
6. Opening covered by mirror of 7.2% transmission.

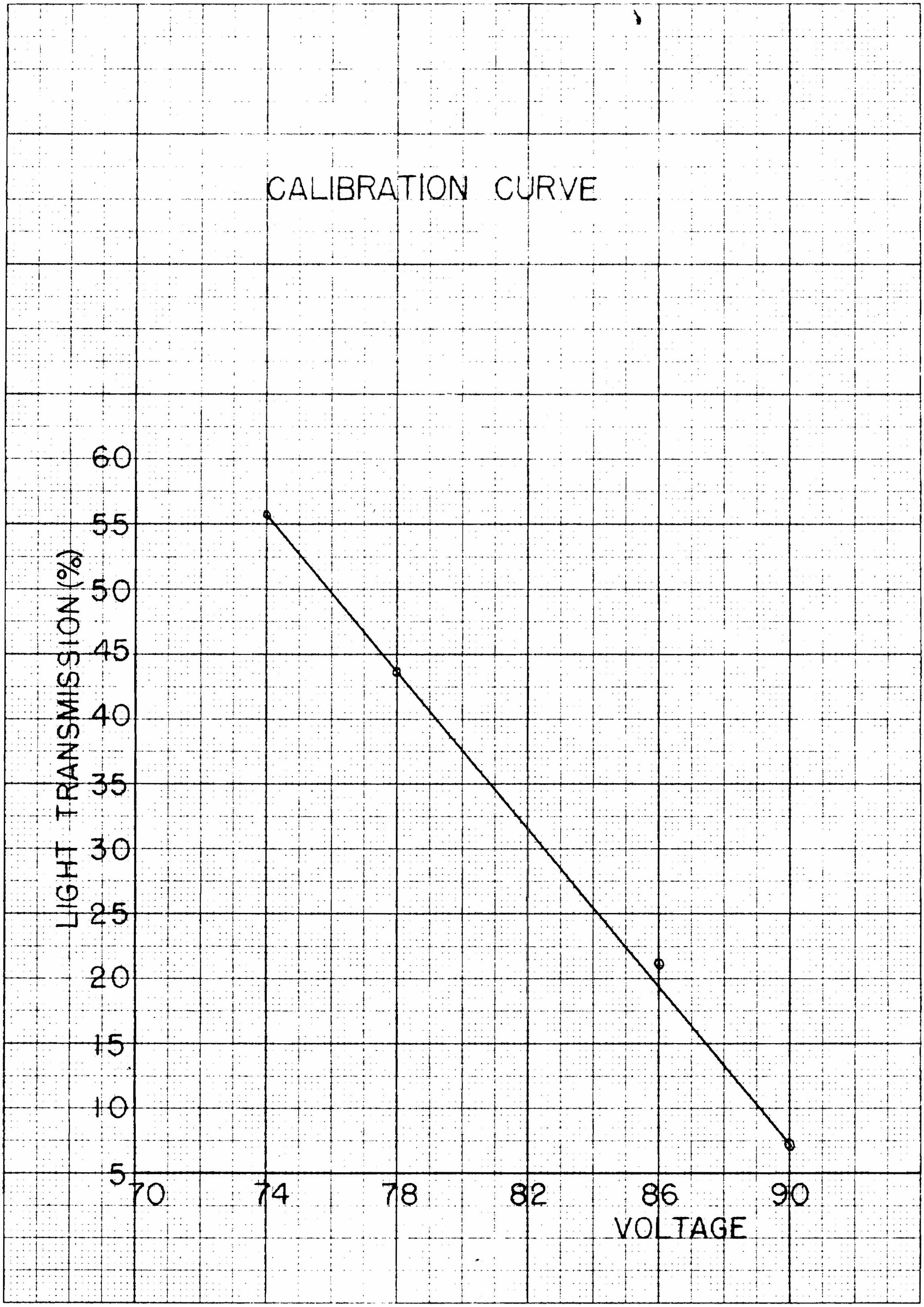
Fig. 16

CALIBRATION MIRRORS								
	74 volts		78 volts		86 volts		90 volts	
	PER CENT TRANSMISSION		PER CENT TRANSMISSION		PER CENT TRANSMISSION		PER CENT TRANSMISSION	
	UPPER	LOWER	UPPER	LOWER	UPPER	LOWER	UPPER	LOWER
	55.5	55.8	42.0	45.0	21.0	20.9	7.0	7.5
	55.0	56.0	42.0	44.2	21.0	21.0	6.8	7.0
	56.0	55.5	43.0	44.6	21.8	21.3	7.1	7.8
Av	55.5	55.76	42.7	44.6	21.27	21.07	6.97	7.43
Av	55.63		43.64		21.17		7.2	

Chart 1

TEST MIRROR		
	75.9 volts	
	PER CENT TRANSMISSION	
	UPPER	LOWER
	50.0	50
	49.0	50
	48.5	50
Av	49.3	50
Av	49.68	

Data for Test Mirror


CALIBRATION CURVE
LIGHT TRANSMISSION (%)
60
55
50
45
40
35
30
25
20
15
10
5
70
74
78
82
86
90
VOLTAGE

Discussion

While the writers are not dissatisfied with the results of the research, they believe that there are several changes that could be effected on the apparatus and the evaporation technique to improve its operation. These changes were not carried out on account of the time and expense involved.

Although the pumping time was not excessively long, it could be improved by increasing the diameter of the pumping channel.[1] This would not only increase the pumping speed but, also, would enable the pump to maintain the high vacuum necessary during the evaporation process. It would also aid in maintaining this high vacuum by placing the pumping channel nearer to the filaments so that the region of lowest pressure would be the active portion of the chamber.

Studs and guide pins should be placed in the control plate end of the evaporation chamber and winged nuts used to secure the plate to the chamber. This would aid in handling of the plate as well as in insuring the correct position of the plate upon reassembling. Also, since the plate and the rubber gasket adhered so strongly to the end of the chamber when the system had been pumped down, some device, such as a slot into which a lever could be inserted for prying the plate loose from the end of the chamber, should be added.

The shutter arrangement should be changed so that the fila-

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[1]Yarwood states that pumping speed is directly proportional to the cube of the diameter. High Vacuum Technique, p.54.

ments are more completely screened with the shutter in the closed position. This would insure a more complete stopage of the molecular rays from the evaporator to the substrate not only during the prefusing operation but also when the deposition was stopped by the shutter. A small window should be placed in the evaporation plate so that the prefusing operation could be observed with the shutter closed.

When the apparatus was used it was necessary to wait until the oil diffusion pump cooled before air could be admitted to the system. Since this involved an unnecessary delay in the procedure, it would be advisable to place a stopcock in the system between the pump and the air inlet to the evaporation chamber so that the pumps could be kept under a vacuum while air was admitted to the rest of the system.

The McLeod gauge does not give an indication of pressure of water vapor or other vapors in the system, nor can instantaneous readings be made, consequently, the writers favor the inclusion of a gauge such as a Pirani gauge to avoid both these disadvantages.

A standard lamp should be substituted for the control light to give a more dependable source of illumination. A collimating lens should be placed in front of the light so that the rays are parallel, and the phototubes placed in a light tight box with baffles so that it receives only the parallel light from the control light. Corresponding values of intensity of the control light and the voltage to the primary of its transformer should be recorded as constants of the instrument so that it can be checked for variation of calibration.

A form should be made for winding the coils to insure that all coils are of equal length and have same number of loops per inch.

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