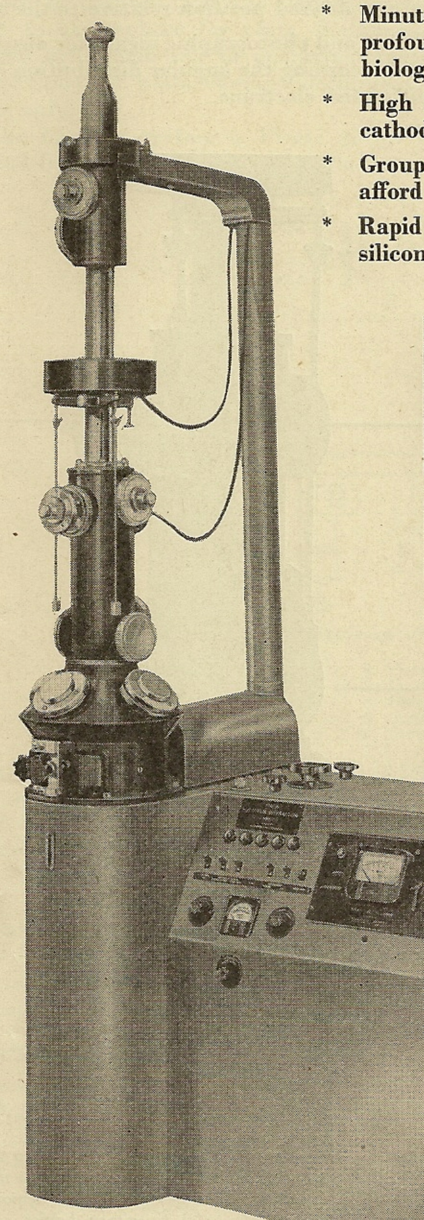
TECHNICAL SCIEX BULLETIN

THE FINCH ELECTRON DIFFRACTION CAMERA

A Unique Instrument for the Study of Surface Phenomena

- **Outstanding for the examination of monomolecular layers, thin films, crystal structures, etc.**
- **Minute specimen amounts can be examined—a feature of profound importance to physicists, chemists, metallurgists, biologists.**
- **High intensity stabilised electron beam obtained from cold cathode, a feature of proved reliability in the Finch Camera.**
- **Groups of four ports at 50 and 25 cm. diffracted beam lengths afford extreme flexibility in operation.**
- **Rapid exposure times; rapid pump down cycles with fast silicone oil pumps and isolation valves.**



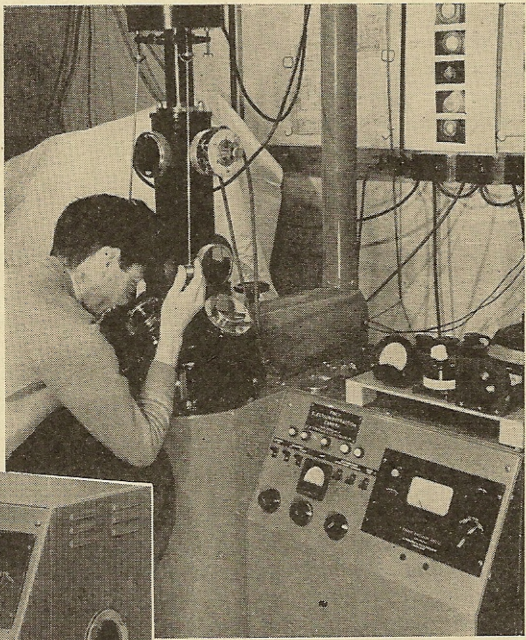


Fig. 1. The Finch Electron Diffraction Camera in use at the Laboratory for the Physics and Chemistry of Rubbing Solids, Department of Physical Chemistry, Cambridge University.

Some of the users of the new type Finch Camera are:—

British Iron and Steel Research Association, Swansea; Chemical Research Laboratory, Teddington; Farouk University, Alexandria; Sir John Cass Technical Institute; Anglo Iranian Oil Company; National Physical Laboratory, Teddington; Imperial College, London; Cambridge University.

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Wave properties were first attributed to the moving electron by de Broglie in 1924 whilst developing a wave theory of matter. His theory was verified experimentally, by other pioneer workers[1, 2], almost simultaneously, by successfully diffracting beams of electrons with the aid of the regular array of atoms in crystal lattices acting as three dimensional gratings. Numerous workers have confirmed these results and extended electron diffraction to its present position as a powerful research technique for the investigation of the structure of matter, and particularly for the study of thin films, surfaces and the structure of gases.

In order fully to appreciate the usefulness of electron diffraction it is necessary to understand the special features distinguishing it from X-ray diffraction, and also the type of problem which can most readily be investigated with its aid.

The outstanding difference between the two techniques is that even the fastest electrons used for diffraction work have a very small penetrating power, making the process particularly suitable for the investigation of surface phenomena. A further advantage, resulting from the ease with which electrons are scattered, is the intensity of the diffraction pattern, so that very short exposure times are necessary for photographic record, being of the order of seconds compared with hours for X-ray work. X-ray crystal analysis and electron diffraction are complementary techniques, the former being pre-eminent for the examination of material in bulk, while the latter has unique advantages for the investigation of surfaces, thin films and free molecules.

The applications of electron diffraction are continually extending as its possibilities become appreciated; it has been successfully used for the investigation of the structure of thin films and surface layers of metals, metallic oxides and compounds generally, oils, lacquers, elastomers, fibres, etc. Its practical application has already met with outstanding success in investigating surface phenomena connected with, for example, corrosion, metal to rubber bonding, plating, lubrication, catalytic activities of surface films, poisoning of oxide coated filaments, etc.[3]

Fundamentals of the Electron Diffraction Camera

In principle the Camera consists of :—

(1) A device for producing a fine beam of electrons moving at a controlled velocity. Electrons accelerated through a difference of potential of 50 KV. have a wave length of about 5 x 10^{-8}cm. which is of the order required for the examination of crystal lattices. The practical applications of electron diffraction which have been employed up to date find the voltage range 40—65KV. to be the most useful.

(2) An arrangement for holding the specimen in any desired position relative to the beam.

(3) Visual and photographic means for observing and recording the angular distribution of the diffracted electrons.

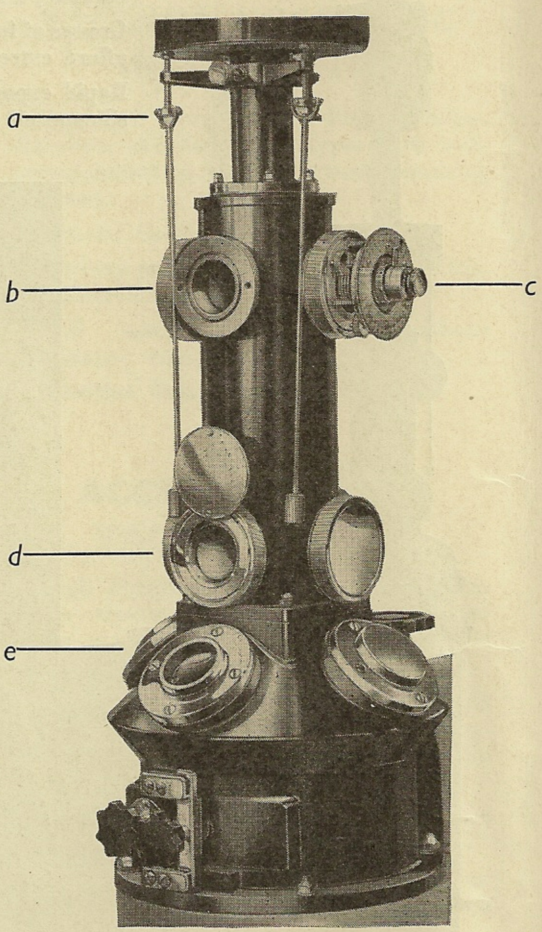

Fig. 2. Close up view of specimen and camera sections showing :—

(a) Gimbal movements on focusing lens for scanning electron beam. (b) One open port of the 50 cm. group; note the two-inch aperture for mounting accessories, simple demountable flange and "O" ring seal. (c) Specimen holder allowing movements in two planes and rotation. (d) Ports of the 25 cm. group with aperture cover seals and inspection window fitted. (e) Large viewing ports in camera section.

From Bragg's Law relating the diameter of any given diffraction ring (a polycrystalline specimen for example) to the lattice spacing, it can be shown that the diameter of the ring depends also on the wave length. In consequence, any change in accelerating voltage during exposure will lead to a broadening of the diffraction rings or possibly doubling of the pattern. For the best definition it is therefore imperative for the accelerating voltage to have a high order of stability; the high tension system has to be designed accordingly.

Electron Beam Production. The methods of stabilization depend primarily on the type of source used for the electron beam. In the hot cathode system, stabilization of the accelerating voltage is usually obtained by the use of complex electronic circuits.

The Edwards Diffraction Camera uses the cold cathode discharge as a source of electrons together with the Finch H.T. circuit [4, 5, 6] because of the excellent performance, inherent simplicity and reliability, which can be obtained with properly designed apparatus of this type. The cold cathode discharge gives an electron beam of high current density[7] and can be used for accelerating voltages of 15 to 80 KV. The most useful range of accelerating voltages for structure analysis lies between 30 and 70 KV. and under these conditions a cold cathode source is robust and simple, a monochromatic electron beam being easily obtained.

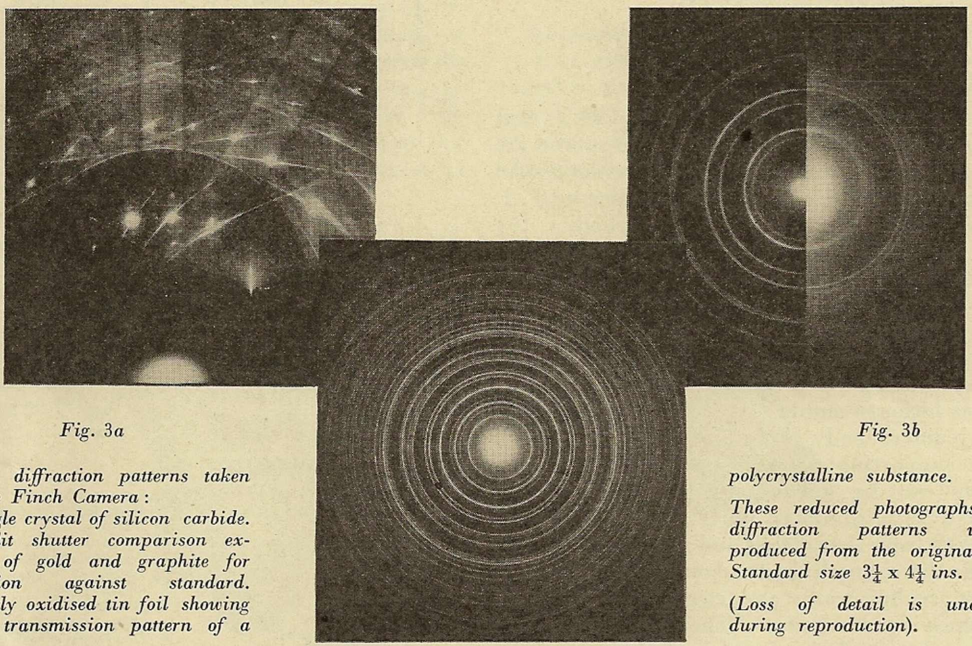

Fig. 3a

Fig. 3b

Fig. 3c

Typical diffraction patterns taken with the Finch Camera:
(a) Single crystal of silicon carbide.
(b) Split shutter comparison exposure of gold and graphite for calibration against standard.
(c) Partly oxidised tin foil showing typical transmission pattern of a polycrystalline substance.

These reduced photographs of the diffraction patterns were reproduced from the original plates. Standard size $3\frac{1}{4}$ x $4\frac{1}{4}$ ins.

(Loss of detail is unavoidable during reproduction).

The discharge conditions in the cold cathode tube are inter-dependent, *i.e.*, the gas pressure, current passing in the discharge and accelerating voltage are related, so that any one is a function of the other two factors.[7] Use is made of this property in the stabilization. The gas pressure is kept constant by a special leak system and a circuit employing the saturation characteristics of a diode ensures a constant current, so that constant acceleration voltage is obtained with relatively simple equipment.

Diffraction Techniques. There are three principal methods of electron diffraction [3, 5, 6]:

(*a*) Transmission—in which exceedingly thin films of the specimen material are prepared from foils, by evaporation in vacuo, or by cathodic sputtering, etc.

(*b*) Reflection—in which the electron beam falls upon the specimen at grazing incidence, for the examination of specimens such as surface layers of massive material or very thin films.

(*c*) The examination of substances in the gaseous or vaporous state, providing an extremely useful method of analysis.

Methods of Measuring. In the interpretation of the diffraction patterns, the lattice parameters may be calculated within 2% from the constants of the apparatus, and the value of the accelerating voltage. This accuracy is dependent only on the measurement of the accelerating voltage. A salient feature of this instrument, however, is the fact that it is not necessary to attempt any absolute measurements involving these various constants, as diffraction patterns obtained from specimens under investigation may be referred to comparison patterns obtained from standard substances of which the lattice spacings are already known,[8] thus giving to electron diffraction techniques, the accuracy of measurement of X-ray diffraction. To facilitate this reference technique, a special split plate shutter has been designed so that separate exposures can be obtained in two independent halves, one from the specimen and one from the reference substance. The specimen carrier permits either substance to replace the other in the electron beam by simple manipulation.

The Finch Camera Made by W. Edwards

The electron diffraction camera and H.T. unit for the operation of the cold cathode source are based on the well-known and highly successful form and principles developed by Professor G. I. Finch. The camera equipment embodies the most modern vacuum engineering design, the various demountable joints being quickly and easily made. The joints and operating movements are effectively sealed with elastomer O-rings, metal bellows and Wilson shaft seals, thus dispensing with the grease-sealed mating surfaces hitherto employed.

The camera body is designed integrally with the supporting cabinet so that all services can be adequately and conveniently provided as a complete unit. A compartment, extending from the cabinet and running vertically behind the camera body, houses the cathode leakage, cooling water and electrical services to the upper camera body. The electrical instruments and controls are fitted to the sloping panel of the aluminium cabinet. The camera body and component parts are fabricated from non-magnetic material either solid or drawn or cast specially to ensure freedom from porosity.

The camera consists of four principal sections :

(*a*) Discharge chamber assembly
(*b*) Collimating and focusing section
(*c*) Specimen section
(*d*) Camera chamber

These sections are readily dismantled for cleaning purposes or for facilitating special alterations to suit particular work.

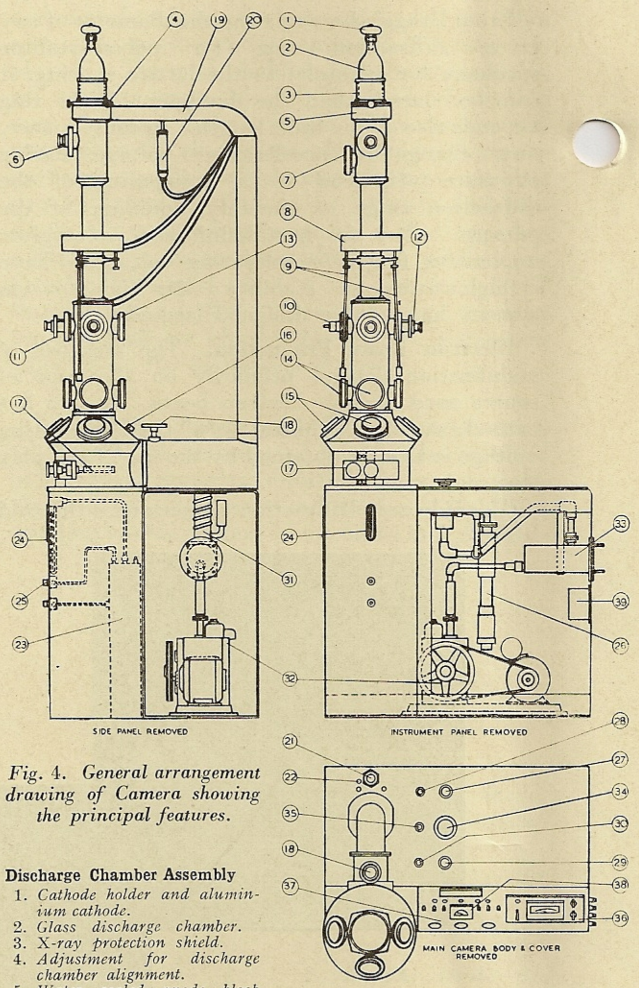

Fig. 4. General arrangement drawing of Camera showing the principal features.

Discharge Chamber Assembly
1. *Cathode holder and aluminium cathode.*
2. *Glass discharge chamber.*
3. *X-ray protection shield.*
4. *Adjustment for discharge chamber alignment.*
5. *Water cooled anode block containing anode diaphragm.*

Collimating and Focusing Section
6. *Collimating diaphragm holder*
7. *Inspection port.*
8. *Magnetic focusing and scanning lens*
9. *Scanning adjustment.*

Specimen Section
10. *Decharger spray gun (low power electron gun).*
11. *Diaphragm holder.*
12. *Specimen holder.*
13. *Inspection port.*
14. *Lower four ports at 25 cm. diffracted beam lengths.*

Camera Section
15. *Inspection window for visual observation.*
16. *Air admittance valve.*
17. *Photographic plate cassette.*

Vacuum Equipment and Components
18. *High vacuum isolation valve.*
19. *Cover for pumping, leak and water lines.*
20. *Pirani Gauge Head.*
21. *Discharge chamber pumping line.*
22. *Fixed leak to discharge chamber.*
23. *Leak reservoir.*
24. *Manometer for leak chamber pressure.*
25. *Isolation valve between leak line and chamber.*
26. *Discharge chamber diffusion pump.*
27. *Discharge chamber isolation valve.*
28. *Discharge chamber roughing valve.*
29. *Reservoir pumping valve.*
30. *Needle valve to increase reservoir pressure.*
31. *Diffusion pump for camera (60 lit/sec.).*
32. *Rotary pump (displacement 150 cu.ft/min.).*
33. P_2O_5 *moisture trap.*
34. *Rotary pump selector valve.*
35. *Air release to rotary pump.*
36. *Pirani gauge, for leak testing and recording of backing and discharge pressures.*
37. *Instrument panel for lens supply decharger, etc.*
38. *Focusing lens coil supply meter.*
39. *Water flow switch giving protection to diffusion pump in the event of water failure.*

Discharge Chamber Assembly. The glass cathode chamber is demountably but adjustably vacuum sealed with respect to the water-cooled anode block. This arrangement permits easy dismantling, for cleaning sputtered cathode material from the glass vessel, and facilitates the preliminary alignment of the electron beam. The aluminium cathode is removable from the top of the glass chamber, so that repolishing of the cathode can be carried out as required.

The discharge pressure is maintained at an easily adjusted constant value by a separate small oil diffusion pump and a fixed leak from a "leak chamber." The leakage rate is controlled by the leak chamber pressure which can be accurately adjusted by a fine control needle valve to raise the pressure, and a special valve and connection to an evacuated reservoir chamber to decrease the pressure, as required. The latter adjustment can be carried out without interfering with the pumping of the camera, i.e., while the H.T. is on. A small manometer indicates the leak chamber pressure and a Pirani Gauge in the cathode chamber pumping line indicates the discharge pressure. A steady and easily controllable discharge is obtained by the use of this arrangement. Provision is also made for using other gases in the discharge—to prevent, for example, oxidation of a delicate specimen.

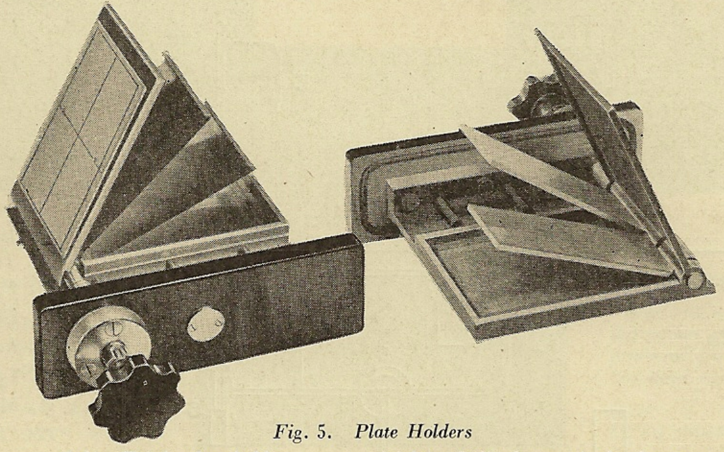

Fig. 5. Plate Holders

(left) Triple plate holder. *(right) Split shutter single plate type*

Collimating and Focusing Section. The beam is collimated by a fixed diaphragm in the water-cooled anode block and an aligning diaphragm, followed by magnetic concentration. The adjustable diaphragm is held in a demountable carrier which permits reciprocal and rotary motion. In this way, the beam containing high speed electrons, molecular rays and electrons which have lost energy by elastic collisions, can be inclined to the main axis of the apparatus and thus, with the concentrating field suitably adjusted and biased, the molecular rays remain trapped, but the electrons, both high and low speed, are released to pass into the specimen chamber in the form of a magnetic spectrum, the undesirable electrons being eliminated in the Specimen Section as described below. An inspection port is provided adjacent to the diaphragm port.

The focusing coil can be tilted with adjusting rods easily operable whilst viewing, to move the electron beam for the purpose already described, to direct it at any part of the specimen or to explore it ; the beam is focused on or just below the fluorescent screen by adjusting the field strength of the focusing coil. The focusing coil current is derived from a stabilized mains-operated power unit.

Specimen Section. Specimens can be mounted at two different levels to give diffracted beam lengths of 50 and 25 cm. At each level four ports are provided, each of which is designed to carry any of the following fittings in interchangeable positions as required :—

(*a*) A specimen carrier with movements permitting inclination of the specimen plane to electron beam, translation into beam, rotation in the azimuthal plane, lateral motion of the specimen plane at right angles to the electron beam.

(*b*) A diaphragm carrier with movements similar to the specimen carrier and carried immediately above the specimen for the filtration of the slower and unwanted electrons from the electron beam.

(*c*) A decharger (consisting, essentially, of a low-voltage electron gun for which the filament current and accelerating voltage is supplied from a suitable power unit) for neutralizing the charge acquired by non-conducting specimens, which otherwise result in distortion of the diffraction pattern[9].

(*d*) Inspection window.

Port blanks are provided which may be modified according to the special requirements of the individual research worker, for specialized work such as, e.g., fitting insulators to carry filaments for the evaporation of metals and salts, or thermo-couples, etc., into the specimen section. Specimens are mounted in interchangeable holders for the transmission and reflection techniques.

Camera Chamber. The plate holder is quickly demountable from the camera body, a rubber seal and quick-action securing bar being employed. The plate holders at present available are of three types—single plate holder for taking one exposure only; triple plate holder for exposing up to three plates without breaking the vacuum; a split plate shutter which permits the two halves of a plate to be exposed independently for comparison purposes. Each type has a fluorescent screen upon which the preliminary focusing is carried out. A roll film holder will shortly be available. Three inspection windows are provided for observation of the diffraction patterns on the fluorescent screen.

Evacuating System and General Accessories. Silicone or oil diffusion pumps can be installed as desired. Separate diffusion pumps are employed for the discharge and camera chambers, a common backing rotary pump being independent-

1. *Camera Body.*
2. *Air admittance valve.*
3. *High vacuum isolation valve.*
4. *Diffusion pump for camera.*
5. *Rotary pump selector valve.*
6. *Discharge chamber diffusion pump.*
7. *P_2O_5 moisture trap.*
8. *Rotary pump.*
9. *Leak reservoir.*
10. *Leak chamber.*
11. *Manometer.*

12*a*. *Isolation valve.*

12*b*. *Leak chamber pressure control & isolation valve.*

13. *Reservoir pumping valve.*
14. *Discharge chamber 'roughing' valve.*
15. *Pirani gauge head.*
16. *Needle valve.*
17. *Gas or atmosphere inlet to leak chamber.*
18. *Discharge chamber isolation valve.*

Fig. 6. Schematic diagram of pumping and control system.

ly mounted to eliminate cabinet vibration. The use of two diffusion pumps enables quick exhaustion to be obtained to the necessary high vacuum independent of the higher pressures prevailing in the cathode chamber. A feature of the evacuation system is the provision of high vacuum isolation valves by which the pumps, both rotary and diffusion, can, whilst still operating, be isolated from the camera when this is open to atmosphere for plate changing, etc. The time for exhaustion to operating pressures after specimen or plate changes occupies a few minutes only.

In the backing system a Pirani gauge head (operated from the same power unit as the gauge in the discharge chamber pumping line) is fitted, to indicate the pressure conditions and to carry out leak detection when required. The instrument panel accommodates the control and power unit of the sensitive Pirani gauge; focusing coil current control and meter, decharger filament switch and H.T. regulator; switches for diffusion and rotary pumps. All electrical systems are adequately fused and protected, and provided with panel indicating lights.

High Tension Supply. The constant current high tension supply system for the operation of the cold cathode tube follows Professor Finch's basic specification for the saturated diode circuit which has been so successfully employed by him, (see Professor Finch's various publications—Proc. Roy Soc. A. vol. 141, Page 402, 1933—Transactions of the Faraday Society, September, 1935, etc.).

A half-wave rectified high voltage supply is applied to a high voltage condenser. Current flows to the cathode of the discharge chamber through a diode, run under conditions of saturation, the filament temperature of which controls the high tension current.

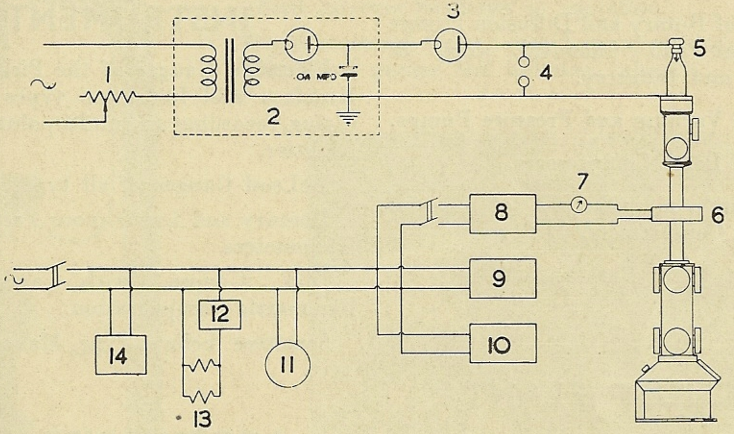

Fig. 7. Electrical circuit of the saturated diode system.

1. *HT Control.*
2. *HT Supply.*
3. *Saturated Diode.*
4. *Sphere Gap Voltmeter.*
5. *Cathode of Camera.*
6. *Focusing Coil.*
7. *Focusing Lens Meter.*
8. *Focusing Coil Stabilizer.*
9. *Decharger HT supply.*
10. *Decharger Filament Supply.*
11. *Pump Motor.*
12. *Flowtrol for Pump Protection.*
13. *Pump Heaters.*
14. *Pirani Gauge.*

The H.T. equipment has been designed for compactness and ease of operation and consists essentially of two parts : (*a*) the generator, comprising the rectifier and saturating diode valves, high tension and valve filament transformers, together with the condenser, all oil-immersed in a single tank ; (*b*) control table of the portable desk type, incorporating mains switch, KV control and indicator, and a spark gap for measuring the accelerating voltage.

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