THE CONSTRUCTION OF AN ELECTRON DIFFRACTION UNIT

by

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INTRODUCTION

This thesis is presented to describe the construction and operation of an electron diffraction unit built in the Department of Physics at Kansas State College, to discuss the techniques in the use of the unit and the preparation of specimens, and to suggest modifications and improvements of the completed unit.

Theory of Waves Associated with Material Particles

The wave nature of material particles was first postulated by Louis de Broglie (6) in 1924. He considers a moving body, whose rest mass is m_o, as moving with respect to a given observer with a velocity u. From the theory of relativity the particle possesses an inertial energy m_oc^2 where c is the velocity of light; and for the given observer the mass possesses an energy $m_oc^2/\sqrt{1-\beta^2}$, where $\beta = u/c$. If then Planck's quantum relation, $E = h\nu$, where E is the energy in a periodic phenomenon, h is Planck's constant, and ν is the frequency, is regarded as a fundamental relation between energy and frequency, a periodic phenomenon should be ascribed to the particle, whose frequency is $\nu_o = m_oc^2/h$. Louis de Broglie identifies this frequency with a pulsation in the space surrounding the particle.

With respect to the system in which the electron is at rest, the amplitude equation for the pulsations may be written

$$\psi = a \sin 2\pi \nu_0 t_0 \qquad (1)$$

Then upon transforming to the system of the observer by means of the Lorentz transformation, one obtains the equation of a traveling wave moving in the coordinates of the observer:

$$\psi = a \sin \frac{2\pi \nu_0}{\sqrt{1-\beta^2}} (t - ux/c^2) \qquad (2)$$

The frequency then becomes

$$\nu = \frac{\nu_0}{\sqrt{1-\beta^2}} = \frac{m_0 c^2}{h \sqrt{1-\beta^2}} = \frac{mc^2}{h} \qquad (3)$$

where $m = m_0/\sqrt{1-\beta^2}$ is the mass of the particle as measured in the system of the observer.

Equation (2) may be written

$$\psi = a \sin 2\pi \nu (t - x/V) \qquad (4)$$

where $V = c^2/u$ is the wave velocity and is defined as the velocity with which a point of constant phase moves forward. It should be remarked that, although the velocity of this wave is greater than the velocity of light, energy is not transmitted by these waves and hence the principle of relativity is not contradicted. It can be shown that the velocity of the material particle is the same as the group velocity of a number of waves associated with the particle, but having slightly different wave velocities.

Using the relation $V = \lambda\nu$, together with Equation (3) and the expression $V = c^2/u$, one obtains

$$\lambda = V/\nu = (c^2/u)/(mc^2/h) = h/mu \qquad (5)$$

which gives the wave length of the traveling wave associated with the particle in terms of its momentum (2,1;6;22,57;28,344).

Application of Theory to Electrons

Since for low velocities the energy of an electron accelerated through a potential P in volts is $\frac{1}{2}mu^2 = eP/300$, where e is the charge of the electron in electrostatic units,

$$\lambda = \sqrt{150/P} \text{ A.U.} \qquad (6)$$

where now λ is given in Angstrom units and the numerical values of e, m, and h have been substituted (3).

If account is taken of the relativistic correction of mass then Equation (5) becomes

$$\lambda = h\sqrt{1-\beta^2}/m_0u, \text{ and} \qquad (7)$$

$$eP/300 = m_0c^2/\sqrt{1-\beta^2} - m_0c^2. \qquad (8)$$

Eliminating the velocity u between these equations gives

$$\lambda = \frac{h\sqrt{150/em_0}}{\sqrt{P + (e/600\, m_0c^2)P^2}} \text{ cm.} \qquad (9)$$

Substituting in Equation (9) for e, m_0, and h, and converting to Angstrom units,

$$\lambda = \frac{12.26}{\sqrt{P + 0.978 \times 10^{-6}P^2}} \text{ A.U.} \qquad (10)$$

The error in using Equation (6) for accelerating potentials less than 20,000 volts is less than one percent (28, 350).

Early Investigations of Electron Diffraction

It was suggested by Elsasser (7) in 1925 that the wave nature of electrons could be established by the interaction of the electrons with crystals.

The theory of the interactions of x-rays with crystals had been well developed, and, as shown by later experiments, could be extensively applied to the interaction of electrons with crystals. The diffraction of x-rays by crystals is thoroughly discussed by Sproull (22), Compton and Allison (4), and by James (15). The diffraction of electrons by crystals is discussed by Thomson and Cochrane (25).

Davisson and Germer (5) in 1927 confirmed de Broglie's hypothesis of the wave nature of electrons by diffracting a beam of electrons from the (111) planes of a single large crystal of nickel.

Shortly thereafter the following experiments were conducted: Reid (20) diffracted electrons through a thin film of celluloid; Thomson used films of aluminum, gold, and platinium (24, 23); and Nishikawa and Kikuchi (18) were the first to diffract electrons from the surface of a crystal at a glancing angle.

Since that time much work has been done in the field of electron diffraction. Sproull (22, 536) and Thomson and Cochrane (25) have given an extensive bibliography of this work.

A dynamical theory of electron diffraction is given by Heidenreich (12) using a wave mechanical treatment of the approximation to nearly free electrons in Brillouin zones.

THE CONSTRUCTION OF AN ELECTRON DIFFRACTION UNIT

The actual construction of the electron diffraction unit was started in September, 1949. The original design was similar to a unit constructed by Ruedy (21). It was to have used an electrostatic focusing electron gun. However, the simplification of the high voltage power supply unit in regards to stabilization made it feasible to resort to the use of electromagnetic focusing. The plate chamber was modified to provide a means of removing the photographic plate from the camera while maintaining a vacuum in the diffraction chamber. A radio frequency high voltage power supply was used instead of a 60 cycle power supply. Other modifications of Ruedy's design were made as found necessary in the construction.

The electron diffraction unit shown in Plates I and II is composed of a number of components which are here described.

The Plate Chamber

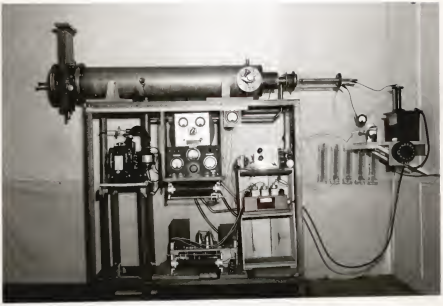
The plate chamber shown in Plate III, Fig. 1, and Plate IV was designed and constructed by E. J. Marak. It consists of a vacuum tight compartment in which is contained the film cassette, the viewing screen, the slit system, and the airlock mechanism.

Shown in Plate IV is the film cassette (A) with its sliding cover (B). The cassette is lowered into position by means of

EXPLANATION OF PLATE I

The electron diffraction unit.

PLATE I



EXPLANATION OF PLATE II

The electron diffraction unit.

A - the diffraction chamber

B - end plate

C - aperture tube

D - main exhaust tube

E - oil diffusion pump

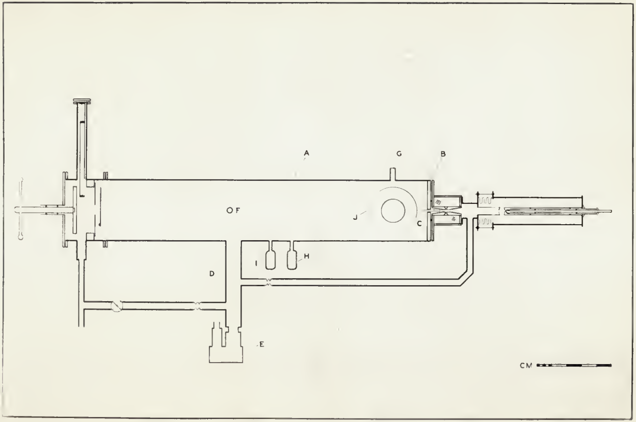
F - screen viewing port

G - specimen viewing port

H - thermocouple vacuum gauge

I - ionization vacuum gauge

PLATE II



EXPLANATION OF PLATE III

Fig. 1. The plate chamber

Fig. 2. The specimen holder

PLATE III

Fig. 1

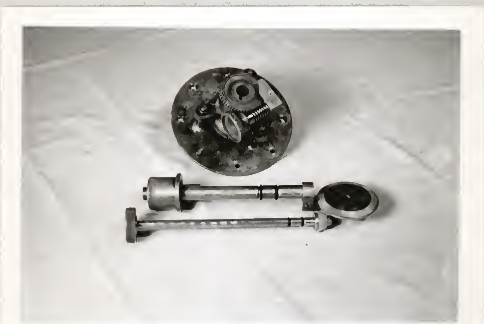


Fig. 2

EXPLANATION OF PLATE IV

The plate chamber.

A - film cassette

B - cassette cover

C - cassette rack pinion

D - cassette control

E - cassette cover catch

F - viewing screen

G - plate chamber valve seat

H - plate chamber valve

I - plate chamber valve shaft

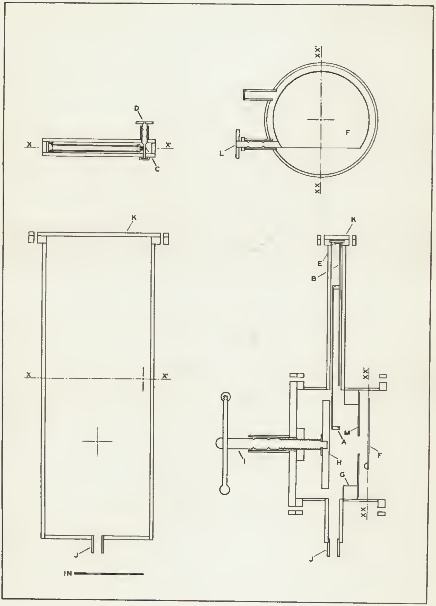
J - plate chamber exhaust tube

K - cassette port

L - shutter control

M - image border slit

PLATE IV



a rack along one side of the cassette and a pinion (C). The pinion is driven from the outside by a shaft and cassette control (D). A vacuum seal between the pinion shaft and the shaft sleeve is effected by "O" rings set in "O" ring glands cut in the shaft. As the cassette is lowered a bar (E) across the top of cassette cover engages in slots cut in the plate chamber. Thus, the cover remains fixed as the cassette is lowered and the film is in a position to be exposed. As the cassette is raised the cover closes the cassette and it may be removed from the plate chamber.

The viewing screen (F) was made of Patterson X-ray Screen Type B-2, mounted on a brass shutter. The position of the screen may be controlled from the outside by a shaft rotating through "O" ring seals.

The image border slits (M) were made of brass sheet in the form of half circles and mounted on the plate chamber valve seat. This arrangement allows for five images to be formed on a 13 cm by 18 cm film plate. The slits are removable for the purpose of photographing transmission patterns.

The air-lock mechanism consists of a plate chamber valve (H) and a valve seat (G). With the film cassette in the "up" position the valve may be moved forward to press against the seat by means of a threaded shaft (I). When the valve is seated the exhaust tube (J) to the plate chamber is closed and air is admitted into the chamber. The cassette port (K) may then be opened for removal of the film. After closing the cassette port the plate chamber must be evacuated through the exhaust tube before the valve can be moved away from the seat.

The Diffraction Chamber

The diffraction chamber (A), shown in Plate II, is a cylindrical brass tube 20.3 cm (8 in) in diameter and 108 cm long. One end was flanged for connection to the plate chamber, the other end being closed by a brass plate (B) soft soldered into place. A hole one-half inch in diameter was drilled in the center of the plate for admission of the electron beam into the diffraction chamber and for the insertion of the aperture tube (C) into the magnet.

The main exhaust tube (D) is on the under side of the chamber. This is a brass tube 5 cm in diameter and 29 cm long. The oil diffusion pump (E) was connected directly to the main exhaust tube by a silver solder connection.

There are two observation ports in the diffraction chamber. The screen viewing port (F) is on the side of the chamber. This is a brass tube 2 cm in diameter and 6 cm long located 44 cm from the viewing screen. A glass window was waxed into a recess in the tube. A right angle prism in the chamber allows viewing the screen. The specimen viewing port (G) is directly above the position of a specimen in the electron beam. It is similar in construction to the screen viewing port tube.

The connections for the two types of vacuum gauges, a thermocouple gauge (H) and an ionization gauge (I), used with

EXPLANATION OF PLATE V

The focusing magnet.

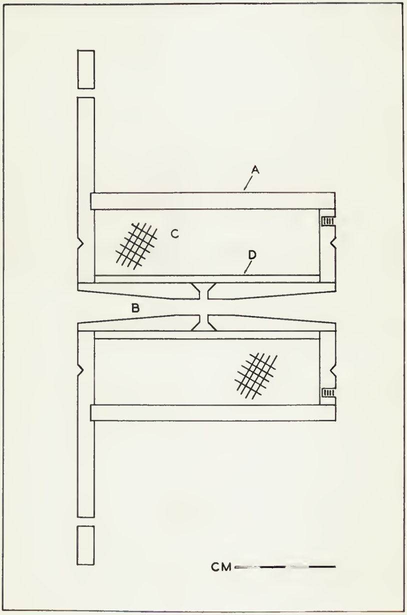
A - outer case

B - pole pieces

C - coil

D - brass separator

PLATE V



the unit are on the under side of the diffraction chamber.

The interior of the diffraction chamber is lined by a tube of Mu-metal, manufactured by the Allegheny Ludlum Steel Corporation, Pittsburgh, Pennsylvania. This provides magnetic shielding of the electron beam from the earth's magnetic field and from stray fields originating in various local power supplies.

The Focusing Magnet and Current Supply

The beam focusing magnet consists of an iron-encased coil. Plate V shows the iron case. It was made of one-fourth inch Alleghany Ludlum Steel Corporation's No. 2 relay iron plate. The outer case (A) was formed by heating and bending a flat plate into a cylinder. The pole pieces (B) were made of Alleghany Ludlum No. 2SS round stock. The air gap is 3.2 mm in length and the inside diameter of the pole pieces is 6.4 mm. The coil (C) consists of 6072 turns of No. 23 B and S gauge, Formax covered wire, having a resistance of 54 ohms.

"O" ring glands were cut in the flat end plates to give a vacuum seal in joining the magnet to the specimen chamber and the electron gun to the magnet. The current for the magnet was supplied by storage batteries through a potentiometer circuit.

EXPLANATION OF PLATE VI

Fig. 1. The electron gun

Fig. 2. The high voltage power supply unit

PLATE VI

Fig. 1

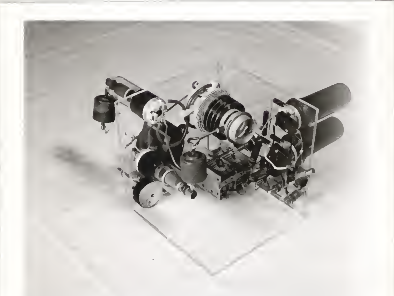


Fig. 2

EXPLANATION OF PLATE VII

The electron gun.

A - accelerating anode

B - grid

C - accelerating anode support

D - grid support

E - filament support

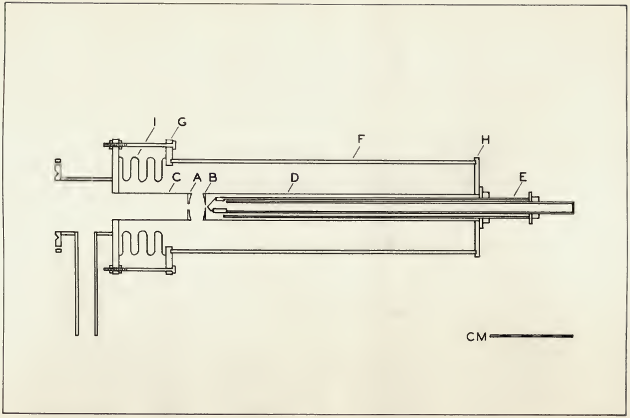
F - glass tube

G - bellows ring

H - grid end plate

I - flexible bellows

PLATE VII



The Electron Gun and The Filament Supply

The electron gun assembly is shown in Plate VI, Fig. 1, and Plate VII. The accelerating anode (A) and the grid (B) are brass disks having concave surfaces. They are supported by brass tubes, (C) and (D). The filament support (E) consists of two concentric tubes inserted in the grid support tube. "O" rings are used both for electrical insulation and for vacuum seals. The accelerating anode, the grid, and the filament are enclosed in a glass tube (F) 9 cm in diameter and 30 cm long. The glass tube was bonded to the bellows ring (G) and the grid end plate (H) by Cerroseal joints.

A flexible bronze bellows (I) allows alignment of the grid aperture with respect to the accelerating anode aperture for directing the electron beam into the diffraction chamber.

An auxiliary exhaust tube connection was inserted between the magnet and the flexible bellows to facilitate evacuation of the electron gun.

The filament was made of 3 mil tungsten wire, and its heating current was supplied by an x-ray tube filament transformer whose primary voltage was controlled by a variac.

The Specimen Holder

The specimen holder shown in Plate III, Fig. 2, and Plate VIII, which was constructed by W. G. Wilson, provides four adjustments for aligning the specimen in the beam. The

EXPLANATION OF PLATE VIII

The specimen holder.

A - vertical positioning control

B - flexible bellows

C - mounting flange

D - sleeve case

E - threaded cap

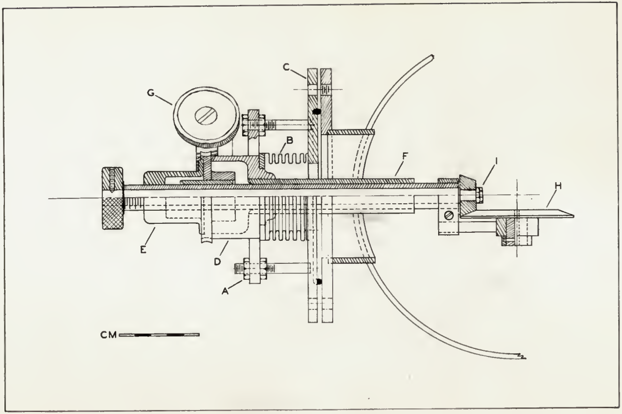
F - outer shaft

G - tilt control

H - specimen mounting table

I - inner shaft

PLATE VIII



lower threaded nut (A) allows vertical positioning of the specimen by lever action on the other two supports. This is accomplished by distorting a flexible bellows (B) between the mounting flange (C) and the sleeve case (D). Motion in a horizontal direction perpendicular to the beam is effected by a threaded cap (E) driving the outer shaft (F). The outer shaft is keyed to a geared wheel driven by a worm gear (G) for tilting of the specimen mounting table (H). The specimen mounting table may be rotated by the inner shaft (I) through a bevelled friction drive.

"O" rings are used as vacuum seals for all rotating and sliding parts of the specimen holder.

The Vacuum System, Pumps, and Gauges

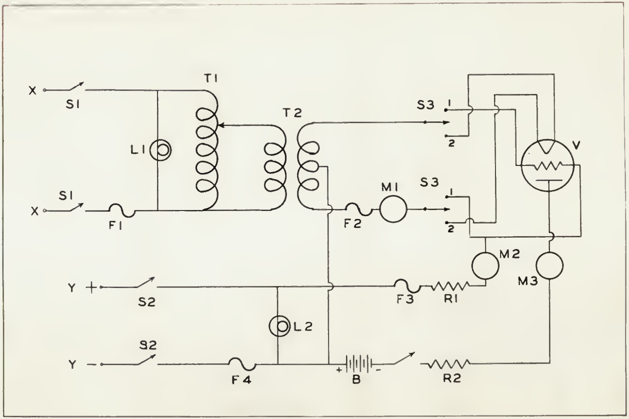
A Welch Duo-Seal Vacuum Pump, Model 1400B, was used to provide forepump pressure to a Distillation Products oil diffusion pump, Type VMF10-W. Pressures in the order of 10^{-4} mm of mercury were obtainable. The pressures were determined in the range of 1 mm to 10^{-3} mm of mercury by means of a vacuum thermocouple gauge, Type 501, manufactured by the National Research Corporation. For the measurement of pressures less than 10^{-3} mm of mercury, an ionization gauge, Type VG-1A, Distillation Products, was used. The associated circuit used with the ionization gauge is shown in Plate IX and was constructed by M. R. Lee.

EXPLANATION OF PLATE IX

The ionization gauge circuit.

B - 9 volt battery	S1 - A.C. power switch
F1 - .75 a. fuse	S2 - D.C. power switch
F2 - 10 a. fuse	S3 - outgas-operate switch
F3 - 1/32 a. fuse	Position 1 - outgas
F4 - 1/8 a. fuse	Position 2 - operate
L1 - A.C. pilot lamp	S4 - plate switch
L2 - D.C. pilot lamp	T1 - variac
M1 - 0-10 ammeter	T2 - 6.3V C.T. transformer
M2 - 0-25 milliammeter	V - VG-1A ionization tube
M3 - 0-200 microammeter	XX - A.C. power connection
R1 - 1,000 ohm resistor	YY - D.C. power connection
R2 - 10,000 ohm resistor	

PLATE IX



Two types of vacuum seals between flanged joints were used. One type consisted of "O" rings set into glands, the flanges then being bolted together. The other type of seal was made from sheets of Polythene, a chemically inert gasket material manufactured by Du Pont, and was found to be superior to the "O" ring seal in this type of connection. All seals involving the rotation or sliding of shafts in sleeves were of the "O" ring type and were found to be highly satisfactory.

The glass to metal bonding in the electron gun assembly was accomplished by the use of Cerroseal, manufactured by the Cerro de Pasco Copper Company, New York.

The joining of metal to metal in the construction of the unit involved three types of bonding processes: brazing, silver soldering, and soft soldering. Silver soldering was used in the bonding of small parts. Brazing was used for the larger sections, as in the construction of the plate chamber. It was found that small air pockets were left in the brazed joints. These were filled with soft solder. All metal to metal and metal to glass joints were finally given a coating of glyptal.

The High Voltage Power Supply Unit

The high voltage power supply unit shown in Plate VI, Fig. 2, and Plate X, was a conventional type grid-tickler feedback oscillator whose plate circuit inductance was the primary winding of a high voltage transformer. The secondary

EXPLANATION OF PLATE X

The high voltage power supply unit and the electron gun filament supply.

A - grid tickler winding

B - primary winding

C - filament winding

D - secondary winding

E - 2 mh choke

F - high voltage output

G - Pos. 300V D.C. input

C1,C2,C3,C5 - .01 mf condensers

C4 - 500 - 700 mmf variable condenser

C6,C7 - .01 mf 30,000V condensers

R1,R2 - 600 ohm resistors

R3 - 100,000 ohm resistor

R4,R6 - 10 ohm resistors

R5,R7 - 10,000 ohm resistors

R8 - 150 megohm resistor bank

R9 - 2 megohm variable resistor

M1 - 0-1 milliammeter

M2 - 0-1 milliammeter

M3 - 0-5 ammeter

V1,V2 - Type 6L6 vacuum tubes

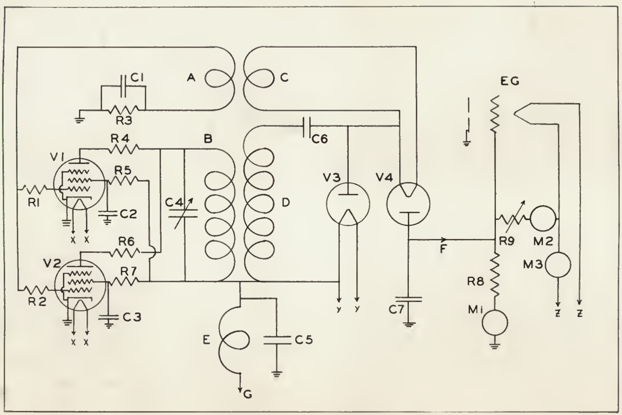
V3,V4 - Type 1B3 vacuum tubes

XX - 6.3V A.C. filament connection

YY - 1.3V A.C. filament connection

ZZ - electron gun filament connection to transformer

PLATE X


C1
A
C
R3
R4
B
C6
V1
R5
V3
V4
EG
R1
C4
D
C2
R9
M2
R6
F
V2
R7
M3
R8
R2
C3
y y
C7
M1
z z
X X
X X
E
C5
G

winding of the transformer fed into a voltage doubling rectifier circuit.

The oscillator tubes were Type 6L6 connected in parallel. The rectifier tubes were Type 1B3. The filament of the first rectifier was at a positive potential of 300 volts provided by a "B" battery. The current through the filament was provided by a conventional 6.3 volt filament transformer operating on 110 volt primary alternating current. The filament of the doubling rectifier tube, being at a negative potential of 10,000 volts, was supplied by a single turn of wire wound on the transformer coil form.

The output of the supply was continuously variable from minus 5,000 volts to minus 22,000 volts D.C. by varying the B+ supply voltage. A control of the output voltage was also obtained by variation of the screen voltage on the Type 6L6 tubes.

The high voltage output was connected to the grid of the electron gun. A bleeder resistance of 150 megohms was connected to ground in series with a 0-1 milliammeter. The grid of the electron gun was connected to the filament circuit through a 2 megohm variable resistance to provide self-biasing of the grid. The biasing of the grid with respect to the filament effectively reduces the size of the electron source giving sharper focusing and greater resolution.

THE USES OF THE ELECTRON DIFFRACTION UNIT AND THE PREPARATION OF SAMPLES

The major difference between the use of x-rays and the use of electrons for diffraction purposes is their penetrating properties. It can be shown that in general, the probability of an electron with 50,000 electron volts energy suffering an elastic collision, when it encounters an atom, is of the order of 10^7 times as great as the probability that an x-ray photon of ordinary energy or hardness will be scattered when it encounters such an atom. Thus the very slight penetration of the electron makes it suitable for the study of surfaces and films.

Among the many uses of electron diffraction are studies of the following: the growth of crystals in thin films, on substrates, in evaporated or sputtered layers, and in thin electrolytic depositions; the oxidation of metals and the corrosion and oxidation of surfaces; the measurements of the electfical conductivity of surface layers and the inner potential; the grinding and polishing of metals and non-metals; and the thermionic and photoelectric phenomena of surfaces.

Electron diffraction has been used with gases and vapors, being superior to x-rays for this purpose, but involving a special technique: a jet of gas is directed through the electron beam in the vacuum chamber and is then removed from the system by liquid-air traps and fast pumps.

Sproull (22, Chap. 24) has included a large number of references concerning the uses of electron diffraction. Some applications not mentioned by Sproull are the studies of high temperature surface reactions (9), particle size determination in thin films (16), and metal crystal textures, aided by the electron microscope (12).

The preparation of samples presents a major difficulty in electron diffraction analysis in that adsorption and chemical union with the gases in air may completely obliterate the diffraction pattern of the sample during an hour's exposure to the atmosphere.

Obtaining films of the proper thinness for transmission patterns is another problem. Many samples can be prepared by floating the specimen, especially metals, on etching solutions and picking them up just before disintegration occurs. Others may be made by evaporating metal onto organic films, then dissolving away the organic material.

Much of the technique developed for electron microscopy seems applicable to electron diffraction. Williams and Backus (27, Appendix) have prepared a summary of the general procedures for the electron micrograph, and Gulbransen (9) treats the preparation of specimens for reflection studies.

RECOMMENDATIONS FOR IMPROVEMENT OF THE ELECTRON DIFFRACTION UNIT

At the writing of this paper the air-lock mechanism in the plate chamber had not been installed. The installation requires the insertion of a valve in the plate chamber exhaust tube, and an auxiliary pump to evacuate the plate chamber.

A sleeve above the viewing screen shutter sleeve has been provided for the purpose of installing a comparison shutter similar to that described by Weber and Dahm (26). This allows the photographing of a standard such as gold or aluminum adjacent to an unknown sample.

The vertical positioning control of the specimen holder has been found unsatisfactory in that the lugs on the sleeve case catch in the threads on the control rod. An arrangement such as that found on the Finch form of camera (8) would result in smoother action of the control.

A furnace operating in the diffraction chamber would be desirable for the study of surfaces at elevated temperatures, surface reactions, and the outgassing of samples. Such furnaces have been described by Allessandrini (1) and Gulbransen (9).

It is suggested that in future designs of electromagnetic lenses for the diffraction unit the removal of the pole pieces be considered for the purposes of cleaning and, possibly, experimentation with different shaped pole pieces (14). Particular attention should be given to the machining of the pole pieces (11). This is of special importance if shadow micrograph uses be made of the unit because of aberrations introduced by imperfections (10).

A method for cleaning the interior of the electron gun is not now available. It is suggested that this assembly be made demountable in much the same manner as those made by Pierce (19), Moss (17), and Keogh and Weber (16).

It is recommended that the unit described in this paper might be converted to an electron microscope. Hillier and Baker (13) have described an electron diffraction unit of improved resolution having two electromagnetic lenses. The only change necessary to convert it from a diffraction unit into an electron micrograph is a reduction of the focal length of the second lens. This places a highly reduced image of the electron source close to the plane of the specimen.

The present unit has one electromagnetic lens and is so constructed that it could be converted by the addition of another lens.

ACKNOWLEDGMENTS

The author is indebted to several persons for their assistance during this research. Acknowledgment is extended to Dr. R. D. Dragsdorf for his helpful advice and criticism during the construction of this unit. Special appreciation is expressed to Mr. E. J. Marak for his assistance in building portions of the unit. Grateful recognition is given to Mr. W. G. Wilson for the construction of the specimen holder, and to Mr. D. A. Rittis whose assistance in the Shop was invaluable.

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