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An application of cathodic sputtering to the etching of metallographic specimens

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AN APPLICATION OF CATHODIC SPUTTERING TO THE ETCHING OF METALLOGRAPHIC SPECIMENS

BY

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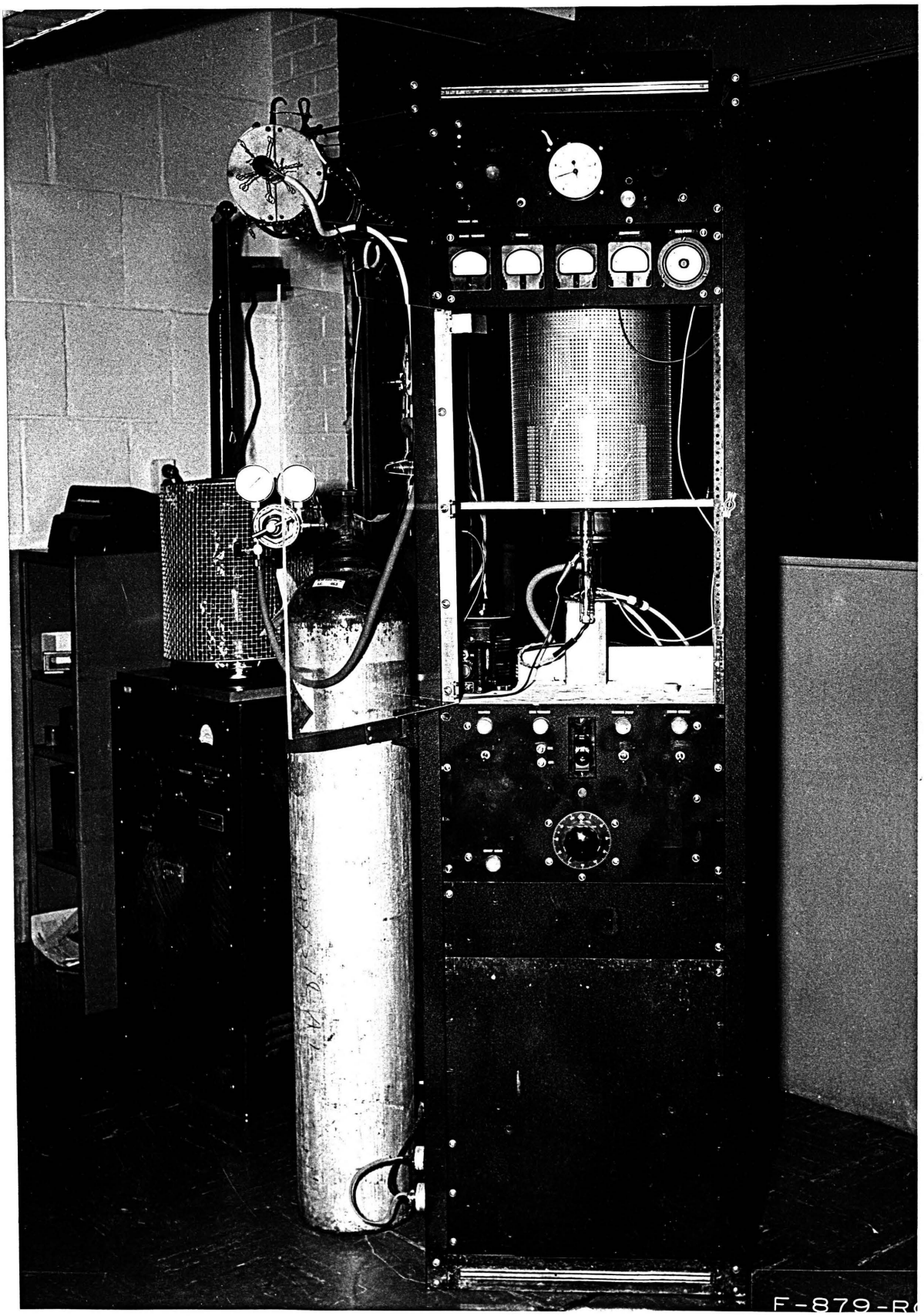
Professor of Physics

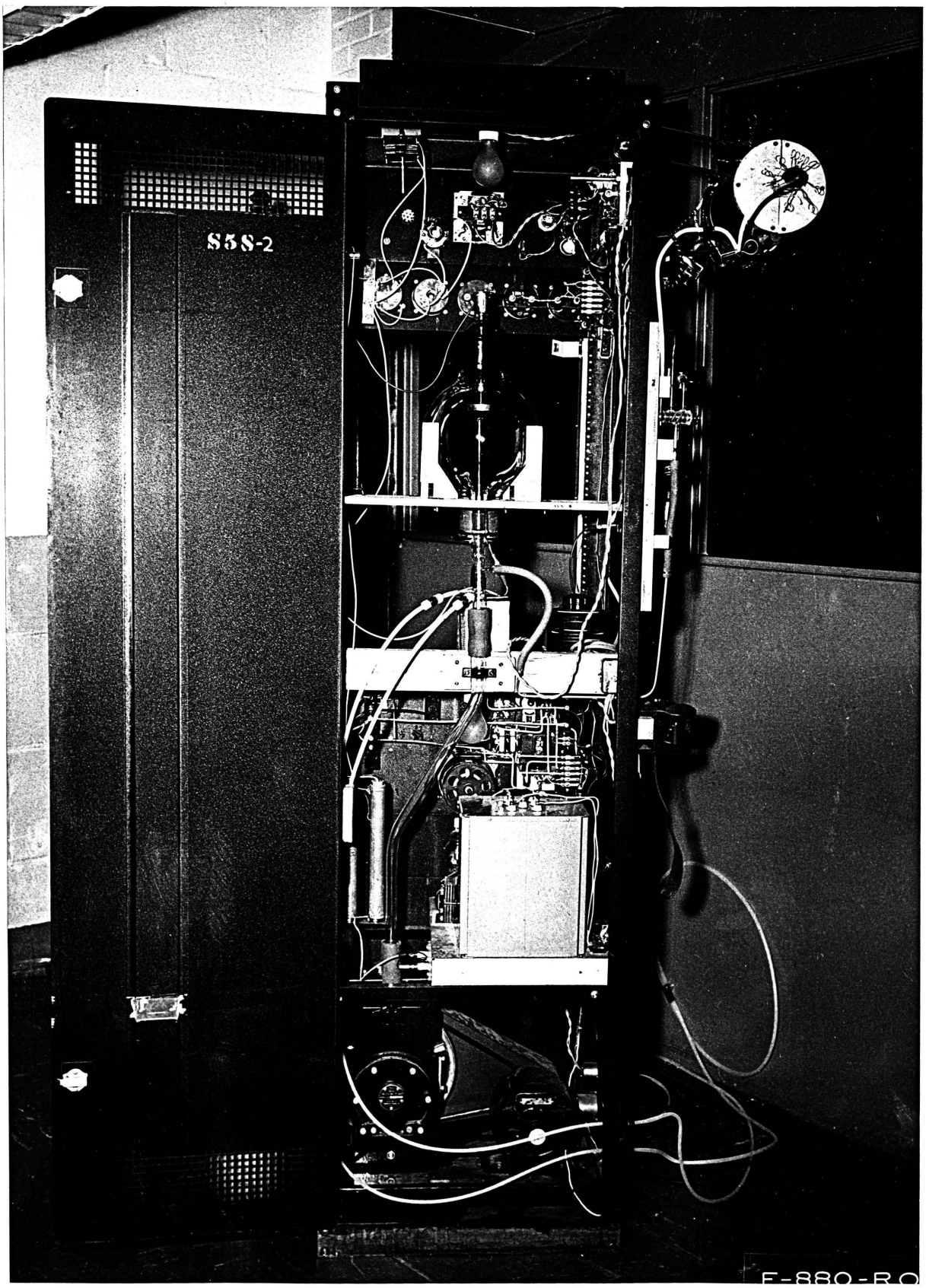
Cathodic Vacuum Etcher

Front View



Cathodic Vacuum Etcher

Rear View



ACKNOWLEDGEMENT

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INTRODUCTION

That branch of Physical Metallurgy known as metallography is largely concerned with the study of the characteristics of metals and alloys and the relationship of these characteristics to its physical and mechanical properties. One of the most important fields of metallography is the study, by means of microscopic examination, of the microstructure of the metal or alloy. This field of study is important because many details of microstructure such as grain size, number and distribution of nonmetallic inclusions, segregation, etc., have considerable influence on the mechanical properties of the metal.

Before being examined microscopically, the surface of the metal to be examined must first be specially prepared to reveal the details of microstructure. The method of preparation of the specimen for examination is relatively simple, although the actual preparation is sometimes quite difficult. The surface of the specimen is first ground flat and is then polished on a series of emery papers, each with decreasing grit size. This process is followed by fine and final polishing on one or more cloth covered lap wheels. These operations, if properly performed, ultimately produce a scratch-free, mirror-like surface. If this surface is now examined microscopically, it will be discovered

that very little, if any, detail of the structure of the metal can be seen.

A thin layer of amorphous metal smeared over the surface by the polishing operation obscures the microstructure. This layer of metal and the disturbed cold-worked material directly below it must be removed before the true structural details of the metal are revealed.

The removal of this layer of disturbed metal is usually by means of chemical or electro-chemical etching. It has been stated recently(1) that etching by cathodic bombardment often gives superior results when compared with chemically or electro-chemically etched specimens, and that it is especially adapted to the problem of etching dissimilar metallic couples. These couples are in general very difficult to etch chemically.

The U. S. Bureau of Mines at Rolla, Missouri has for some years been conducting research on manganese-copper alloys. The study of the physical metallurgy of these alloys -- dissimilar metal couples -- has always been complicated by the difficulty of satisfactorily etching this material. It was felt that by introducing a new etching technique, cathodic bombardment, satisfactory etching might be accomplished. On this basis the following research program was proposed:

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(1) "Metallography." Metal Progress, Vol. 66, p. 174 (1954)

1) To survey the present state of art of cathodic etching, etching by means of cathodic sputtering;

2) To design and construct suitable equipment for the experimental study of etching of manganese copper alloys by means of cathodic sputtering;

3) To investigate the cathodic etching of cast and wrought manganese copper alloys, considering the effects produced by variations in atmosphere, voltage current, and geometry of the etching chamber, and in composition, grain size, orientation, electrical properties, and heat treatment of the alloy specimens;

4) To master the necessary techniques of polishing, etching, replicating and shadowing to present ultimate results of the above investigation in the form of electron and light micrographs of good quality.

REVIEW OF LITERATURE

It can often be observed in a gas discharge tube that has been operated for a long period of time, that a black deposit has formed on the glass near the cathode end of the tube. This deposit consists of metal that has been removed from the cathode. This phenomenon is called sputtering. According to Francis[1] sputtering was first observed in 1852 by Grove, and in 1858 by Plücker.

During the one hundred years since sputtering was first discovered, a great mass of experimental data, much of it contradictory, has been accumulated. We shall first examine some of these experimental results and empirical relationships derived from them before going on to discuss a number of the many theories of sputtering.

It is generally agreed[2] that the cause of sputtering is the bombardment of the cathode by high velocity ions of the gas in the discharge. Generally, there is less sputtering with the gases of lighter atomic weight, being very slight with helium, which apparently tends to penetrate into the metal rather than liberate atoms from it. The rate of sputtering increases less than linearly with the mass of the bombarding ion, except

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(1) Francis, Gordon. "The Glow Discharge at Low Pressure." Handbuch der Physik. Berlin: Springer-Verlag, 1956. Vol. 22, p. 154.

(2) Penning, F. M. Electrical Discharges in Gases. Netherlands: Philips Technical Library, 1957, p. 49.

when chemical action occurs between the ion and the cathode material. In this case very high sputtering rates may be found.

The rate of sputtering depends to a large extent on the metal of which the cathode is made. It is also dependent upon the energy of the ions. An empirical relationship for sputtering rate is sometimes given as $\varphi = C(V-V_o)$, where C and V_o are constants for a given gas and cathode, and V is anode to cathode voltage drop. Francis[1] states that V_o is usually between 100 and 300 volts. This would indicate a threshold energy for sputtering, but he further indicates that V_o is actually 0 for a carefully cleaned surface in a high vacuum. Loeb[2] shows the same relationship, but gives values of V_o of 400 to 600 volts.

The condition of the surface may also have great effect on the rate of sputtering. Wehner[3] states that oxide layers on aluminum, zirconium, titanium, thorium, tantalum, and uranium may cause sputtering to be very slow until the layer of oxide is removed, and then sputtering may proceed rapidly.

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(1) Francis, Gordon, op. cit. p. 154.
(2) Loeb, L. B. Fundamental Processes of Electrical Discharges in Gases. New York: Wiley and Sons, Inc., 1939, p. 599.
(3) Wehner, Gottfried K. "Controlled Sputtering of Metals by Low Energy Hg Ions." Physical Review, Vol. 102, p. 697 (1956).

During sputtering[1] the spectral lines of the cathode material are strongly represented in the spectrum of the negative glow. Cathode material lines may even be predominant in the negative glow, and a copper cathode may, for example, give the negative glow a green color. By a study of the spectral lines of the cathode material,[2] it was shown that a large part, if not all, of the sputtered material was present in the atomic state. Many attempts have also been made to deflect the sputtered particles by electric and magnetic fields, and the result has been the general conclusion that the particles are uncharged.

Attempts have been made to measure the velocity of the sputtered particles as they are removed from the cathode. According to Massey,[3] Berkley and Mason determined this velocity to be 2×10^6 centimeters per second, which corresponds to a temperature of 500,000°C.; however, Baum estimated that silver atoms sputtered by hydrogen had a velocity of 570 meters per second, corresponding to a temperature of about 1,000°, just above the melting point of silver, 961°C.

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(1) Penning, F. M., op. cit., p. 49.
(2) Townes, Charles Hard. "Theory of Cathode Sputtering in Low Voltage Gaseous Discharges." Physical Review, Vol. 65, p. 319 (1944).
(3) Massey, H. S. and Burhop, E. H. S. Electronic and Ionic Impact Phenomena. London: Oxford University Press, 1952, p. 589.

It has usually been assumed[1] that when an alloy cathode is sputtered, the various constituents that make up the alloy will sputter as though they were separate, and that in time the cathode will become more and more concentrated in the slowest sputtering component. Recent work has shown that this may not be true. Hanau,[2] while studying the intensity of the spectral lines from a helium low voltage arc, used aluminum alloy electrodes to reduce sputtering. This alloy consisted of aluminum plus 4% copper, .5% magnesium, and .5% manganese. Spectrographic analysis of the sputtered material showed that it had the same analysis as the original alloy. The sputtering may have been accompanied by general high temperature evaporation, which makes it difficult to interpret the results.

Fisher and Weber[3] used sputtering to remove thin uniform layers of metal from cylindrical specimens in order to study diffusion. Most of the sputtered metal was deposited on thin metal foil which lined the inner surface of the anode. This foil was easily removable, and the relative amount of each component deposited on it could be determined by chemical analysis. They

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(1) Francis, Gordon, op. cit., p. 154.
(2) Hanau, Richard. "Cathode Sputtering in the Abnormal Glow Discharge." Physical Review, Vol. 76, p. 153 (1949).
(3) Fisher, T. F. and Weber, C. E. "Cathodic Sputtering for Micro-diffusion Studies." Journal of Applied Physics, Vol. 23, p. 181 (1952).

found that the sputtered material from two different alloys, #347 stainless steel and 70-30 brass, had the same composition as the original alloy. Since the temperature of the specimen always remained below 325°C., no evaporation could have taken place.

Wehner[1] has performed a series of very interesting experiments in which the cathode was immersed in a mercury plasma formed inside a pool type vacuum arc. With this type of discharge, the gas pressure could be kept low, at one micron of mercury pressure, and the energy of the mercury ions could be maintained below 300 electron volts. High current densities could still be maintained -- as high as ten milliamperes per square centimeter. With this equipment he was able to measure the threshold energies (the minimum ion energy for sputtering to occur) for a large number of substances. He found the threshold energy could be empirically related to: the mass of the ion, the mass of the sputtered atom, the heat of sublimation of the sputtered material, and the velocity of sound in the sputtered material. He was also able to show[2] that at low energy (less than 200 electron volts) ions impinging on the surface at oblique incidence were more efficient at sputtering and also had a lower threshold than normal incidence ions.

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(1) Wehner, Gottfried K., op. cit., p. 696.
(2) Ibid., p. 694.

A series of pictures of deposits sputtered by Wehner from single crystals of certain metals[1] indicate very clearly that sputtering occurs most easily in the direction of closest packing.

Considering the great number of measurements of the rate of sputtering, it is rather surprising how little agreement there is among them. Unfortunately most of the older measurements of sputtering rate[2] were made in a gaseous discharge of pressures of the order of one millimeter of mercury. At these pressures the mean free path of the sputtered atom is small compared with the dimension of the sputtering chamber, and the number of atoms released from the cathode and the number permanently removed from the cathode are quite different because of back diffusion of released atoms to the cathode. Because of back diffusion the apparent sputtering rate is inversely proportional to the gas pressure and to the distance from the cathode to a surface on which the ejected particles can deposit themselves. When the gas pressure becomes low enough that the mean free path of the sputtered particle is on the same order as the dimensions of the apparatus, the rate of sputtering becomes independent of pressure. In spite of the variations of observed sputtering rate with changes in

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(1) Ibid., p. 699.
(2) Massey, H. S. and Burhop, E. H. S., op. cit., p. 579.

pressure and geometry, it might still be expected that the relative rates of sputtering of different materials in the same gases should be the same for all observers. However, this is not the case[1] and is probably because surface conditions of the cathode, which are difficult to control, have a considerable effect on the sputtering rate.

A number of theories involving various processes have been suggested to explain cathode sputtering. It is entirely possible that the actual process of sputtering may actually be a combination of a number of different processes. It was first proposed[2] that sputtering occurred because heating of the cathode by a gas discharge caused expansion of occluded gas which disrupted the surface of the cathode. Since sputtering may take place from very hot solid surfaces and even liquid surfaces which would contain very little occluded gas, this seems very unlikely. Measurements of sputtering rate were made by Kingdon and Langmuir[3] by measuring the electron emission of a thoriated tungsten filament. A mono-atomic layer of thorium was allowed to diffuse to the surface of the tungsten filament.

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(1) Ibid., p. 580.
(2) Francis, Gordon, op. cit., p. 158.
(3) Kingdon, K. H. and Langmuir, Irving. "The Removal of Thorium from the Surface of a Thoriated Tungsten Filament by Positive Ion Bombardment." Physical Review, Vol. 22, p. 148 (1923).

This layer of thorium would cause the electron emission of the filament to increase by a factor of 10^5 over that of the tungsten filament alone. The emission decreases according to known laws as smaller and smaller areas of the surface are covered. By sputtering the filament and measuring its emission from time to time, a very sensitive measurement of the amount of thorium removed could be obtained. Kingdon and Langmuir(1) had expected to find the rate of removal of thorium to be proportional to the amount present. However, the sputtering rate was low at first and gradually increased to a more or less uniform value which it maintained until most of the thorium was gone. They(2) explained these results with a mechanical collision model in which a high velocity ion impinging on the surface will drive the thorium atom which it strikes into the surface and will itself be reflected and fly off. This will result in a large number of depressions of about atomic size on the surface of the filament. After a time a second atom will strike the bottom of one of these depressions and be reflected from the thorium atom there. If this is an elastic impact, the ion can still be energetic enough to knock off one of the surface thorium atoms around the edge of the depression on its way out of the depression. Accordingly,

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(1) Ibid., p. 152.
(2) Ibid., p. 154.

no sputtering can take place until two or more atoms have struck the same point of surface. This accounts for the initial slow sputtering.

According to Francis[1] a theory that has gained wide acceptance is originally due to Von Hippel and Blechschmidt, and was later developed by Townes. According to this group, sputtering is evaporation from small local regions where the impact of ions has produced extremely high temperatures. The calculations and assumptions of Townes[2] show that there should be small areas (a few atomic radii) that will have very high temperatures for short periods of time (10^{-16} seconds). In this short interval of time no other ion would be likely to strike in the same area, and heat radiation should be negligible. Therefore the rate of evaporation of the cathode material can be found from vapor pressure data. It is difficult to assess the accuracy of this theory since the original assumptions limit its application to low voltage discharges in which the rate of sputtering is very small. Also[3] the values for specific heat and thermal conductivity of the cathode material which are needed for the calculation of sputtering rate are generally uncertain or unknown at temperatures near the melting point. The

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(1) Francis, Gordon, op. cit., p. 158.
(2) Townes, Charles Hard, op. cit., p. 326.
(3) Francis, Gordon, op. cit., p. 158.

theory,[1] however, agrees to within the limits of experimental error with the experimental data that have been obtained.

Harrison[2] has applied the mathematical methods of neutron diffusion to the problem of cathode sputtering. This has resulted in an equation for sputtering rate that involves the use of four parameters, only one of which, the mass ratio of the ion to the sputtered atom, is known with any degree of certainty. He shows values of parameters for a number of systems. These parameters were obtained by curve fitting the theory to experimental data.

Keywell[3] used a Phillips ionization gauge discharge as a source for an ion beam of uniform energy. The target was placed in this beam and supported inside a negative shield which returned the secondary electrons to the target. Absolute sputtering ratios were then determined by measuring the weight loss of the target, the true positive ion current, and the time of bombardment. These ratios were obtained for various gas and metal combinations. For silver bombarded with krypton ions, the sputtering ratio varied from 6.4 to 11.5 atoms per ion as the target voltage was raised from

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(1) Townes, Charles Hard, op. cit., p. 326.
(2) Harrison, Don E. "Theory of the Sputtering Process." Physical Review, Vol. 102, p. 1480 (1956).
(3) Keywell, Frank. "Measurements and Collision -- Radiation Damage Theory of High Vacuum Sputtering." Physical Review, Vol. 97, p. 1613 (1955).

1075 to 5160 volts. For the silver argon combination, he noted that the atom per ion ratio is about the same as the number of collisions required to cool an argon ion to 40 electron volts using elementary neutron cooling theory. This method[1] will not apply to the case of sputtering by light ions since the trend of collision numbers and sputtering ratios are inverse with decreasing ion mass.

Keywell[2] went on to consider that cathode sputtering may be an irradiation damage phenomenon. By applying the theory of radiation damage developed by Seitz, Keywell arrived at an involved mathematical formula for the sputtering ratios. This formula gives a fair fit to experimental data and shows that there should be only small temperature dependence for sputtering rate.

There is some objection[3] to the evaporation theory on the grounds that at the high local temperatures supposed to be reached in this process, thermionic emission should be very great, giving values of secondary electron emission much higher than those observed. According to Massey, measurements made by Oliphant and Moon of the energy distribution of secondary electrons from positive ion bombardment found that the electrons

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(1) Ibid., p. 1614.
(2) Ibid., p. 1617.
(3) Massey, H. S. and Burhop, E. H. S., op. cit., p. 589.

had velocities that would be expected from thermionic emission at temperatures of 10,000°C. for aluminum, 30,000°C. for nickel, and 66,000°C. for platinum.

Wehner[1] believes that sputtering by low energy ions (less than 300 electron volts) is connected with a shock wave which travels most effectively along the direction of closest packing from the point of impact of the ion and the surface. This shock wave is eventually reflected and some of the energy returns to the surface where it removes an atom. He concludes that at higher energies the process is probably different.

According to Holland[2] the fact that etch figures could be produced on the surface of polished metal specimens by positive ion bombardment was first discovered by Feitknecht, Baum and Smith about thirty years ago. As a metallurgical technique, this method was ignored until 1949 when McCutcheon and Pahl[3] found that for some purposes the process was superior to chemical etching. The equipment of McCutcheon and Pahl consisted of a converted vacuum evaporation unit. Inside the large bell jar of the evaporation equipment they mounted a separate glass sputtering cell in such a manner that the entire system could be evacuated. The

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(1) Wehner, Gottfried K., op. cit., p. 704.
(2) Holland, L. Vacuum Deposition of Thin Films. New York: Wiley and Sons, Inc., 1956, p. 439.
(3) McCutcheon, Don M. "Cathodic Vacuum Etching of Metals." Journal of Applied Physics, Vol. 20 P. 414 (1949).

discharge was therefore contained in a small isolated cell within the larger bell jar. The cathode to anode distance was about six inches; the anode three inches in diameter. An argon atmosphere of twenty microns pressure was used. A 12,000 volt neon sign transformer was connected directly from anode to cathode. Etching took about 1 to 4 hours to complete. Tests with the equipment on steel forgings showed that the flow lines could be brought out with a sharpness and clarity that exceeded anything that had ever been obtained by chemical etching. Flow lines were also revealed in steel samples that all other metallographic methods had failed to show.

McCutcheon and Pahl[1] later obtained a high voltage DC power supply and using this and the same sputtering chamber, found they could satisfactorily etch a large number of metallic alloys. They found that a part of each bombardment time consisted of an induction period during which nothing at all appeared to happen. It was also noted that an etched pattern could be partially removed simply by reducing the gas pressure and the ion current. In contrast to chemically etched surfaces which exhibit diffuse reflection, cathodically etched surfaces exhibit mostly specular reflection. This makes

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(1) McCutcheon, D. M. and Pahl, W. "Cathodic Vacuum Etching of Metals." Metal Progress, Vol. 56, p. 676 (1949).

little difference when taking microphotos, but requires slightly different technique than is generally employed for production of macrophotos.(1)

Although McCutcheon and Pahl(2) apparently made no attempt to cool the cathode of their sputtering apparatus, most subsequent experimenters have used a cooled cathode. Experiments have shown(3) that between 30% and 85% of the energy dissipated in the cathode fall is liberated as heat at the cathode. Unless the cathode is cooled, its subsequent rise in temperature due to the large amount of heat being dissipated there may cause radical alteration of the structure of the metallographic sample to be etched. Holland(4) suggests that an ungrounded cathode running several thousand volts below ground can be cooled directly with water from the mains if very long rubber hose connections are used, the length of the hose, of course, depending upon the resistivity of the water. In the apparatus constructed by Padden,(5) the cathode cooling is accomplished by mounting the cathode on a large copper rod which extends out of the chamber into a tray of trichlorethylene and dry ice. With this apparatus the temperature of the cathode

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(1) A photograph of gross details.
(2) McCutcheon, D. M. and Pahl, W., op. cit., p. 674.
(3) Loeb, L. B., op. cit., p. 589.
(4) Holland, L., op. cit., p. 420.
(5) Padden, T. R. and Cain, F. M. Jr. "Preparation of Metallographic Specimens by Cathodic Vacuum Etching." Metal Progress, Vol. 66, p. 1-108 (1954).

always remains below 450°C., the melting point of tin.

There seem to be as many different types of equipment for performing cathodic bombardment of metallurgical specimens as there are experimenters in the field. Padden has used a rebuilt vacuum evaporator very similar to the equipment used by McCutcheon and Pahl. Rogers(1) shows a simple compact piece of equipment which consists mostly of a large tube with a ground glass joint at one end.

Bierlein(2) shows a five liter spherical chamber with zirconium cathode and anode, and pictures in another article(3) a remotely operated chamber which consists of three moveable sections of glass tubing with molded neoprene gaskets in between. It is constructed in such a manner that the center section can be discarded after each run since it contains sputtered uranium deposits. Belser(4) has developed a very simple sputtering apparatus using a standard Pyrex pipe reducer as a chamber. Although built to deposit opaque films of nickel and the noble metals, it should

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(1) Rogers, B. A., Manthos, E. J., Carlson, A. J. and Williams, J. T. Etching Metals by Ionic Bombardment. U. S. A. E. C. 1954, p. 7.

(2) Bierlein, T. K. "Investigation on the Microstructure of Uranium." Hanford Atomic Products Operations, Jan., 1955.

(3) Bierlein, T. K., Morgan, J. R. and Mallet, G. R. "The Metallography of Irradiated Uranium." Hanford Atomic Products Operations, May, 1956.

(4) Belser, R. B. and Hicklin, W. H. "Simple, Rapid Sputtering Apparatus." Review of Scientific Instruments, Vol. 27, p. 293 (1956)

be easily adaptable to metallurgical etching.

A great variety of gas pressures have been used in the sputtering chamber by different experimenters. In conjunction with a magnetic field to increase the ionization,[1] pressures as low as one micron have been used. Bierlein uses 75 microns, and 50 microns is recommended by several experimenters.[2,3] Although a number of different gases have been used in the sputtering process, argon appears to be the most popular. A heavier inert gas would sputter more rapidly, and krypton has been used by some experimenters.

There appear to be a number of advantages to cathodic etching. It is said[4] that fine scratches remaining on the specimens after polishing have a tendency to be erased by the bombardment, although Rogers[5] indicated a tendency for scratches not present after polishing to appear on the specimen. He suggested this was an uncovering of flowed-over scratches. The bombarded surfaces[6] exhibit a bright field etch, are free of stains and contaminants, and are suitable for

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(1) Holland, L., op. cit., p. 410.
(2) "Metallography." op. cit., p. 164.
(3) Padden, T. R. and Cain, R. M., Jr., op. cit., p. 1-110.
(4) Holland, L., op. cit., p. 439.
(5) Rogers, B. A., Manthos, E. J., Carlson, A. J. and Williams, J. T., op. cit., p. 15.
(6) Bierlein, T. K., op. cit., p. 12.

examination and photography at all magnification levels, from macroscopic to microscopic as well as by means of electron microscope.

Some experimenters[1,2] have little doubt that there are any, or at least very few, metals and alloys that cannot be etched by cathodic bombardment. In the article by the ASM Committee on Metallography it is stated that the probability of success in etching any particular metallic couple can be estimated from relative sputtering rates given by Strong.[2] If the sputtering rates are too much at variance with each other, the method will not be satisfactory. For example,[3] silver cadmium coupled with steel, and zinc coupled with iron do not etch satisfactorily because of the great differences in sputtering rates; while tolerable differences in rates are to be found in copper nickel alloys, steel with cobalt base alloys, and others. This is a matter of some importance, since dissimilar metal couples are among the most difficult of all metallographic specimens to etch chemically or electrochemically.

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(1) Rogers, B. A., op. cit., p. 15.
(2) Holland, L., op. cit., p. 439.
(3) Strong, John. Procedures in Experimental Physics. New York: Prentice-Hall, Inc., 1953, p. 163.
(4) "Metallography." op. cit., p. 174.

One disadvantage[1] of the method is that the specimen cannot be etched in the small plastic mount in which most metallographic specimens are polished. The specimen could not be efficiently cooled while mounted in the plastic; besides this, the glow discharge decomposes the plastic and contaminates the specimen and apparatus. Another disadvantage[2] is that very little material is removed during etching; therefore, flowed or worked metal may not be completely removed. Electro-polishing and multiple etches with intermediate polishing can minimize this problem.

Cathodic etching when compared to chemical etching is a rather involved and time-consuming process. It will probably never replace chemical etching, except in certain cases where the results may justify the extra time and effort involved in cathodic etching.

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(1) Holland, L., op. cit., p. 439.
(2) Bierlein, T. K., Morgan, J. R. and Mallet, G. R., op. cit., p. 11.

EQUIPMENT AND ALTERATIONS

The etching equipment is built in a large relay rack. This rack has mounted in it and on it all of the equipment necessary to perform cathodic bombardment etching. Within the rack is the etching chamber, the power supply, vacuum pump, pirani gauge power supply, and associated control circuits. Mounted on the outside of the rack is a gas control system, a small tube furnace to purify the argon, and a tank of argon which is strapped to the side of the cabinet.

The etching chamber itself is a standard five liter, three neck reaction flask with an extra ground glass joint placed at the opposite end from the other joints.(1) The two side joints at one end are small, and one is used for the vacuum line, the other for vacuum gauge. The two middle joints at opposing ends are both large enough to admit a two inch anode or cathode. At the time of planning, this was felt to be necessary in case large specimens were to be used. It was also thought that by mounting the cathode and anode in identical ground glass joints they could be exchanged end for end for experimental purposes.

A water cooled cathode was decided upon, and a diagram of this device is shown in Fig. 3. It was

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(1) Fig. 7.

constructed from three-quarter inch brass tubing with copper tubes for inlet and outlet water flow. A threaded copper plug was silver soldered into one end. A one or two inch aluminum cap threaded to match the copper plug at one end of the brass tube formed the specimen rest. The small copper tube shown in the diagram was originally intended to admit the argon atmosphere to the etching chamber. The cathode was mounted in the ground glass joint as shown in Fig. 4. A small brass ring was soldered to the upper end of the cathode to center it in the tubulation on the ground glass joint. A larger brass piece with a shoulder was soldered to the lower end of the cathode. This piece made a snug fit with the tubulation on the lower end of the joint, and was sealed to the glass with vacuum wax. From this diagram it can be seen that the small copper tube enters and leaves the brass tube on opposite sides of the vacuum seal, and consequently can be used to admit gas to the chamber.

The construction of the anode is shown is Fig. 6. One end of the ground glass joint was closed with a glass to tungsten seal. Both ends of the tungsten wire were beaded with nickel, and to the inner end a piece of copper braid was welded. A polystyrene plug was waxed into the other end of the ground glass joint. This plug had several ports to allow the joint to be pumped out, and was drilled and threaded in the center for a

quarter inch threaded rod, to which the aluminum anode was attached. This threaded rod allowed the anode to cathode distance to be easily adjusted. Inside the joint a small strip spring fastened to the plug made constant contact with the brass rod. This spring was in turn attached to the copper braid, thereby making electrical contact to the anode.

It was later found that when operating the chamber for long periods of time, the heat generated in the chamber by the gas discharge softened the wax seal that held the anode in place, and it was feared that the anode would fall out of the joint. It was also felt that conditions within the chamber might cause decomposition of the polystyrene and/or wax with subsequent contamination of the system and specimen. Therefore the polystyrene plug was replaced with an aluminum one of similar design which was held in place by ordinary toggle bolts.

The etching chamber was supported by a plywood shelf inside of the relay rack. This shelf had a hole cut in it to allow the lower three ground glass joints on the chamber to pass through it. Four felt-padded adjustable supports were attached to the upper side of the shelf, and served to support the spherical etching chamber. Below the support shelf of the vacuum chamber was a second plywood shelf that formed a work table.

This shelf did not extend all the way to the back of the relay rack in order to allow ventilation of the power supply below it. Also this allowed the back side of the shelf to be used as a support for the vacuum line extending from the pump below to the chamber above. To allow access to the chamber, a large lucite door extending from the lower shelf all the way up to the top of the etching chamber was installed on the front of the relay rack. Below the lower wooden shelf was a steel angle-iron support which holds the power supply in place.

Diagrams of the power supply and control circuit are shown in Fig. 1. The power supply was a standard bridge rectifier circuit capable of supplying 5,000 volts at 150 milliamperes of unfiltered full wave rectified DC. A 100,000 ohm resistor in series with the chamber limited the maximum bombardment current to 50 milliamperes. The power supply was quite heavy, and although it could probably have been fastened to an ordinary rack panel, it would have been almost unmanageable. By mounting it on angle iron shelf one person could easily remove it from the cabinet for servicing.

A great deal of difficulty with high voltage breakdown was encountered in the construction of the power supply. These problems were finally solved by mounting all of the tube sockets on sixteenth inch

lucite sheet and by discarding all of the commercial feed through insulators that had been purchased and constructing more suitable insulators from polystyrene rod. After this was done, no more difficulties were experienced from the power supply, although it was thought for some time that the power supply still had a high voltage short in it; this was eventually traced to the high voltage end of the 100,000 ohm dropping resistor which can be seen fastened to the side of the cabinet in the picture of the interior. An insulator made from a piece of Pyrex tubing and a small sheet of mica eliminated this problem.

The control circuits for the power supply were all mounted on a standard relay rack panel. This circuitry had several functions. A variac was used to control the power supply output voltage by regulating the primary voltage of the high voltage transformer. A thermal delay relay made it impossible to turn on the high voltage until the filaments of the mercury vapor rectifier tubes were sufficiently heated. Another relay circuit cut off the high voltage in case one of the inter lock switches should be opened or in case the power failed; and even if the power returned, this circuit made it necessary to turn the high voltage back on.

Directly below the power supply in the relay rack was a Cenco vacuum pump, and in the upper right hand

corner of the relay rack may be found the pirani gauge circuit. The pirani gauge and vacuum pump were both controlled by switches on the same panel that controlled the power supply. The pirani gauge(1) had a standard circuit except for the eleven ohm resistors in the two arms of the bridge. When it was built, no eleven ohm resistors were available, so these were specially wound with constantan wire for the purpose. It had been the author's original intention to calibrate this gauge against a McLeod gauge, but this has never been done, and it has only been roughly calibrated against a Hastings thermocouple vacuum gauge.

On the outside of the cabinet on the left hand side is found the gas control system.(2) This system allows argon to be admitted into the chamber either through a flushing line or through a small variable leak. When originally designed, there were no provisions in this system for the drying tube and the tube furnace. These were added later when persistent stains developed on the specimens and it was thought that possibly water vapor and/or oxygen contained in the argon might be affecting the specimens. The drying tube contained activated alumina and the tube furnace contained titanium turnings and was operated at 850°C. to remove any oxygen that may have been present as an impurity

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(1) Fig. 2.
(2) Fig. 8.

in the argon.

The leak valve was an ordinary rubber hose clamp which had been altered by replacing the original threaded piece with one with finer threads, and by placing a large knob on the end to secure finer control. A number of different hoses had been tried in this arrangement to secure a slow, controlable leak. Several sizes of rubber tubing, tygon tubing, and combinations of these tubings had been tried, but the most successful thing to date has been a small piece of polyethylene tubing which was pre-flattened by heating in boiling water and crushing in a hand vice before being placed between the hose clamp. While it still leaves a great deal to be desired, this valve does perform fairly successfully.

An arrangement was also made on the gas inlet system for admitting the atmosphere to the chamber. However, this was seldom used because the chamber was usually brought up to atmospheric pressure by the admission of argon. When admitting argon to the chamber in this manner, it was quite easy to increase the pressure in the chamber above atmospheric, and would even be possible to blow the ground glass joints completely out of the chamber. To prevent this from happening, a small pop-off valve made of brass tubing and a turned-down rubber stopper were inserted in the

line. As soon as atmospheric pressure was exceeded in the chamber, this valve popped open and prevented any further rise in pressure.

The original design of the anode with the exception of the one previously noted change has proved very successful. The cathode design, on the other hand, has caused quite a lot of trouble, and has been changed a number of times. When the chamber was first put into operation, it was found that in spite of the small brass ring at the upper end of the cathode which presumably should have acted as an electro static shield, a discharge took place inside of the ground glass joint on the lower part of the cathode. It was eventually concluded that this discharge was actually taking place through the gas inlet line. When the system of gas admission was changed, and the gas admitted to the chamber through a long glass tube entering through the vacuum line and extending up into the chamber, the discharge condition within the lower ground glass joint was completely eliminated.

Another problem was the wax vacuum seal at the lower end of the cathode. The wax joint would invariably be broken by using the brass piece on the lower end of the cathode as a handle to twist the ground glass joint when removing it. This seal was eliminated entirely by means of a piece of large diameter vacuum hose which fit the glass tubulation and the brass

shoulder below it.

In early operation of the chamber it was found that if the specimen was removed immediately after bombardment it would be very warm, possibly even too hot to handle. Since the specimen was just resting on top of the cathode, held there only by force of gravity, it was felt that clamping it in place would insure better thermal contact and more efficient heat transfer. To effect this clamping, a quarter inch hole was drilled in the end of the one inch aluminum cathode, as is shown in Fig. 5. The specimen could then be placed in direct contact with the threaded copper plug on the end of the cathode and the aluminum cap screwed down over it, clamping it to the copper, bombardment taking place through the quarter inch hole in the aluminum cap. This also resulted in several other benefits: it became much easier to remove or place a specimen in the chamber because it no longer had to be precariously balanced on the end of the cathode; and also, since the whole specimen was no longer bombarded, it allowed direct comparison of the specimen as polished with the specimen after bombardment.

Another cathode problem which did not appear to have any simple solution was the deposition of sputtered metal on the glass tubulation at the upper end of the cathode. This deposit occurred on both sides of the

glass, although generally heavier on the inside. When the deposit had become sufficiently thick, it would become conductive, and since the small brass ring connected it to the cathode, the deposited metal would itself begin to act as a cathode. The metal film was backed only by glass, a very poor conductor of heat, and hot spots would develop on it with small areas rapidly heated to incandescence. The current through the chamber would then increase because of thermionic emission, and the hot spot would jump from point to point on the surface of the metal film. This condition apparently resulted in a large amount of metal ions being present in the chamber. The discharge would take on a deep green color, probably from the ionized copper. This condition always resulted in deposition of a film on the specimen, probably evaporated metal; and once after this had happened it was noticed that a large amount of aluminum had been sputtered from the cathode end piece. This may possibly be attributed to the presence of heavy metal ions in the discharge.

Large greased ground glass joints sealing an evacuated system may become very difficult to remove. Both the anode and the cathode joint have given some trouble in this respect, although the cathode joint has been the greatest offender. The cathode joint may become rather warm during operation of the chamber. This tends to soften the vacuum grease in the joint,

and the atmospheric pressure pushing on the outside of the joint forces this grease up into the chamber. After a period of time the joint itself may become almost completely depleted of grease. When this occurs, the joint can usually only be removed after prolonged heating with a hand torch. It was noticed that every time the cathode joint was re-greased stains would appear on the specimens for a number of runs, even though the bombardment was carried out under conditions that had previously been non-staining. These stains were apparently caused by decomposition of the vacuum grease by the cathodic bombardment.

Because of all these difficulties the cathode was eventually redesigned as shown in Fig. 5. The ground glass joint was completely eliminated, and a rubber gasket was relied upon to seal the brass plate to the lower end of the chamber. On the upper portion of this plate was a brass plug that fit snugly into the ground glass joint. This served both to center the cathode and also to protect the rubber gasket material from the bombardment. A thin aluminum cylinder was placed concentric with the brass tube of the cathode, and the lower portion of this aluminum cylinder was connected to the flat aluminum plate parallel to the brass plate which sealed the chamber. This aluminum piece was insulated from the cathode by glass rods. A small commercial glass to metal seal passed through the brass

plate, as shown in the diagram. A wire passing through this seal was connected to the previously mentioned aluminum cylinder. This cylinder was then grounded and became a second anode. When the pressure in the chamber was reduced below some certain value, the distance between the aluminum piece and the brass cathode became less than the cathode dark space. When this occurred, the discharge was suppressed in the region between the aluminum and brass cylinders, and took place only at the upper end of the cathode.(1)

During the various etching experiments that have been performed with the equipment, staining of the specimens has been a persistent problem. Some of these stains were unusually colored, and in at least one case the grain structure was revealed by different colored stains on adjacent grains. This staining was at first attributed to contaminants such as oxygen and water vapor in the argon. To eliminate these, a drying tube to remove the water vapor and a tube furnace filled with titanium turnings and operated at 850°C. to remove the oxygen were both installed in the gas system.(2) The furnace at first resulted in far more contamination than it cured, for unfortunately the titanium turnings that were placed in it contained a certain quantity of oil. This oil when heated gave off a dense white smoke

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(1) Holland, L., op. cit., p. 420.
(2) Fig. 8.

which completely filled the chamber and vacuum system on several occasions. It was finally necessary to disassemble the furnace and wash the turnings in carbon tetrachloride and acetone to remove the oil before replacing them within the tube. After this treatment, the furnace presumably operated in its intended manner.

Purification of the gas tended to reduce the staining but by no means eliminated it. Clamping of the specimen to the cathode to insure good thermal contact had a great effect on reducing the staining. And finally, replacing the cathode ground glass joint with the brass plate and rubber gasket, thus removing any vacuum grease from the system, reduced staining to the point where it was no longer a serious problem. It is indeed probable that the gas purification system is not needed, since specimen bombardment can be carried out in an unpurified air atmosphere with no staining taking place.

PROCEDURE AND RESULTS

The procedure for operation of the etching equipment has been as follows: The main power switch in the center of the control panel is turned on. Along with this, the power supply filaments and the pirani gauge are also turned on. A polished specimen is clamped to the end of the cathode and the cathode is put in place within the chamber. A piece of four-by-four cut for the purpose is placed underneath the cathode to hold it in the chamber. This block is cut so that it allows about an eighth of an inch space between the end of the glass chamber and the rubber gasket which seals it. If the tube furnace has already been heated, the flushing line is now opened, and the chamber is flushed with argon for about thirty seconds. While gas is still entering the chamber, the cathode is raised up to seal against the bottom of the chamber, and the vacuum pump is turned on. After this, the valve to the flushing line may be closed. The chamber is then allowed to pump down to as low a pressure as can be obtained. While the chamber is pumping down, the water line to the cathode is opened. This should not be done until the chamber is already partially evacuated or else water will condense on the surface of the specimen.

After the chamber has pumped down, the gas inlet valve is turned to the slow leak position; and after about a minute, which is required to pump out the gas

trapped in the valving system, the leak valve may be adjusted to maintain a suitable pressure in the chamber. If the front and back doors are now shut so that their interlock switches are closed, and forty-five seconds have elapsed since the heater switch was first turned on, the ready light on the power control panel should be lit. This indicates that the high voltage may now be turned on. The timing clock which runs only while the high voltage is on should be set to zero, and the variac that controls the power supply voltage turned to zero. The high voltage is then turned on and the variac control slowly advanced until the proper voltage drop or current through the chamber is attained. Since the pressure in the chamber, the voltage drop across the chamber, and the current through the chamber are all interdependent quantities, it will be necessary to adjust both the voltage control and the pressure control to obtain a particular set of conditions of current and voltage drop in the chamber. It is usually necessary to adjust the leak valve from time to time during the etching process in order to maintain constant conditions in the chamber.

After a suitable period of time has elapsed, the high voltage is turned off and about a minute to a minute and a half allowed for the specimen to cool to room temperature before turning off the water to the cathode. After the water is shut off, the vacuum

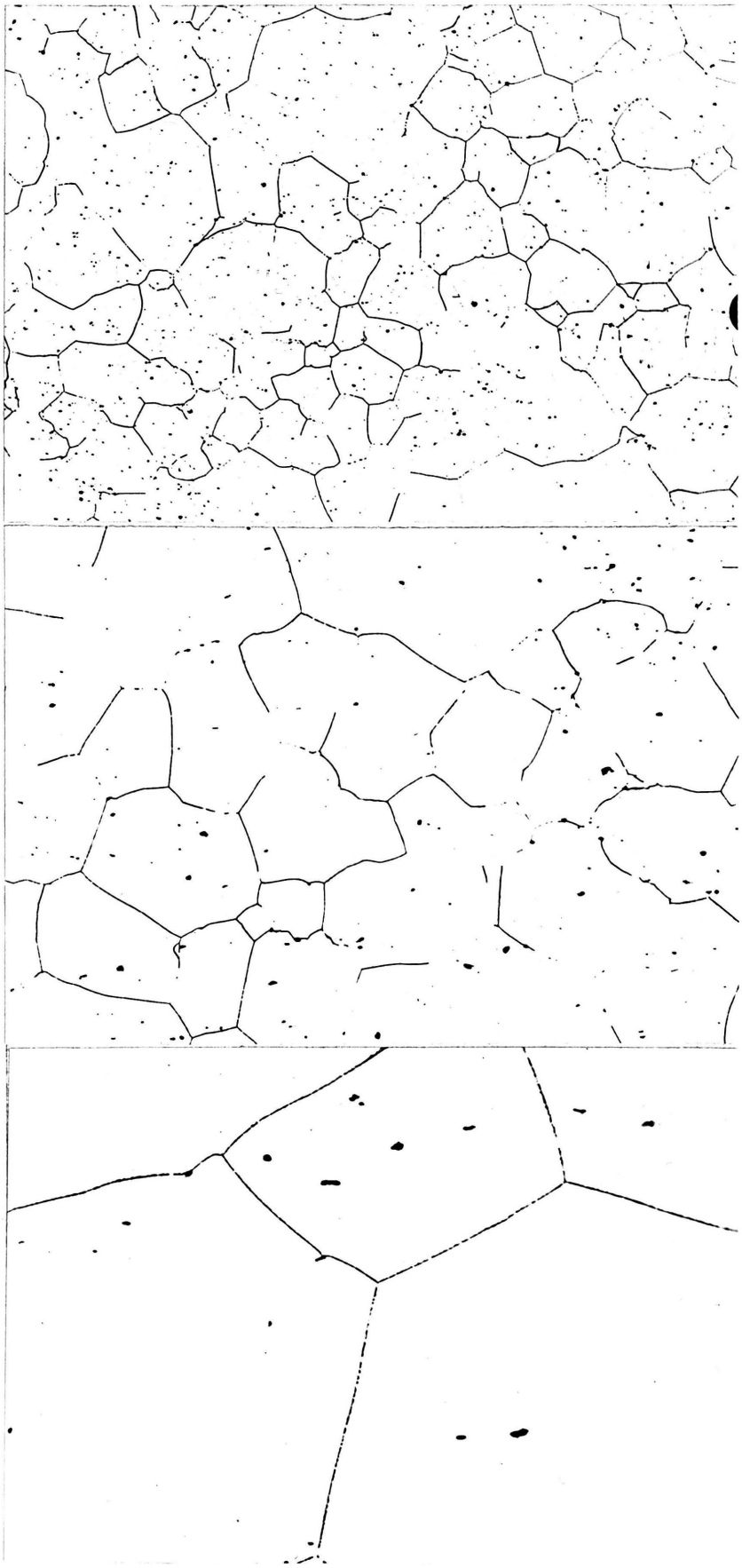
pump is turned off and the gas valve is turned to the flushing line. When the pressure in the chamber has returned to atmospheric, cathode will drop loose onto the wooden block. The block may then be removed and the cathode removed from the chamber. If the proper time was waited before turning off the water, the specimen should be at room temperature and can be removed from the cathode for examination. If too little time was allowed, the specimen may still be hot enough to oxidize when it is brought into the air; or if too much time was allowed, water may condense on its surface.

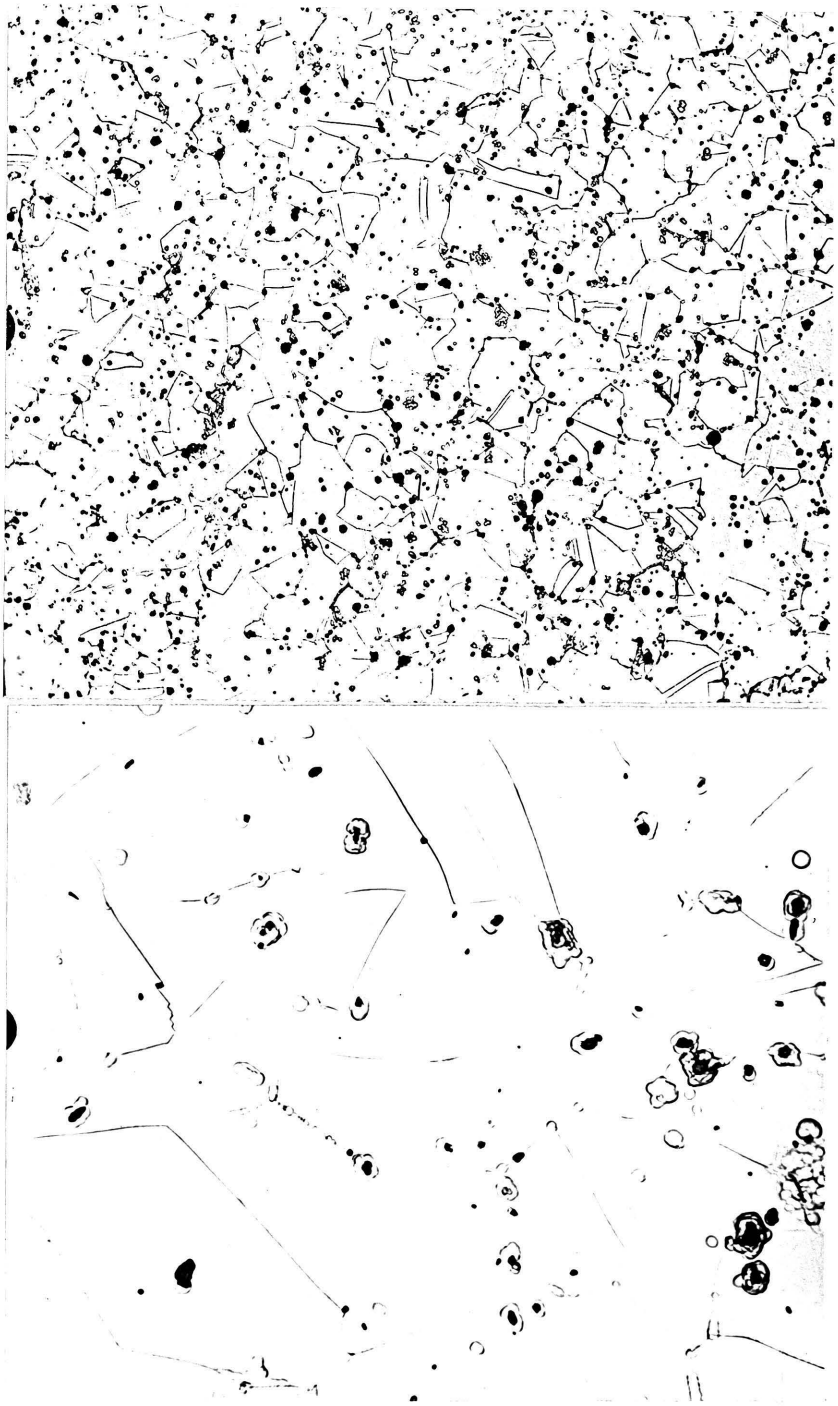
The etching procedure has been tried on a number of manganese copper alloys, and as yet has met with very little success. Some of the earlier results obtained before most of the staining was eliminated are not at all reliable, for rather than indicating etching, they probably show preferential staining at the grain boundaries. In some cases when the bombarded portion of the specimen had shown an etch figure, the polished portions of the specimen that had not been bombarded did also. This effect, of course, could not be noticed until the specimens were clamped to the cathode and only a limited area of the specimen exposed to bombardment.

Two specimens have apparently been etched satisfactorily, and microphotographs of these are shown. The first specimen, shown on page 39, was

39% Cu, 61% Mn and was solution treated. This specimen was polished on grades 0 through 4/0 paper, then with 600 mesh silicon carbide on a canvas wheel followed by a final polish with Linde A on metcloth. It was clamped to the cathode and bombarded for 800 seconds in an argon atmosphere. The pressure in the chamber was 25 microns of mercury, the anode to cathode voltage drop 2.5 kilovolts, and the current 29 milliamperes. A one inch cathode and a two inch anode were used, and the spacing between them was three inches. There was no staining and only the bombarded portion of the specimen was etched. The discontinuous grain boundaries should be noted; these occur with chemical etching also. The three pictures are at magnifications of 100x, 250x and 500x.

The second specimen, shown on page 40, was 75% Mn, 25% Cu and was also solution treated. It was polished according to the above noted procedure. It was clamped to the cathode and bombarded for 3000 seconds in an argon atmosphere. The pressure was 19 microns of mercury, the voltage drop 3 kilovolts, and the current 23 milliamperes. A one inch cathode and two inch anode, with three inch spacing, were used as before. Again there was no staining, and only the bombarded portion of the specimen showed an etch. The two pictures are at magnifications of 100x and 500x.





The author has never attempted to repolish and re-etch the first of these specimens to see if the results could be reproduced. However, he has tried several times to re-etch the second specimen with absolutely no success. This specimen has been bombarded for periods ranging from 30 to 90 minutes. After the 90 minute bombardment period, the bombarded surface had taken on a slightly wavey texture, but no grain boundaries were visible. A great number of pits or possibly pinnacles appeared on the surface, and when the bombarded surface was viewed by oblique light, it seemed to be covered with a great many very small mirrors.

CONCLUSIONS

The choice of chamber design for this experimental cathodic etcher was not a fortuitous one. The inclusion of large ground glass joints in the system, which at the time of design was thought to be a desirable convenience, has actually turned out to be a definite disadvantage. A chamber patterned after the one shown by Belser[1] and adopted to metallographic etching would probably be far more convenient than the one adopted by this author.

Vacuum grease contamination of the inside of the etching chamber definitely contributes to the staining of the specimen, and is one more reason why ground glass joints should be eliminated, if possible.

During bombardment in the chamber, specimens become rather hot; exactly how hot is not known, but could probably be determined by means of bombarding a series of low melting point alloys. This temperature may be low enough that it will not have any deleterious effects on the specimens. However, if it is too high, it could completely alter the microstructure of the bombarded specimen, thus giving an erroneous view of the actual microstructure of the material. More efficient cooling could be obtained simply by increasing the rate of water flow through the cathode, or by

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(1) Belser, R. B. and Hicklin, W. H., op. cit., p. 293.

cooling the water before it enters the cathode.

The inclusion of a vacuum gauge in a device of this nature is probably an unnecessary luxury, since the pressure in the chamber can be fairly accurately estimated from the condition of the glow discharge, the voltage drop across the chamber, and the current through the chamber. In actual operation of the etching chamber the pressure in the chamber is used to control the current and voltage drop across the chamber.

A more accurate and reliable gas leak should be obtained for the operation of this etching device. An accurately made needle valve should be far more satisfactory than the hose pinch clamp now used.

One of the experimental variables that has not had very good control during these experiments is the state of the polished surface of the specimen. This is not because the specimens were polished by someone else, but rather because they were polished by the author. The polishing of metallographic specimens is an art, and requires techniques that must be learned over a period of time. At best it is a very time-consuming process. The author is well aware that the surfaces of some of the specimens were probably severely distorted through the application of too much pressure during the polishing procedure, in the vain attempt to either speed up the process or to remove scratches from the surface that should have been removed during earlier

grinding operations.

With the meagre results so far obtained, no conclusions can as yet be drawn about the applicability of cathodic ion bombardment to the etching of manganese copper alloys. This applicability should be determined by preparing a series of specimens, each of different composition or heat treatment or both. This series would consist of three or more specimens of each composition-heat treatment. All of the specimens should be polished and each one bombarded. The bombardment condition should be changed for each specimen of the same kind. The author had hoped to perform a study of this type, but because of difficulties with the equipment, which have only recently been solved, this has not been possible.

SUMMARY

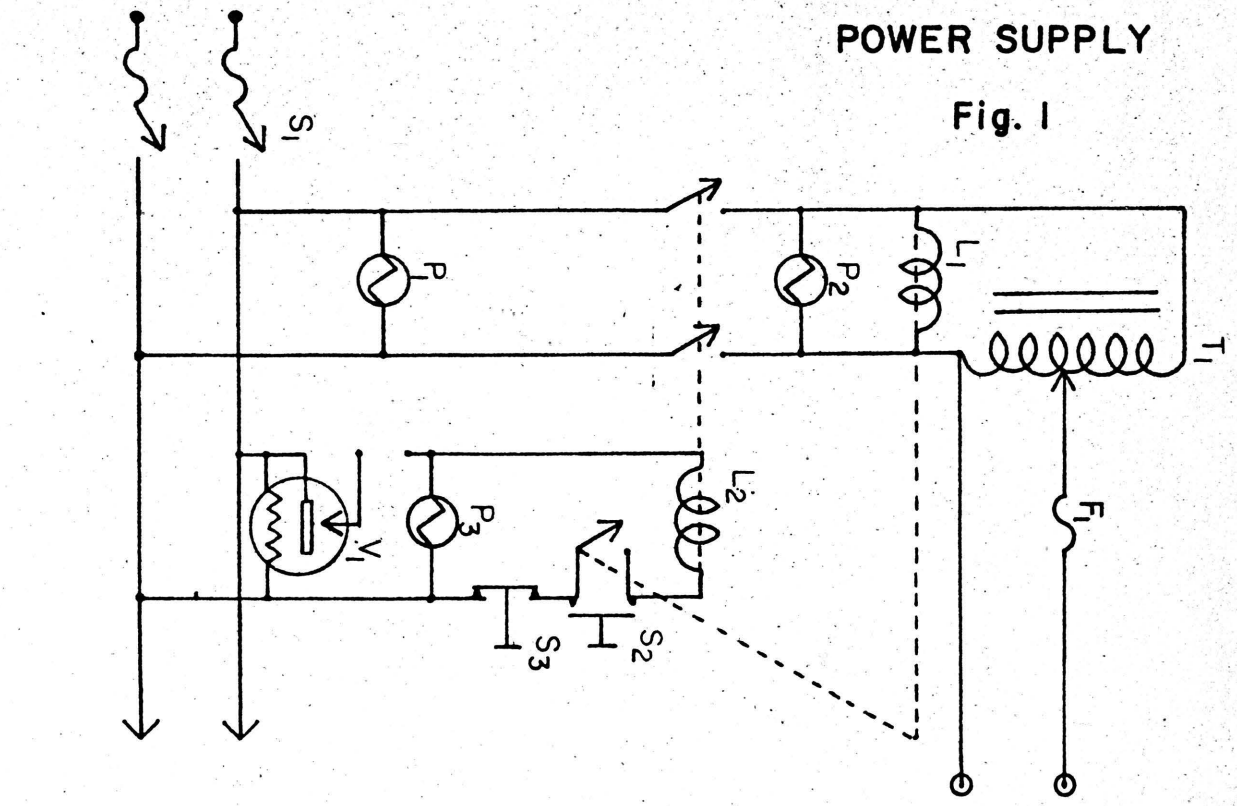
Apparatus has been constructed for an experimental study of the applicability of cathodic ion bombardment to the problem of metallographic etching of manganese copper alloys. This equipment consists of a gas control and purification system, a high voltage DC power supply, a vacuum system including a pirani vacuum gauge, and an etching chamber with an adjustable aluminum anode and a water cooled cathode.

The equipment in its present state has evolved through a great number of changes made in the original design. The cathode has been rebuilt a number of times and was finally constructed in such a manner as to eliminate ground glass joints entirely. This has completely eliminated the problem of the cathode joint sticking, and has also largely eliminated the problem of stained specimens.

The equipment is being used to make a study of the cathodic etching of manganese copper alloys. As yet, not enough data has been obtained, and results are inconclusive as to the applicability of this method to the etching of these particular alloys.

POWER SUPPLY

Fig. 1



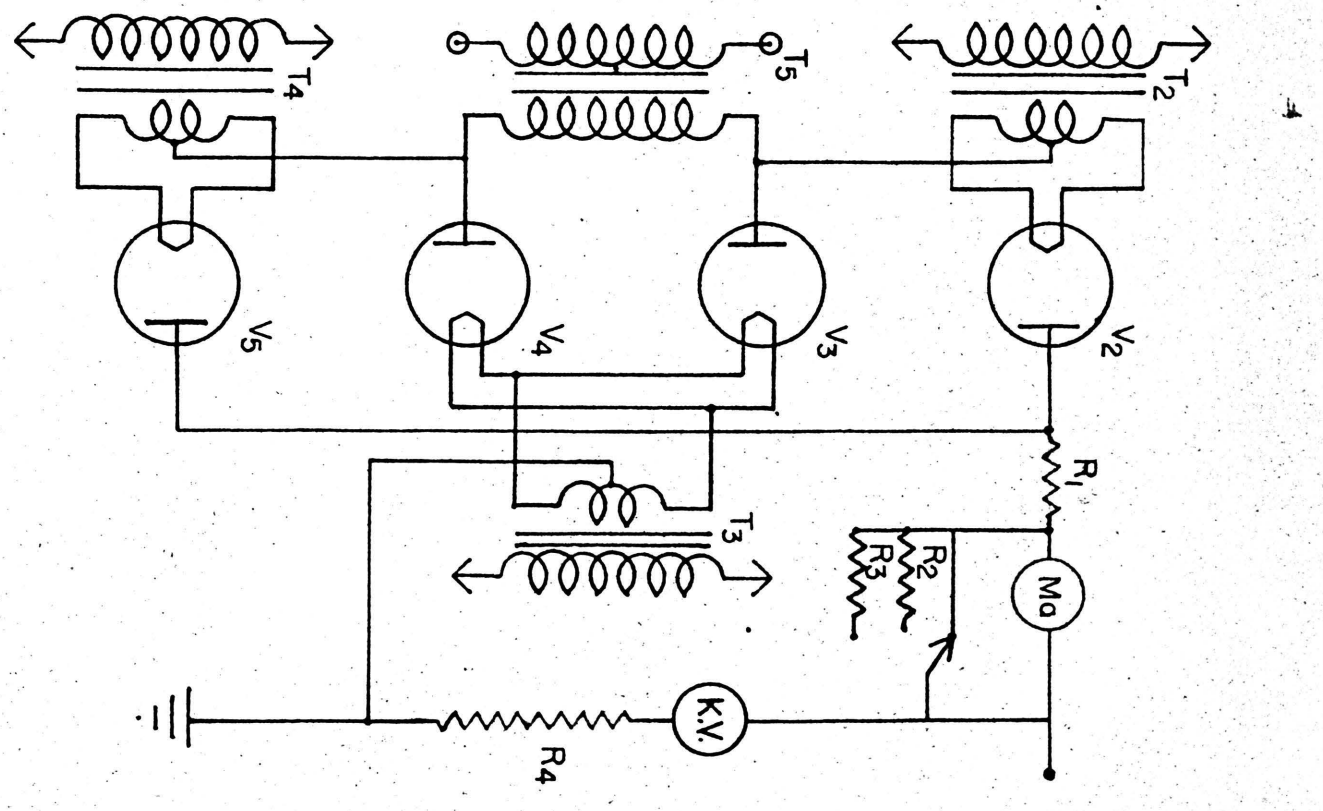
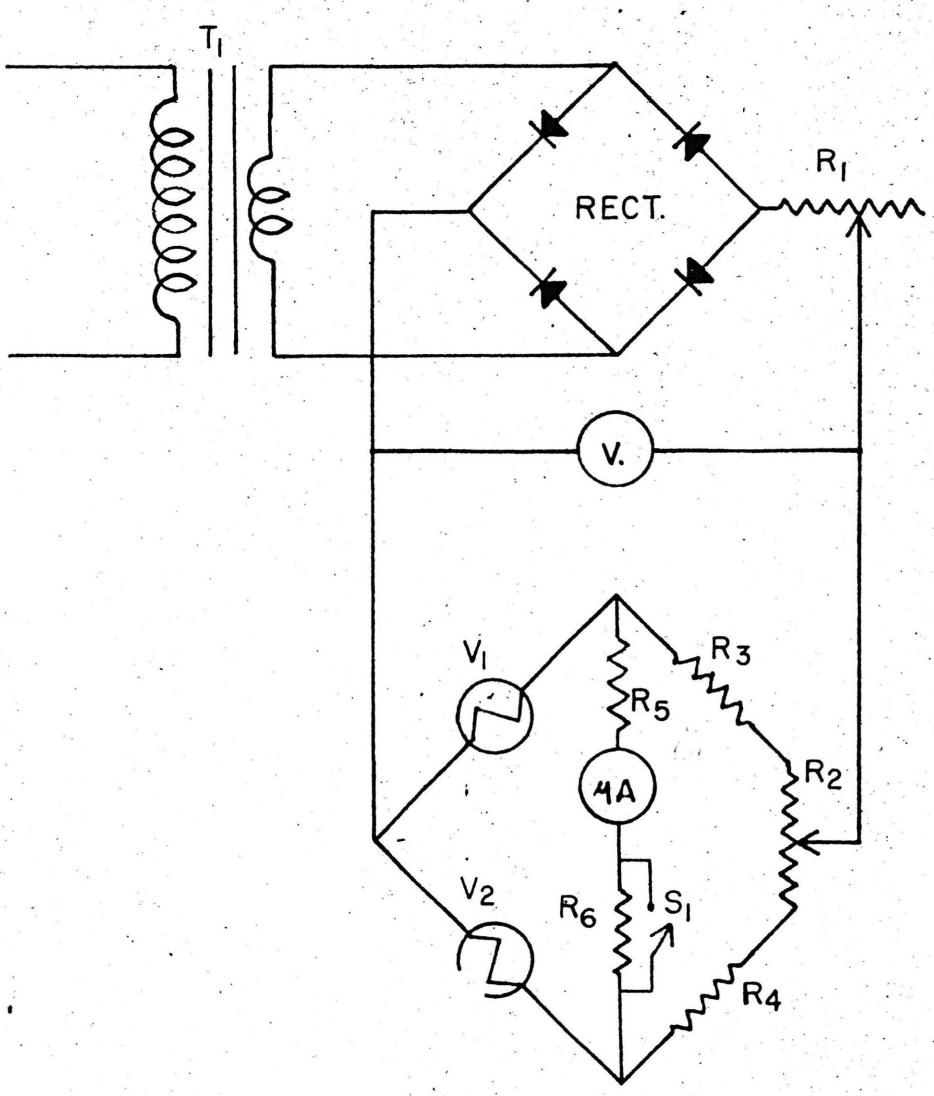


FIGURE 1

T_1	Variable Transformer 115V Pri. 0-115V 7.5 amp sec.
T_2 T_3 T_4	Filament Transformers 115V Pri 2.5VCT 10 amp
T_5	High voltage transformer 115V Pri. 3000, 2500, 0, 2500, 3000 150 ma
L_1	SPST Relay 115 vac coil
L_2	SPDT Relay 10 amp contacts 115 vac coil
V_1	Thermal delay relay normally open. 45 sec. delay
V_2 V_3 V_4 V_5	Mercury vapor rectifiers 866A/866
P_1	115V pilot lamp indicates filaments are on
P_2	115V pilot lamp indicates high voltage is on
P_3	115V pilot lamp indicates high voltage can be turned on
S_1	SPDT switch 10 amp
S_2	SPST normally open momentary push button switch
S_3	SPST normally closed momentary push button switch
R_1	1,000,000Ω 400 watt wire wound resistor
R_2 R_3	Shunts hand wound from constantan wire to make meter read 50 and 100 ma
R_4	5 megΩ
F_1	Fuse 8 amp
Ma	0-10 ma 3" meter
KU	0-1 ma 3" meter



PIRANI GAUGE

Fig. 2

FIGURE 2

T_1	Transformer 115V Pri. 15V sec.
Rect	Selenium bridge rectifier
R_1	50Ω Pot.
R_2	2Ω 2 Watt wire wound Pot.
R_3 R_4	11.1Ω Hand wound with constantan wire
R_5	160Ω resistor
R_6	5000Ω Pot.
S_1	SPST normally open momentary contact
UA	0-200 UA meter
V	0-5 volt meter
V_1 V_2	Standard pirani gauge tube

To calibrate evacuate V_2 to low pressure (less than one micron); adjust R_1 until the voltmeter reads on the line (about 3 volts). This must always be done before reading the pressure. Then zero the microampere meter by adjusting R_2. Raise the pressure on V_2 to atmospheric and, after adjusting the voltmeter to read on the line again, the microampere meter should be made to read full scale by adjusting R_6. The gauge is then ready to be calibrated against a standard vacuum gauge.

CATHODE ASSEMBLY

Fig. 3

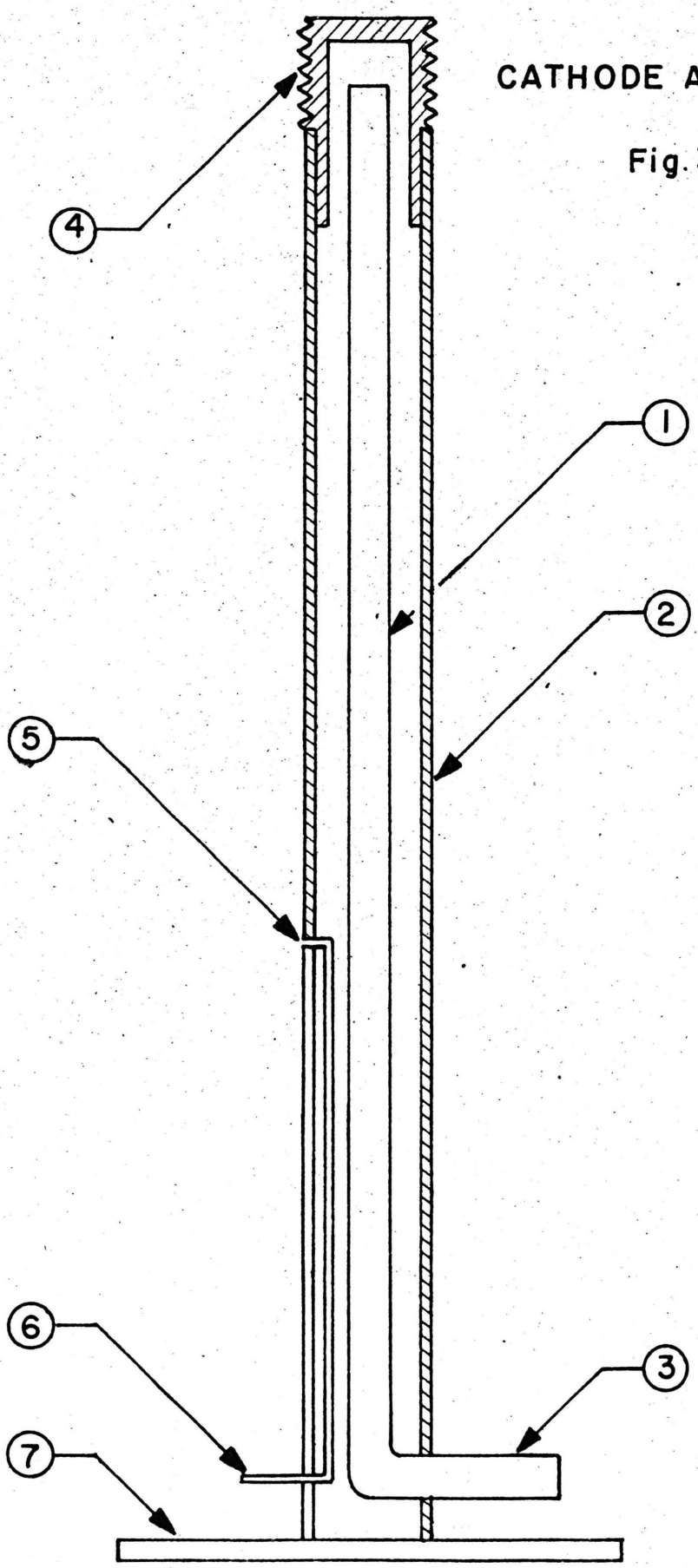


FIGURE 3

(1) 1/4" copper tube

(2) 3/4" O.D. 5/8 I.D. brass tubing

(3) Water outlet

(4) Threaded copper plug silver soldered into end of brass tube

(5) Outlet for inert atmosphere

(6) 1/16" copper tube inlet for inert atmosphere

(7) 1/8" x 3" x 3" brass plate which serves as a base for the cathode assembly

CATHODE ASSEMBLY

Fig. 4

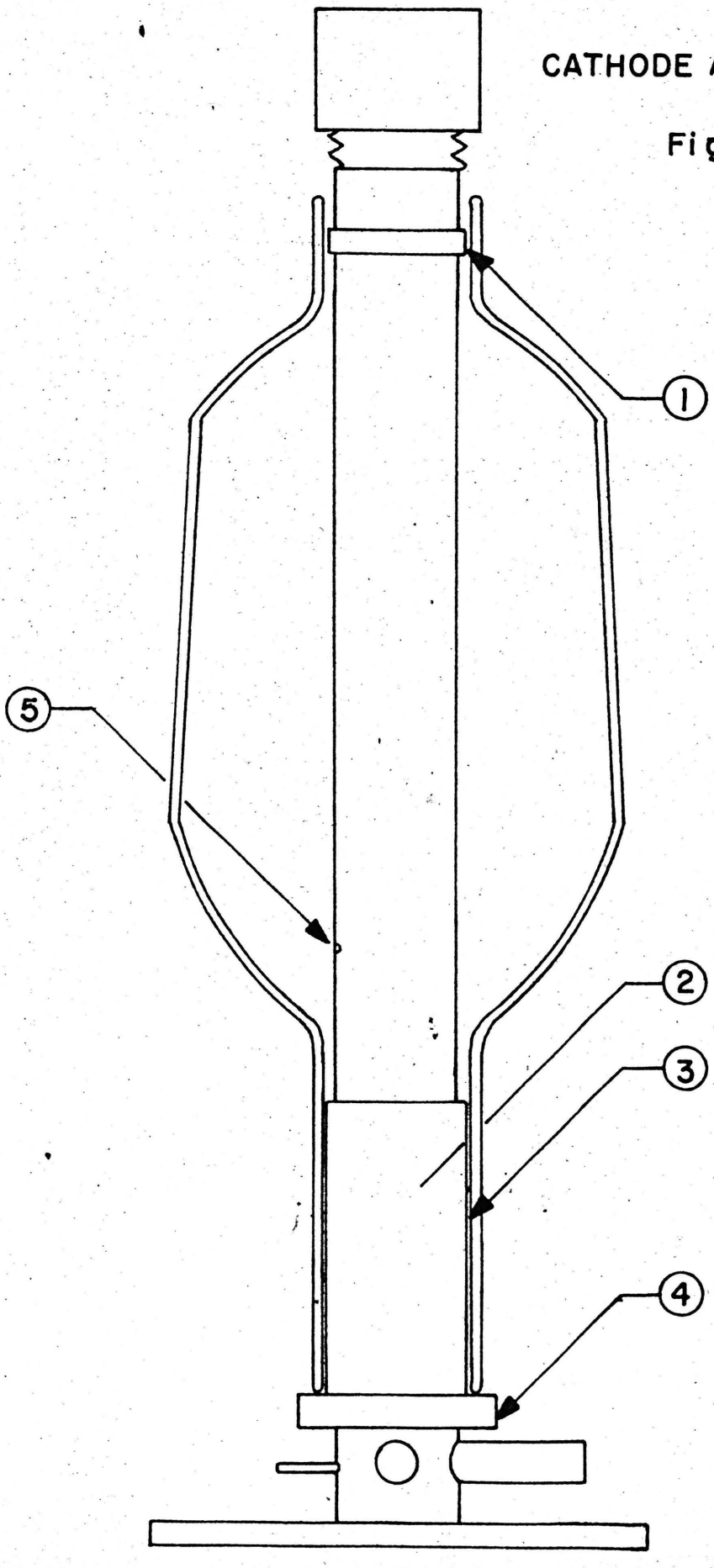


FIGURE 4

(1) Brass ring to center cathode

(2) Brass tube 3/4" I.D. machined to make a snug fit to the glass tubulation. This tube is soft soldered to the center tube.

(3) Vacuum wax seal. This seal was frequently broken when attempting to remove ground glass joint.

(4) Brass shoulder on (2) which keeps the cathode assembly from being further drawn into the ground glass joint.

(5) Inert gas outlet. Note it is now within the evacuated part of the chamber.

CATHODE ASSEMBLY

Fig. 5

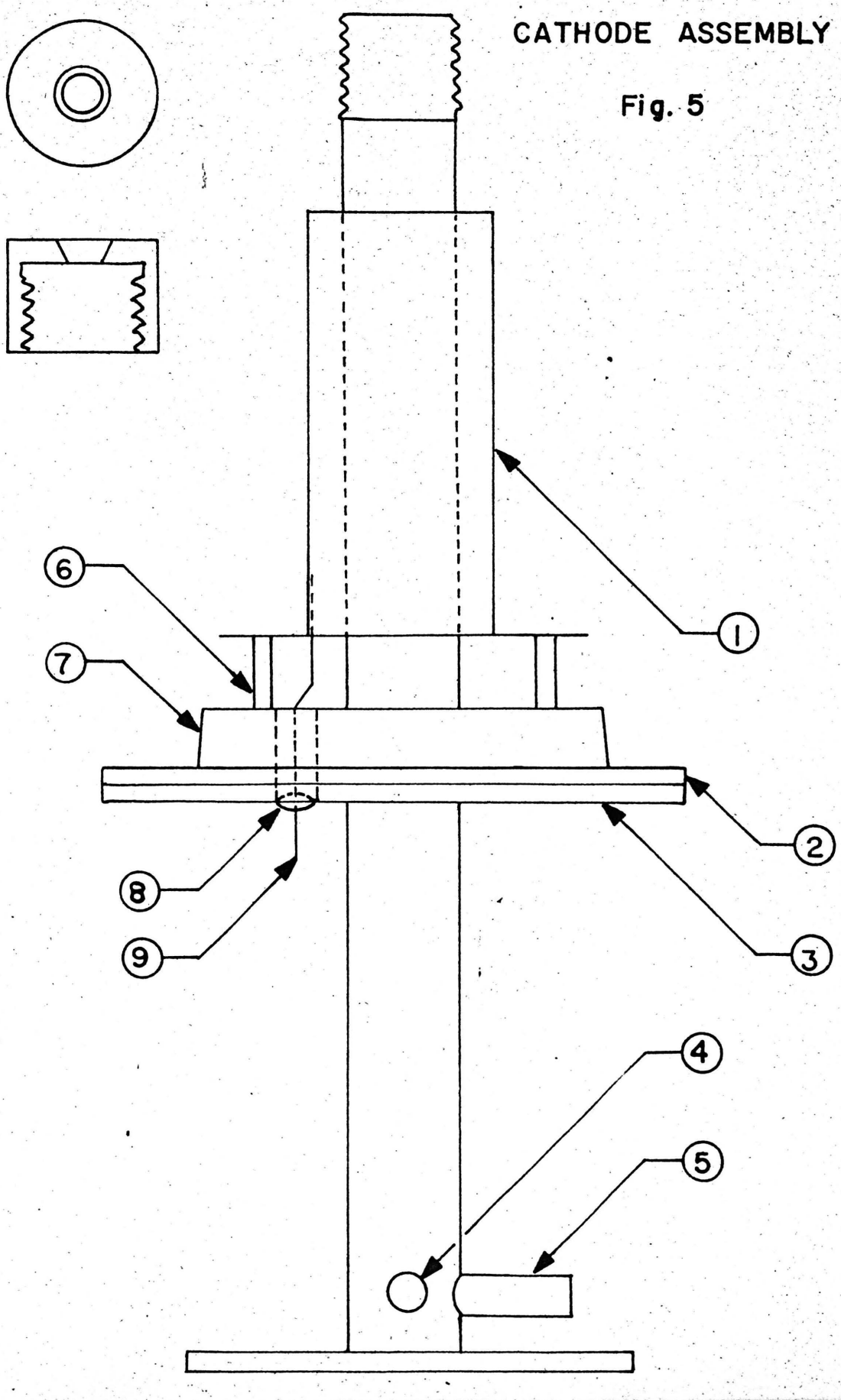


FIGURE 5

(1) $1\frac{1}{4}$" aluminum cylinder made from thin sheet

(2) Rubber vacuum gasket

(3) 1/8" brass plate silver soldered to (7)

(4) Water inlet

(5) Water outlet

(6) Glass rod insulators which hold (1) away from the cathode

(7) $\frac{1}{2}$" thick brass plug machined to fit into the female ground glass joint

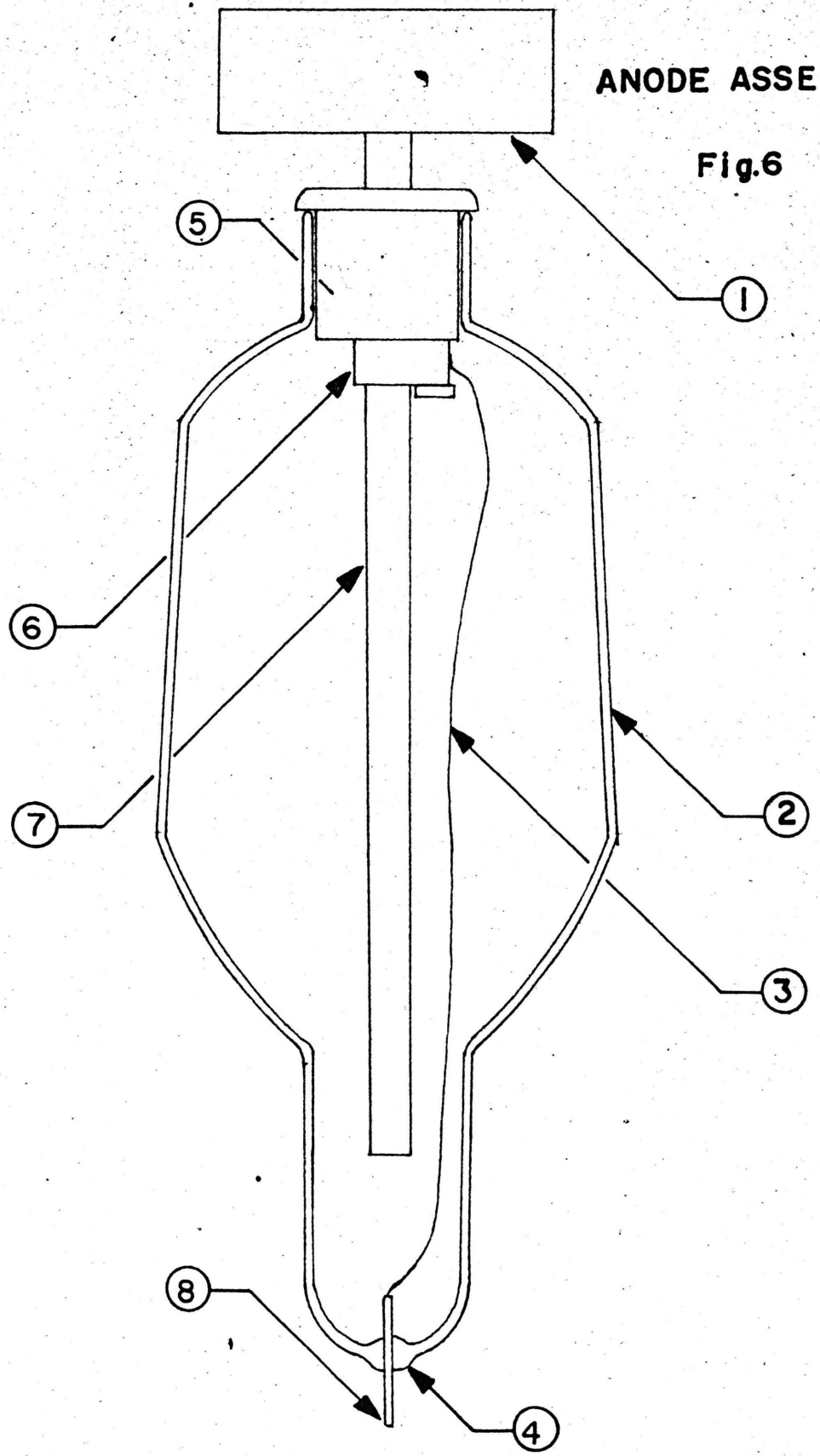
(8) Commercial metal to glass to metal seal, soft soldered to brass plate

(9) Wire which goes through insulator (8) and connects the aluminum cylinder (1) to ground

In the upper left hand corner is a top and side section view of the cap which screws on the top of the cathode assembly.

ANODE ASSEMBLY

Fig.6



VACUUM CHAMBER

Fig. 7

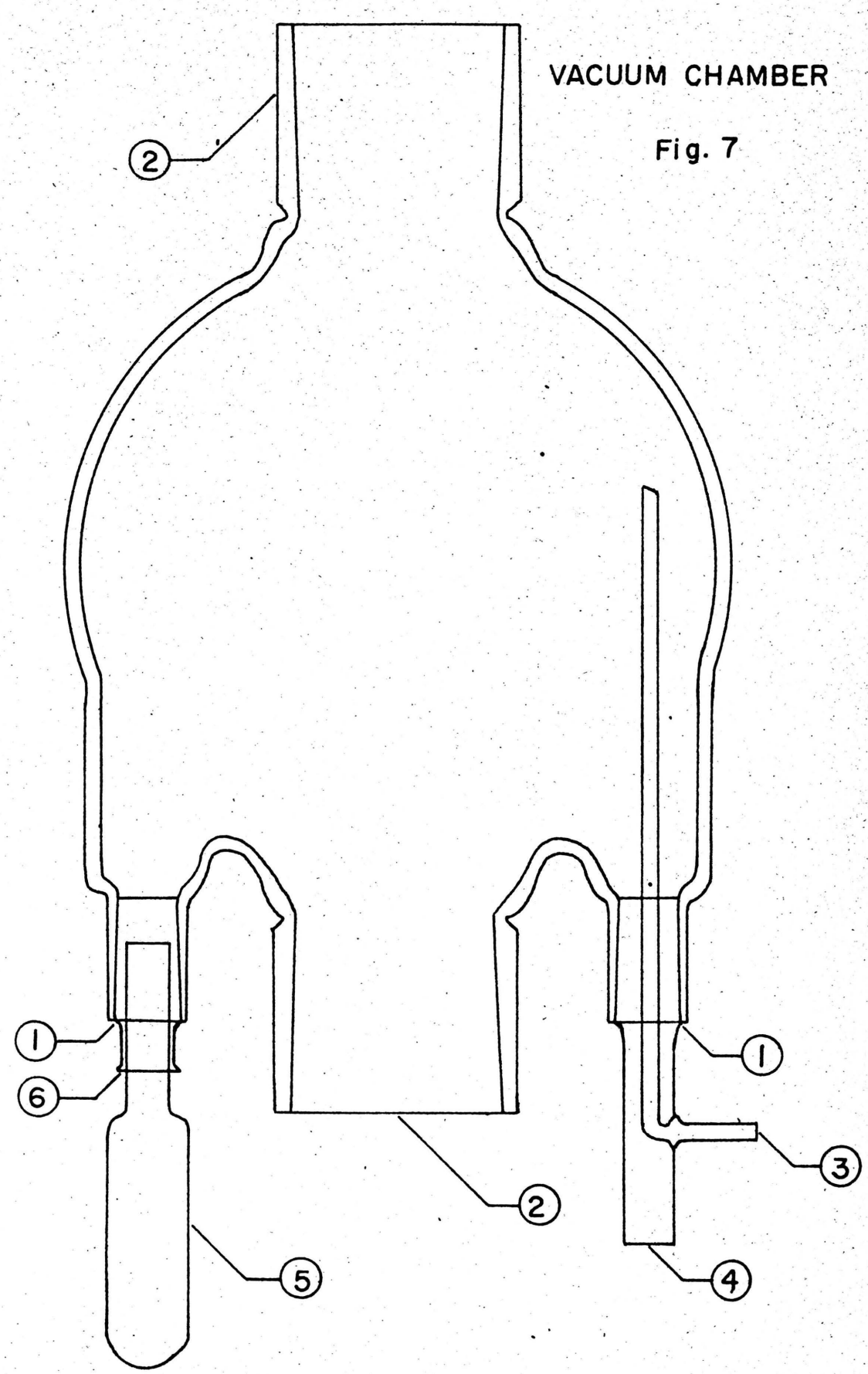


FIGURE 7

(1) 24/40 Standard taper ground glass joints

(2) 71/60 Standard taper ground glass joints

(3) Inert gas inlet. This was installed when the gas inlet through the cathode was discarded.

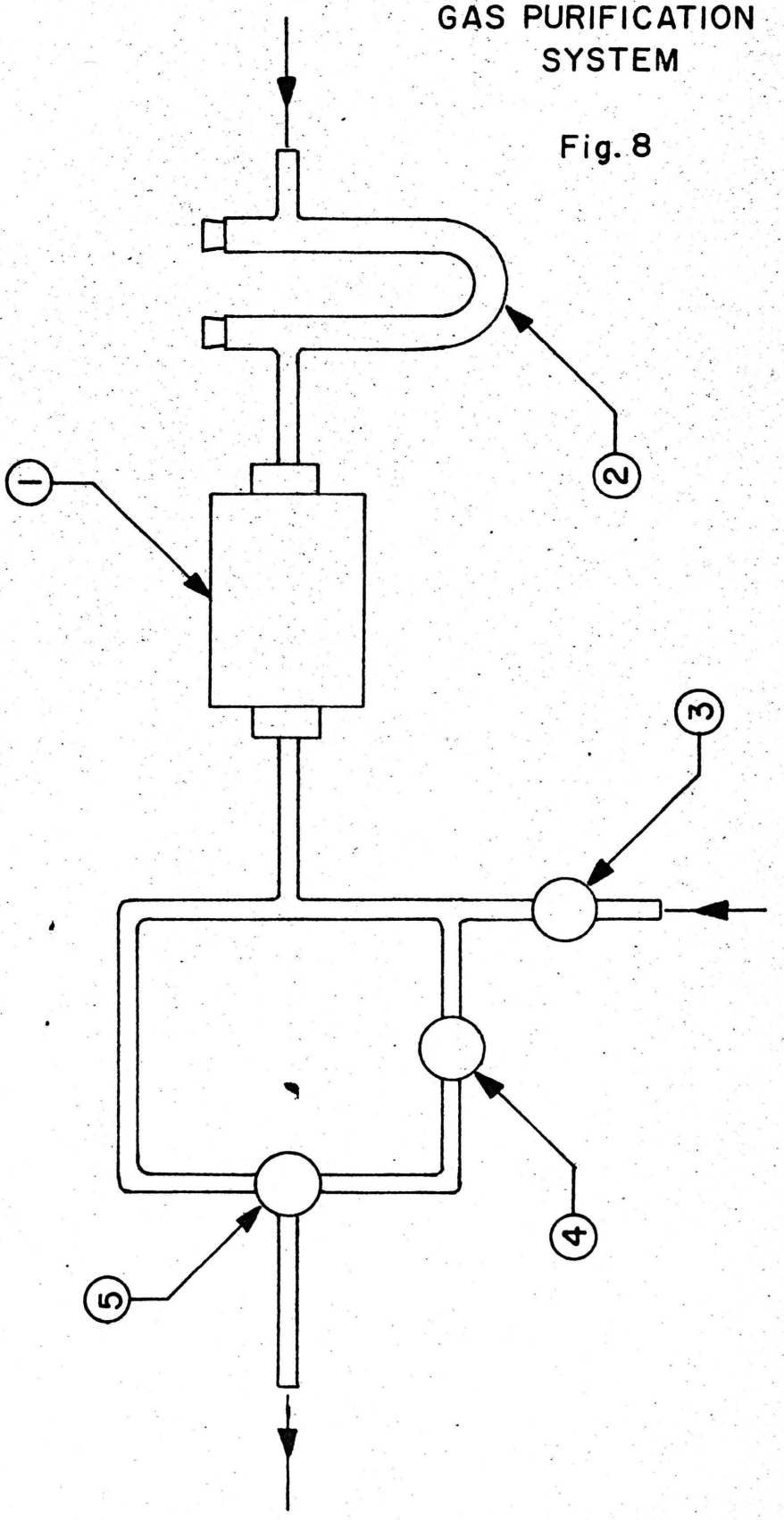
(4) Outlet to vacuum pump

(5) Pirani gauge tube

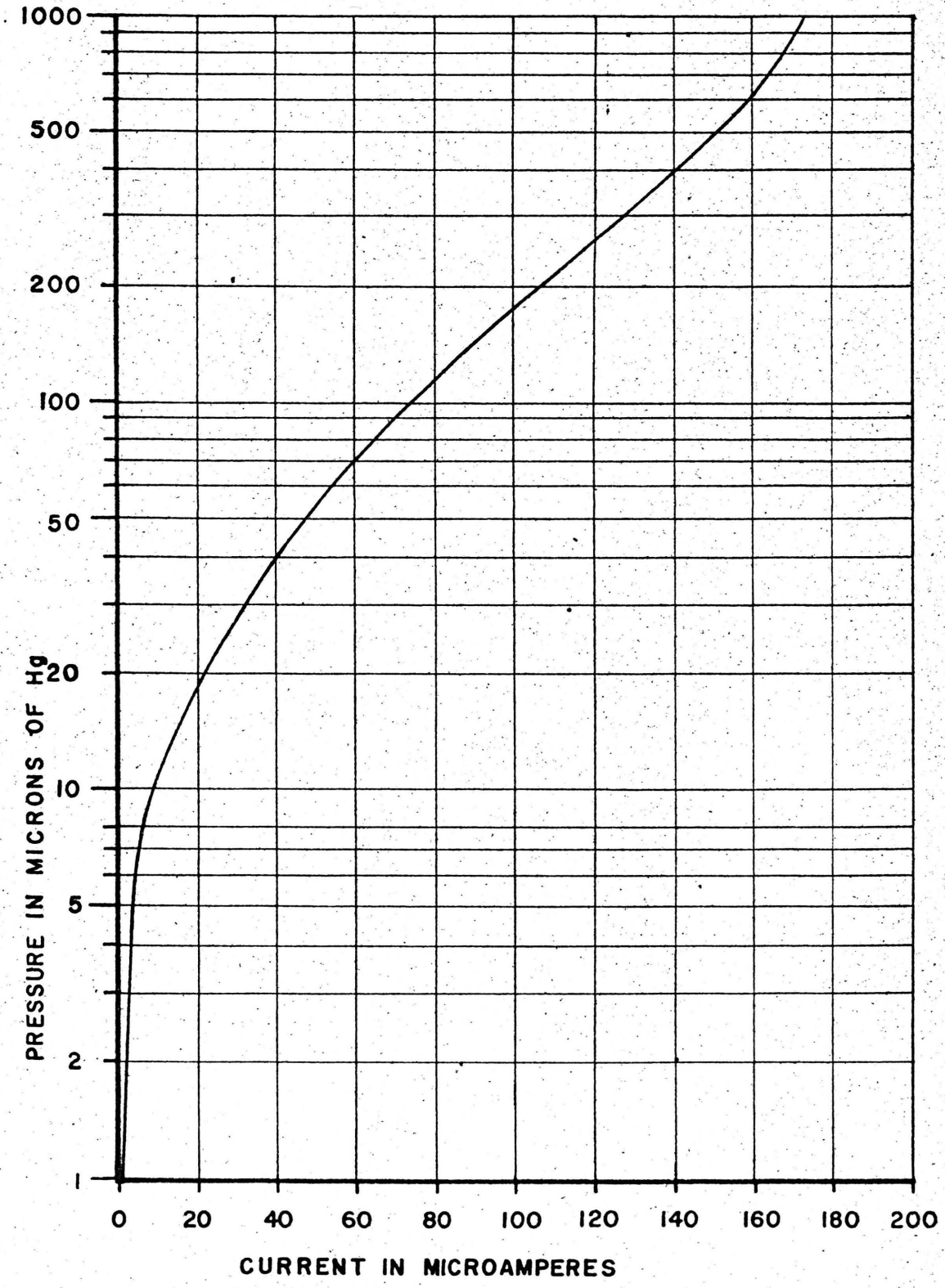
(6) Wax seal

GAS PURIFICATION
SYSTEM

Fig. 8



PIRANI GAUGE CALIBRATION CURVE



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