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LEE, THE LOW-ENERGY ELECTRON-BOMBARDMENT MACHINE FOR VERY-HIGH-DOSE IONIZATION STUDIES

by

W. L. Primak and E. M. Monahan

ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

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ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue
Argonne, Illinois 60439

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Solid State Science Division

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PREFACE

Studies of the effects of ionization on materials have been in progress in this laboratory and its predecessors--the Chemistry Division of the Argonne National Laboratory and Section C-II of the Metallurgical Laboratory. The original studies in the Metallurgical Laboratory were concerned with the radiation chemistry of the coolant system of the graphite reactors. When the Argonne National Laboratory was formed, programs of studies of alkali halides were developed. Investigations of other materials like oxides, silica, quartz, and many others were begun. These programs originally were concerned with behavior in nuclear reactors. The phenomena found were complex, and it became clear that separate studies of the effects of the several kinds of radiation present in nuclear reactors were needed. These needs were met by acquiring a 60-in. cyclotron, by replacing a small van de Graaff machine acquired during the early 40's with a modern 3-MeV machine, and others. In addition, gamma sources and a soft x-ray machine were acquired. These served the needs of the investigators until several years ago when an interest in extremely high doses of ionizing radiation developed.

Among the problems brought to this group were consideration of the radiation damage which would be encountered in radioactive wastes and the radiation effects which would be experienced at the wall of a Tokamak fusion reactor. The doses involved were enormous--10^7 mega R. The only convenient source giving such doses for laboratory purposes is an electron source; an x-ray source has the disadvantage that only a small fraction of the energy used in the source can be converted into x rays. Direct bombardment by electrons also has the advantage that a fairly well defined range is obtained and the dose can be determined easily. To obtain the required dose in any reasonable time in an insulator, the energy may not exceed a few percent of of an MeV because of the problem of heat removal from the target. A machine with the required characteristics was not available. Since it seemed that all that would be required would be the mounting of a small electron gun in a suitable vacuum system and providing a secondary power supply with voltages comparable to those utilized in TV sets, it seemed that such a machine could be assembled in an ordinary laboratory.

LEE was realized although it took a few months longer than planned, mainly because the supplier of the electron gun and the supplier of the vacuum system parts failed to meet their promised schedules. It was hoped that the machine could be controlled completely by the electrostatic control elements in the gun. This was not possible, as will be described below, because of the lens effect of the secondary acceleration; magnetic control elements had to be added, and the beam line had to be extended. The machine has been operational for several years now with satisfactory results. For some special studies an intermediate speed mechanical shutter was planned, but was not built because those studies were halted. Other targetry for specialized applications are also planned. However, the basic aspects of the machine are regarded as completed; hence, it seems appropriate to present a formal description at this time.

We are indebted to George Mavrogenes for advice on control of the electron beam. Al Youngs advised us on the performance of the vacuum pumps and aided in their maintenance. He also advised us on corona leakage, a problem which arose with one of the leads from the 40 kV isolation transformer.

TABLE OF CONTENTS

LIST OF FIGURES

LEE, THE LOW ENERGY ELECTRON BOMBARDMENT MACHINE FOR VERY HIGH DOSE IONIZATION STUDIES

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ABSTRACT

The construction and operation of a low energy electron bombardment machine designed to study the effects of extremely high doses and dose rates of ionization on materials is described.

INTRODUCTION

The range of electrons at energies involved here is but a few microns. A major aspect of the study of materials which have been irradiated in this manner is an examination of the surface. Thus, the formation of deposits on the surface may vitiate the studies. The problem of surface deposition exists in all radiation studies, whether in a gas or in vacuo because compounds present in the gas phases may decompose and deposit on the materials. Thus, deposition has been found after irradiation in canisters[1] placed into reactors; and targets subjected to bombardment by ions or electrons may become covered with such decomposition products.[2,3] The vacuum system had to be selected and designed, therefore, to minimize deposition; and the electron gun could not be directed at the target, as material evaporated from it might condense on the target. If the system were to remain sealed for long periods of time, metal seals and ion pumps would have been feasible. However, this system was to be entered frequently for insertion and removal of targets. Although there has been some controversy on the subject, the experience here with turbomolecular pumps has, on the whole, been found to be satisfactory. Except for the few elastomer gaskets in the valves and on the seal-face of the pump, these were avoided to minimize the introduction of organic materials into the system. Electrical insulation of target sections which must be removed periodically are required. In another machine, glass insulators with indium gaskets were employed for a long time, but when these were replaced with Kel-F seals, no deleterious results occurred. This scheme was therefore used here.

The electron gun which was required was one which could give about 200 μA of electrons. A gun designed for surface studies, available from Varian, utilizes an uncoated filament as the thermionic emission source. Such a source would have simplified the vacuum system design by avoiding the necessity of activating the filament and then having to keep the gun in vacuo. Also, it would have minimized the introduction of foreign material into the vacuum system. It seemed to be the best of several units considered and was therefore chosen. The plan was to mount the gun at a small angle to the beam line, the maximum which the electrostatic deflection plates would deflect the beam, and thus prevent the evaporation of material from the gun onto the target.

The maximum acceleration voltage which could be used with this gun was about 3000 V. Thus it necessary to provide secondary acceleration for the electrons. Of the two choices, operating the target at ground or operating the gun at ground, the former appeared to be the better choice, as the delicate measurement in our experiments is at the target end, and this is where the beam would have to be controlled to perform the tasks at hand. The insulation for the gun was to be a section of Pyrex pipe. It was fabricated with ends suitable for an indium-tin gasket seal. The remainder of this section of the system was assembled with Conflat-style flanges utilizing copper gasket seals. A plastic box was built about the flange supporting the gun, the cables to it were heavily insulated with a plastic tube sheath, and the power supplies to operate the gun were place in a thick plastic box. Power was supplied by an insulated power transformer, placed into its own thick plastic box. Thus the gun and all of its supplies could be operated at -30 kV. This was the common for them.

The original plan was to obtain all of the beam control from the elements in the gun, and the original concern was that the control might not be adequate to prevent the beam from diverging after passing the electrostatic lens formed by the shield about the gun and the grounded vacuum system. Accordingly, the system was designed to make the beam trajectory as short as possible. In fact, what occurred was that this lens straightened out all of the electron trajectories, and this made it impossible to increase the divergence sufficiently for the beam to cover the targets when the electron energy exceeded about 11 keV. When the reason for the loss of control was appreciated, a magnetic lens was designed to focus the beam, and a drift tube was fabricated long enough to place the target section sufficiently beyond the focal point to permit the beam to diverge sufficiently to cover the targets. Now the control of the beam by the electrostatic elements in the gun was not adequate, hence external coils to serve as deflection coils were added.

A pneumatic gate valve was located at the end of the drift tube. The valve could be used as a beam shutter and served to close the vacuum system to the target section for target removal. This valve and the target section were electrically insulated from each other and from the vacuum system for the puspose of measuring target current and to permit these sections to be biassed to study and control secondary emission from apertures and the target plate. An aperture flange and a 4-electrode flange to sense the beam location are immediately below the gate valve. Plans for a movable aperture for multiple irradiations of a single target and a mechanical beam shutter for intermittent irradiations have been planned, but have not yet been realized. Because the gun is not affected by exposure to the atmosphere, and because the pump which was chosen is not injured by a vacuum break, it was not necessary to provide by-pass pumping to evacuate the target chamber after a target had been changed. The target chamber is merely reassembled; and the gate valve is opened, permitting pumping through the main high vacuum system. It was this simplification which, in part, dictated the choice of pumping station. Incidentally, the same procedure is used with the coating chamber described below which has a much larger volume.

Concern about charge storage and voltage "build-up" on insulators being bombarded with charged particles has been expressed by many workers, and various schemes for discharging the targets have been proposed and employed. In his ion bombardment work Hines emplaced a small thermionic emitter in his target Faraday cup to discharge the targets.[2] We did not make any provision for target discharge in the ion bombardment work done in This Laboratory. The evidence for problems from this source was occasional Lichtenberg discharges in our targets when, in our original investigations, they were mounted on aluminum discs with a vaccum wax. When we started mounting the targets in Woods metal, the problem disappeared. It was not until many years later that we found out, when investigating the secondary emission in our Cockcroft-Walton accelerator, that the secondary emission was discharging our targets. From the behavior of the secondary emission as a function of bias, the voltage to which the targets were charged could be estimated. While these voltages, about 1000 V, were not significant for that work, largely in the 100 keV region, they would be significant in the present machine. For this reason it has been regarded as necessary to metallize and ground the surfaces of the samples. To this end a coating chamber was required, and it seemed simplest to mount it on the same pump. Because some of the measurements required that both sides of the samples be coated, and others that face and edges be coated, it was decided to mount the samples on a rotating mount for coating, and for such a scheme horizontal evaporation seemed easiest. The coating chamber consists of a 4-in. Pyrex glass pipe cross. The sample mounting is accessible through the top. The filament or furnace source is on one end, and the thickness monitor and vacuum gauge is on the other end. The chamber is mounted atop a 6-in. gate valve. Since there is no valve on the electron bombardment section, it, too, is evacuated when the coating chamber is used. However, the coating chamber is not evacuated when the electron bombardment section is used.

DESIGN

1. General

The machine was designed as a vertical machine to permit the irradiation of samples without clamping them. A photograph of the machine is shown in Fig. 1. Most of the coating chamber can be discerned in the upper left. The electronics are mounted in two relay racks shown in Fig. 2. Most of the chassis and controls for the gun and the secondary acceleration are in the left rack. Most of the measuring equipment, the current measuring instruments and the integrator, the chart recorder, the controls for the coating chamber, and the film thickness monitor are in the right relay rack.

2. Vacuum System

As described above, the vacuum system was chosen as one in which deposition under irradiation would not be a problem, which was capable of rapid cycling, and would not be injured by a vacuum break or pump-down from atmospheric pressure. Another consideration was minimum maintenance: no low temperature traps. These requirements were met by the Sargent-Welch Model 3106D 400 l/sec Multiple Inlet Molecular Ultra-high Vacuum Pump Set. It had the further advantage that it was a unit in standard use in Building 211, there was a person trained in maintenance of these units, and spare parts for them were kept on hand. The pumping station was permitted to remain mounted on its shipping pallet elevated by placing 6 in. wooden cubes beneath it at its four corners.

The original intention was to purchase a standard 6-in. Conflat flange cross, to connect it to the pump by means of an adapter, to mount vacuum gauges opposite, to mount the gun above and the targetry below. In the course of inquiring about parts for this sytem, in particular, the gate valve to isolate the target chamber, we were advised that a custom part made to our specification would hardly cost more than the cross, and it became apparent that a considerable cost saving would result from fewer joints, adapters, and number of sealing gaskets. Accordingly, a unit was designed which matched the pump flange on the one side, was blanked off with a few small ports facing it for gauges, with a flange to fit the insulating section for the gun above, and a specially designed flange beneath. This specially designed flange could be used to make an electrically insulated vacuum connection either with a Kel-F gasket of special design or with a glass or ceramic ring and an indium or indiumtin seal, or it could be used to make a metal seal against a flat plate using a soft aluminum sheet gasket 20 to 30 mils thick. This flange was sized for 2 in. O.D. stainless steel tubing, 1/16 in. wall. The same flange design for 1 1/2 in. stainless steel tubing is used for our target chamber on the Cockcroft-Walton machine. An adapter 2 1/2 in. long from the 2 in. flange size to the 1 1/2 in. size was constructed to serve as a Faraday cup for the system and to permit interchangeability for targetry between the ion bombardment machine and this electron bombardment machine.

The drawings for the various parts described here are shown in Figs. 3-6. A reference lay-out is shown in Fig. 7.

The parts for the vacuum system were ordered from Thermionics Laboratory

Inc. rather than having them made in our own shops, partly because they would be furnished to us vacuum tested and cleaned, ready for assembly, work which we would have had to perform ourselves if they were made here. Unfortunately, they were poorly wrapped for shipment and some of the flange lips were dented. One was returned for refinishing, two others were stoned to smooth them.

3. Gun Mounting

Information was received from the manufacturer that there would be no difficulty in deflecting the electron beam 7 1/2 deg with the most distant electrostatic deflection plates within the gun. After considering several other mounting possibilities, such as a cap with a narrow insulating section, it was decided to mount the gun on a flat flange and use a 6 in. length of 4 in. Pyrex pipe as the insulator and have a hanging shield to prevent a wedge shaped electrostatic lens from forming between the end of the gun and the lower flange. The long insulator offered the possibility of another degree of freedom in case the gun was not mounted quite correctly on its flange. As it turned out, our guess about the location of the center of deflection of the beam used in designing the angled gun flange was quite accurate and would have brought the electron beam on axis. However, the manufacturer did not mount the flange at the specified angle; it was about 1/4 deg greater than it was supposed to have been. As a result, increasing the secondary acceleration voltage above 20 kV caused the beam to move off target by more than the controlling elements could compensate. This was corrected by having a 1/2 in. thick collar with a Conflat lip on each side machined to a 1/4 deg wedge. This collar withdrew the gun; and, by rotating the collar, an additional adjustment could be obtained. At present, increasing the secondary acceleration does move the beam somewhat, but within limits which can be managed by the control elements.

As described above, the insulator for the secondary acceleration voltage was made from a 6 in.-long section of 4 in. Pyrex pipe. The ends were ground flat and polished, and then the internal diameter was ground for a distance about 3/16 in. from each end to provide a fit with appropriate clearance for the metal flanges. To make the indium-tin solder seal (Cerroseal-35 solder; it is a fraction of the cost of indium metal wire) a length of the wire barely sufficient to encircle the lip of the flange is cut to length with a razor blade. The two ends of this length of wire are then joined by melting them with a fine pencil point soldering iron as they sit in a groove in a wooden board the radius of the wires, pressed lightly together with the fingers. The slight bulge formed at the joint is reduced with the aid of needle-nose pliers. The ring formed in this manner is then stretched very carefully over the lip, the Pyrex pipe is set atop it, held by the split aluminum flanges supplied for flat gasket connections, and bolted carefully in place using a torque wrench on opposite bolts. After the vacuum pumps are started and leak testing is begun, additional compression of the seal may be needed to reduce the leaking. These joints have to be checked occasionally, as they may loosen from vibration and plastic flow. Four jacking screws are provided in each plate for breaking the indium-tin seal should it have to be replaced.

4. Electron Gun

The electron gun is Varian model 981-2454 Auger gun. The unit has a metal shield and mounts on a Conflat flange for 1 1/2 O.D. stainless steel tubing, but the flange is bored out nearly to the diameter of the biting lip of the flange to accomodate the shield surrounding the gun. This gun is also described as a "glancing incidence gun" supplied for their surface analysis equipment. It uses a tungsten ribbon filament part 981-0248 in a replaceable pre-aligned assembly. The gun contains an extraction electrode, an einzel lens, and two sets of electrostatic deflection plates oriented perpendicular to each other.

5. Gun Electronics

The original intent was to purchase the control unit supplied for this gun by Varian, but since the control unit had to be modified to permit the beam deflection we required, and since it was very costly, because it was designed for the precise current control required for Auger analysis but unnecessary for the present application, we decided to construct the unit ourselves. This had an additional advantage, that the components could be arranged more satisfactorily for mounting in an insulated box made of 1/2 in. thick transparent plastic with plastic rod extensions for the control elements.

The circuitry for the electron gun is shown in Fig. 8. As can be seen, there are three power supplies. A 5 kV isolation transformer is used to protect the filament power supply from the 3 kV power supply used for the other gun elements. Both these power supplies are protected from the secondary acceleration voltage by means of a 40 kV isolation transformer located in a separate insulated box. Voltages for the gun elements other than the filament are obtained from a resistor string and controlling potentiometers. In addition, there is a 250 V supply to give positive voltage for the deflection plates. A stop is provided on the filament supply to limit filament current to 3.2 A. The filament voltage is adjusted from nil to whatever is required to give the desired current. The digital panel meter may be switched to give the primary accelerating voltage in units of 10 V or the gun anode current (by this is meant the internal current in the gun; there is no specific anode as the two extreme elements of the einzel lens are at the gun "ground" potential and the filament is at -3000 V relative to it) in units of 10 µA. Other controls are used to adjust the potential on the extractor electrode and on the focus element (this is the central element of the einzel lens). These potentials are normally set at maximum but may be changed slightly to control beam uniformity. Controls are provided for changing sign and potential on each set of deflection plates.

6. Secondary Acceleration

Secondary acceleration is provided by a Bertan 0--30 kV power supply. The positive terminal of this power supply is connected to ground, and the negative terminal is connected to the electron gun flange. An analogue meter on this power supply can be switched to read either voltage or current. Additionally, the total electron beam current can be read on a digital panel meter associated with the lower deflection coils supplies. The power supply voltage can be set in 1 V increments by step switches. A cylinder having a small clearance from the wall of the Pyrex pipe insulator is suspended from the upper flange (which holds the gun flange) and extends with 1/2 in. of the lower flange (which is at ground potential). This cylinder should minimize the divergence of the field.

7. Focusing Coil

This coil consists of 1600 turns of No. 22 enameled copper wire set in an ingeniously split (to permit assembly) pancake iron core, effective coil diameter 4 1/2 in. There is a construction defect in the coil resulting in a core to coil contact at about 5% from one end. Accordingly, it is necessary to energize the coil with a floating power supply. The coil core is clamped at the upper end of the drift tube. It is designed to focus the electron beam above the target to permit the beam to diverge to cover the target.

8. Deflection Coils

There are two sets of quadrupole deflection coils. The upper set consists of a pair of 6 in. diameter 100 turn coils of No. 30 copper wire in the direction the gun is tilted and a pair of 6 in. diameter coils of 200 turns each of No. 24 copper wire orthogonal to the first pair.

The second quadrupole consists of four coils 2 1/2 in. diameter, 200 turns each of No. 30 copper wire located 8 1/2 in. below the focusing coil. These coils can be used to sweep the beam across the target.

9. Deflection Circuits

The circuitry for the upper deflection coils is shown in Fig. 9. Four current regulator circuits, one for each coil, are energized by a common power supply. The polarity of each can be reversed. The current in each can be read in turn with the digital panel meter. The circuit for one of the Y-axis coils has a point marked A which is accessible for external connection. It will be indicated below that current in this Y-coil is required to deflect the beam to the target. When +5 V is supplied to point A, the current is turned off; hence this can be used to deflect the beam off-target. This technique may be used to deflect the beam from the target automatically when the desired integrated charge is reached or to provide intermittent irradiation by attaching a timing cicuit at this point. At present, the latter has limited usefulness for high current pulse bombardments as the beam tends to drift off-target after several pulses. A servo-system to keep the beam on-target has not yet been realized.

The lower deflection coils are currently used to sweep the beam over the target. For this purpose, two function generators which can provide square, sine, or triangular wave forms from 5 to 40,000 Hz are mounted in a panel. Controls for the one generator are on the panel face; the controls for the other are on the rear. The outputs are fed to a Crown 2-channel power amplifier whose output is used to drive the deflection coils. From the power amplifier the outputs are placed on a panel containing a digital panel meter which has been modified with an integrated circuit "true rms to dc converter" (Analog Devices AD536A) to permit it to read the power amplifier output voltages. The panel meter is also used as a dc instrument to read the total electron beam current (see above). The lower deflection coils are fed from this panel. The circuitry is shown in Fig. 10.

10. Targetry

The section below the drift tube is very flexible as it consists of a series of electrically isolated members. Currently these are in order: electrically actuated pneumatically operated gate valve, 4-section aperture (for servo-centering the beam), aperture plate and up to air valve, 2 1/2 in. long adapter from the 2 in. to the 1 1/2 in. flange. Currently, three target plates are available: a glass window with an electrically conductive transparent metallic oxide coating, a stainless steel blank-off flange, and a flange for determining the deflection of cantilevers by a capacitive measurement.

The 4-section aperture consists of a ring machined to mate with the Kel-F gaskets on both sides and within which are mounted 4 sections cut from a toroidal flat disc, each connected to a feed-through and intended for sensing the electron beam. It was intended to use this section for a servo-system connected to a set of deflection coils which would center the beam; but this system has not yet been realized.

The aperture plate is a ring similar to the above one, about 3/4 in. thick with an internal shoulder on each side. An aperture is fixed against each shoulder, the one shadowing the other in an arrangement to increase the pumping speed when a small aperture for the electrons is required. The aperture consists of a layer of graphite resting on stainless steel and on tantalum to minimize the generation of x rays and facilitate their absorption. This ring also contains an up-to-air valve to permit removal of the target and admit gas to the system when it is shut down. A deflection shield is located internally at the inlet to prevent destruction of the graphite aperture plates. The original aperture diameter was 5/8 in. For some experiments for which a smaller aperture was desired, an aluminum plate with a 3/8 in. hole was inserted in the assembly.

Several assemblies for holding samples on the glass window or the blank-off plate have been assembled from sheet aluminum or by spot welding thin stainless steel sheet. Evidence that samples mounted in them experience a considerable temperature rise (50--100 deg) on bombardment at high current has accumulated. A massive holder will have to be machined for these flanges, and a means for thermostating them will have to be provided. A coolant channel was built into the flange used for the cantilever bombardments.

11. Target Current and Charge Measurement

In most applications, the target plate and the adapter above it are electrically connected to form a Faraday cup configuration; and these are connected to the return ground through a Keithley 480 picoammeter. The analogue-retransmitting circuit of the picoammeter is connected to an integrator circuit connected to a counter to measure the charge, and the counter is connected to a relay circuit to trip a relay when the desired charge has accumulated on target. A circuit diagram is given in Fig. 11. No problems in measurement have been noted when circulating water directly from a thermostat through the target plate.

The aperture current is monitored by a second Keithley 480 picoammeter. Its analogue output is connected to a recorder to monitor the course of the irradiation.

OPERATION

1. Beam Characteristics

The beam current as a function of filament current is shown in Fig. 12.

The electrostatic deflection plate settings required to center the beam as a function of the secondary acceleration voltage is shown in Fig. 13. Note the change in the setting required in the plane in which the gun is tilted.

The focusing coil current required to focus the beam as a function of the secondary acceleration voltage is shown in Fig. 14. The current must be increased to expand the beam; *i. e.*, the beam must be focused to a point closer to the lens.

The voltages for the lower deflection coils vary only slowly with electron energy. When a focused beam is swept over a 1 cm path, the meter readings are between 0.03 and 0.05 V. The actual voltages are about 10 times these readings.

2. Vacuum Characteristics

When the system is pumped overnight, a pressure of 8 x 10^{-8} torr is reached at the gauge. The pressure may rise when the gun is actuated, but this falls after a time. The pressure may rise when a target is being bombarded. No effort has been made to bake the system to obtain lower pressures.

3. Shielding for X-ray Generation

Health physics surveys for external x rays were made 6/7/79 and 8/24/79. No radiation was detected from the upper part of the machine. No radiation was detected at 11 keV operation. At higher voltages, x-ray radiation was detected with the glass window in place. At 28 keV and a 2 μA spot, 80 mR/hr was measured. Lower values were found at the Kel-F seals. Shields made of 1/8 in. lead sheet were cut and covered with plastic tape. They were used to encircle the drift tube and the pertinent flanges. Observation of the window is always done from above using a mirror placed beneath the window. Bombardments with 28 keV electrons at 200 μA in which luminescence was measured have been conducted using the glass window. For this purpose, a lead shielded diagonal mirror was constructed which reflected the light into a 1/16 in. wall stainless steel tube 1 1/2 in. diameter. A lens whose position could be altered was placed within the stainless steel tube and, with the aid of a light located beneath the mirror, the target could be focused on a ground glass screen which was then replaced with a photodetector. There was no significant radiation in the operator location. No radiation has been reported on personnel monitor devices.

4. Operation Procedure

Determine the electron energy and current density desired. Insert the appropriate aperture. Attach the glass window flange with an aluminum annulus lying upon it whose diameter (or area) is known. Turn on the electronics to permit them to warm up. Turn on the pumps. When the pressure has fallen below 5×10^{-7} torr, the electron gun may be actuated. Set the gun voltage to 2.7 kV by the digital panel meter (in fact, this is 2.53 kV according to its calibration). Set the Bertan power supply (secondary acceleration voltage) to the required voltage less 2.53 kV. Increase the filament voltage to obtain a 10 μA (approximately) current reading on the Bertan power supply meter. Adjust the electrostatic deflection plate controls to bring the beam onto the aperture and window. If the electron energy is about 10 keV or greater, luminescence of the window can be observed when the room is darkened. Adjust the focusing coil current to give an aperture current about 1/5 the target current. Some adjustment for uniformity can be made with the gun focus and extraction controls. Additionally, sweeping voltage may be applied to the lower deflection coils. To obtain the beam area, photograph the window with a CRT polaroid camera with the aluminum annulus on the window illuminated. Then note the settings for each control element. Finally, turn the gun off, close the gate valve, pressurize the target area with argon gas, insert the sample mounted on the appropriate target flange, and open the gate valve. When the system is properly evacuated, restore the control settings and bombard the target.

5. Operating Characteristics

In bombardments which are continuous at low currents, very little drifting of the beam occurs; and it is slow enough that an occasional adjustment to maintain the aperture to target current ratio is adequate. When it was attempted to secure high current bombardments for 1/2 sec intervals every 5 min by deflecting the beam, it would drift off target after several pulses. The cause was not determined, but heating of the gun parts was suspected.

6. Target Exposure and Dose

The beam area is determined from a polaroid picture of the glass window. If the beam area is small, the print can be placed into our scanning microscope, and an enlarged tracing can be made on its associated X-Y recorder. The area can then be determined with a planimeter and compared with the area of the aluminum annulus enlarged in the same way. Alternatively, the beam and the annulus images can be traced with one of our coordinate comparators, and the data can be fed into our data logger for integration. If the beam area is large, the manual digitizer can be used instead.

Range data for electrons in the material of interest are taken from published tables and plotted on dual logarithmic paper to derive a power law for the range as a function of energy. This law can be inverted to calculate the range for the energy of the electrons employed. From the range, the energy, and the mean dose required, the incident charge desired is calculated. Then the current is decided upon, and the appropriate range is set on the Keithley 480 picoammeter employed to measure the current. The following adjustments are now made on the integrator panel: set the polarity switch to negative, and with the divider switch in the OFF position adjust the null potentiometer until counting ceases. Calculate the total counts required from the desired charge and the sensitivity of the integrator:

$$S = 10^{-4} R$$

where R is the full scale reading (amperes) of the picoammeter and S is given in coulombs/count. Dial the number of counts required with the appropriate divider switch setting on the controller. When the system is reset and zero'ed, the integrator is ready, and the bombardment may be started.

A relay trips when the integrator reaches the controller setting. This relay has been used to close the pneumatic valve. Unfortunately, its action may create an electrical disturbance in the lines and alter the counter reading. Accordingly, the arrangement described above, to deflect the beam by altering the current in one of the upper deflection coils was devised. After the beam is deflected, the beam current should be reduced manually by lowering the filament voltage.

Dose for ionization is given in energy absorbed per gram of material. The mean dose in the bombarded layer can be calculated from the energy of the electrons, their range, the incident charge, and the density of the material.

OPERATIONAL EXPERIENCE

The first electron beam was secured with LEE about two years ago. In the interim, various aspects of the machine have undergone development and modification, and working experience with it has been acquired. Although some elements of beam control remain to be developed (as described above) and some minor aspects of the vacuum system remain to be permanently installed (an additional up-to-air valve and forepump exhaust arrangements) enough experience has been gained in its operation to state that the basic elements of the machine are functioning satisfactorily and will probably remain fixed. During the past two years the machine has been used to study effects of ionization in a variety of materials: vitreous silica, several commercial optical glasses, several facsimile waste storage glasses intended for the immobilization of radioactive wastes, zinc selenide, sapphire, and magnesium flouride. As described above, these materials are of interest in fusion reactor program or in the radioactive waste storage program. Additionally, the results obtained for vitreous silica are of fundamental interest for understanding the effect of radiation on the silica structure.

All of the materials mentioned above were fabricated to form thin plates 1/4 x 5/8 in. and 10 to 15 mils thick. They were mounted as cantilevers with a free end. Upon irradiation, a deflection of the cantilever takes place if the bombarded layer experiences a dilatation; and from the measurement of the deflection, the dilatation can be calculated. These dilatations were measured as a function of dose. As yet, most of the results are reported only in internal correspondence.

Two samples of sapphire and one sample of vitreous silica were bombarded with electrons atop the glass window, and the luminescence was measured with a photodetector which was subsequently calibrated to determine the energy emitted by the luminescing target. The results are described in a forthcoming report.[5]

Blocks of vitreous silica and magnesium fluoride were bombarded through a small aperture. The effect on the optical absorption introduced into magnesium flouride and the destructive effects on the surface were determined. In the case of vitreous silica, which undergoes a contraction on irradiation, a plastic flow stress relaxation was found, the first time this has been found for ionization. The total contraction was experienced vertically, and the sample was stress free.[5]

In pursuit of these investigations, LEE was operated to give electrons of energies from 11.5 to 28 keV, ranges in the various materials from 1 to 7 μm, according to the requirements of the particular investigation. Currents as low as 1.5 μA distributed over a 5/8 in. aperture were used to minimize heating, and as high as 150 μA in a 3 mm diameter spot for 1/2 sec periods to attempt to reach the dose rates which would be experienced in the TFTR pulses. Bombardment at 90 μA over an extended period was employed to reach doses over 10^6 megaR.

APPENDIX I

SAMPLE PREPARATION

1. General

All of the samples which have been studied by electron bombardment in this laboratory have been non-conducting. They have included glasses, crystals, compacts, and inhomogeneous materials. They were prepared by optical workshop procedures: ground, lapped, and polished; or alternatively, they were lapped with successively finer diamond or alumina grits. Recently, glasses have been lapped and polished with 35% n-propyl alcohol-water media to minimize leaching. At higher alcohol concentrations, the pitch laps dissolved. The n-propyl alcohol was used because it has a boiling point close to that of water, hence the concentration does not change significantly during use.

To conduct the charge during bombardment, the surfaces were metallized. Three types of coatings have been used: chemically deposited silver, evaporated aluminum, and evaporated gold. Three coating configurations have been used. For chemical silvering, the sample was set on a glass plate after cleaning and covered with a greatly diluted Brashear's solution. Thus, all faces but the bottom were coated. The thickness of the coating was determined by weighing on a microbalance before and after coating. Reproducible weighings could be obtained if the samples were washed with absolute alcohol and air dried. For aluminum coating, the samples were cleaned ultrasonically with organic solvents, the final one being absolute alcohol. They were mounted as described below and were coated either on the opposite faces or on a face and two edges. The same procedure was used for gold coating. The disadvantage of the silver coating is that the silvering solution is aqueous and strongly alkaline and may therefore attack the sample. Also, the deposit may sometimes be granular, hence discontinuous, in the thicknesses employed, about 800 Å; and the thicknesses are difficult to control. The disadvantage of the aluminum deposition is that satisfactory coatings (highly conducting) are not obtained with some materials, and this is particularly true if the filament is overheated. Also, if it is desired to strip the coatings, the chemical solutions required may attack the sample. Gold coating has the advantage that it is readily stripped without injury to most materials merely by bringing the sample into contact with liquid mercury. However, to evaporate it economically, a furnace must be used; and this entails considerable heating of the chamber which raises the pressure because of outgassing.

2. Coating Chamber

A photograph of the coating chamber is shown in Fig. 15. As can be seen, it sits atop a 6 in. manually operated gate valve. It consists of a standard 4 in. Pyrex pipe cross. On the plate at the left are mounted the vacuum gauges, the Sloan monitor, and the up-to-air valve. The source is mounted on the plate at the right. The top plate is an access plate. The Pyrex pipe cross was prepared by lapping and polishing all of the ends to flat surfaces. The vacuum seals were made with Parker Viton Gaskoseals set in stainless steel annular plates. These were modified only by machining the internal diameter to that of the Pyrex pipe and then electropolishing them. They are used dry. The Pyrex pipe is clamped with the standard split aluminum flanges supplied for flat gasket seals. The end flanges were 5/8 in. thick stainless steel machined from 3/4 in. thick plate to accept the 1/4 in. bolts used with them. The elements which were used in the chamber, except for the sample supports and source shield, are all standard commercial components. The vacuum gauge is a Varian "Smart Gauge" which can be used to measure the pressure and hunt for leaks. The thickness monitor is a Sloan Digital Thickness Monitor DTM-200 which utilizes a quartz crystal oscillator. An Ultek ring holding an up-to-air valve and a thermocouple gauge is mounted on the same Conflat flange with the Smart Gauge. The source is mounted on a 1/4 in. copper rod 2-electrode flange sealed with a flat viton gasket. Nickel electrodes were machined for the copper rods to support the source: either a tungsten filament or, for the gold, a small furnace. Excessive outgassing occurred, particularly with the furnace, until the electrodes were cooled by water cooled copper clamps for the transformer leads. The furnace was made by winding stranded tungsten wire about a 3/4 in. length of 1/4 in. diameter alumina tubing, and then covering the wire and plugging one end with alundum cement. A short length of 65 mm Pyrex tubing surrounded the source, suspended on a spot-welded structure made of thin stainless steel sheet strips. One end was obstructed with a metal shield suspended on the source. On the other end, a shutter was balanced with a short length of permanently magnetized rod to permit its operation with an external small horseshoe magnet. Within the Pyrex tube shield are two wire supports into which a glass microscope slide can be slipped afresh for each coating operation, should this be desired, to provide a clear view of the source during preliminary heating. The shield arrangement protects the sample during preliminary heating and outgassing of the source and prevents excessive coating of the chamber. The shield can be removed for cleaning, obviating the necessity for frequent cleaning of the chamber. The source is supplied from a Signal DL 10-50 step-down transformer which can supply 100 A at 5 V. It is energized by a small variable autotransformer with a 5 A ammeter and a switch in its primary circuit.

3. Coating Chamber Vacuum

Within 20 min of starting the pumps, the coating chamber may be pumped down to $2x10^{-6}$ torr. According to the Smart Gauge, the nitrogen content of the chamber may be between 5 and 20%, showing that most of the gas present is caused by outgassing rather than by leaks. The pressure may be reduced to $8x10^{-7}$ torr when the system is pumped overnight. Typically, coating is started within 20 min of starting the pumps. Operating the filament source after metal is melted on it may cause the pressure to rise to $8x10^{-6}$ torr or higher. The use of the furnace will cause an even greater pressure rise, several time 10^{-5} torr or higher. This is caused partially by outgassing of the furnace, possibly decomposition of its material, but perhaps more by the heating of the chamber and its parts, as the furnace radiates more heat than the filament.

4. Sample Mounting

A nichrome wire gauze (cut form a piece of the material used to support beakers on ring clamps in the chemistry laboratory) was prepared for insertion at the lower end of the Pyrex cross to prevent material from falling into the pump. To it was spot welded a stainless steel clamp in which a piece of glass capillary tubing was held. This serves as the bearing to support a rotor mounted on 3/32 in. stainless steel rod supporting two small cylindrical permanent magnets. A variety of frames capable of supporting various kinds of samples can be attached to the rotor. The position of the rotor is controlled with a small external horseshoe magnet so that the faces or edges can be coated in succession without breaking the vacuum. One holder can support four cantilever plates, clamping them at one end, for 2-face coating. Another holder is a plate on which two samples can be clamped on each face, and then faces and edges can be coated in succession.

5. Coating Procedure

The samples are cleaned. They are mounted in the appropriate frame by an operator wearing gloves. They are then examined for dust particles. To minimize charging, the operator may wear a grounded wrist strap and perform the operations on a grounded conductive pad. It is advisable to discharge the samples with an "electrostatic gun" prior to mounting in the frame as it is more difficult to neutralize the samples when they are in contact with metal. Dust particles may be blown off with a gas stream or pushed off with a tool. The frame should then be placed in the oven to dry it before it is placed into the chamber. The rotor is removed from the chamber, the frame is inserted into a sleeve on the rotor in proper orientation, and the rotor is dropped into its bearing. The access plate is then placed onto its bolts (to locate it) which are then tightened with a torque wrench, gradually, to 20 in. lb. If the system is pumped down, the gate valve is opened gradually. If the system is at atmospheric pressure, the gate valve is opened, the mechanical pump is started; and when the pressure reaches about 10 torr, the turbomolecular pumps is started. The Smart Gauge rarely operating when it is turned on or when it is tapped according to its instructions. To start it, disconnect the Sloan Monitor cable and touch a Tesla coil to the chamber. After the pressure falls below 10^{-5} torr, the source may be warmed. This speeds pump down by warming the shield area and the source. It is best to raise the voltage to the source gradually because its resistance increases greatly with temperature, which increases the power in the source. It may be necessary to lower the source power several times and raise it again if outgassing is excessive. After this preliminary heating of the source, the shutter is opened, and the coating is continued until the desired thickness is read on the monitor. The variable transformer and its switch provides adequate control of the coating.

The monitor is more distant from the source than the sample location. If the thickness of the coating varies as the square of the distance from the source, the sample coatings should be 1.6 times the monitor readings. It has not been possible to check the film thicknesses by weighing, as the samples invariably lose weight when placed into the vacuum chamber and gain weight when they are removed. It was planned to determine the coating thicknesses by chemical analysis, but this has not yet been done.

After coating, if the vacuum is to be maintained, the gate valve is closed and argon gas is admitted to the chamber through the up-to-air valve. The samples are removed through the access plate.

6. Aluminum Coating

Although the source plate was designed to permit the mounting of two filaments between the electrodes and the plate (as a common), it has been more convenient to mount a single filament between the electrodes because the use of a double filament would require the removal of the plate for service. The filament is prepared by winding stranded tungsten wire about a form (a 3/16 in. or 1/4 in. rod) to give 2 or 3 loops. The ends are bent with a needle nose pliers to the configuration which will permit the filament to be inserted into the clamps with little deformation. The filament is then cleaned by electropolishing it in a sodium hydroxide solution. After clamping, a half dozen lengths of aluminum wire, each 3/8 to 1/2 in. long, are looped tightly about the filament with clean forceps. When the chamber is evacuated, the heating is started very gradually until the aluminum melts on the filament and spreads over it. At this point, outgassing ceases abruptly; and as soon as evaporation starts, gettering takes place, and the pressure drops. The amount of aluminum which can be evaporated from these filaments is quite unpredictable as it does not take place from the drops; rather, it occurs from the neighboring filament, which is hotter. Typically, if the filament is heated too rapidly, the aluminum is driven into two large drops near the respective clamps. These drops are so cool that evaporation ceases unless the temperature of the filament is raised greatly. If coating is continued by continuing to raise the filament temperature, high resistance coatings are obtained. Thus, if this condition is approaching, it is best to stop coating, pressurize the chamber, remove the electrodes, and add more aluminum. It will not do to add excessive aluminum, as this merely causes the large drops to form sooner. With a new filament, carefully manipulated, it may be possible to coat 4 faces to the typical thicknesses required, monitor readings of 140 to 200 Å. After several coating operations, it may not be possible to coat more than 2 faces with a loading of the filament.

7. Gold Coating

Gold coating with the furnace described above has been without incident. The heating is considerably greater than with a filament, and the pressure is much higher during coating, but all coatings obtained thus far have been satisfactory. Perhaps half dozen coatings, in each of which 3 or 4 sides had to be coated, have been performed with a piece of gold scrap wire perhaps half an inch long and 10 - 15 mils thick. At last viewing there appeared to be sufficient gold in the furnace to perform a number of additional coatings. This procedure is very economical in its use of gold.

REFERENCES

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3. W. Primak and J. Luthra, J. Appl. Phys., 37, 2287 (1966).
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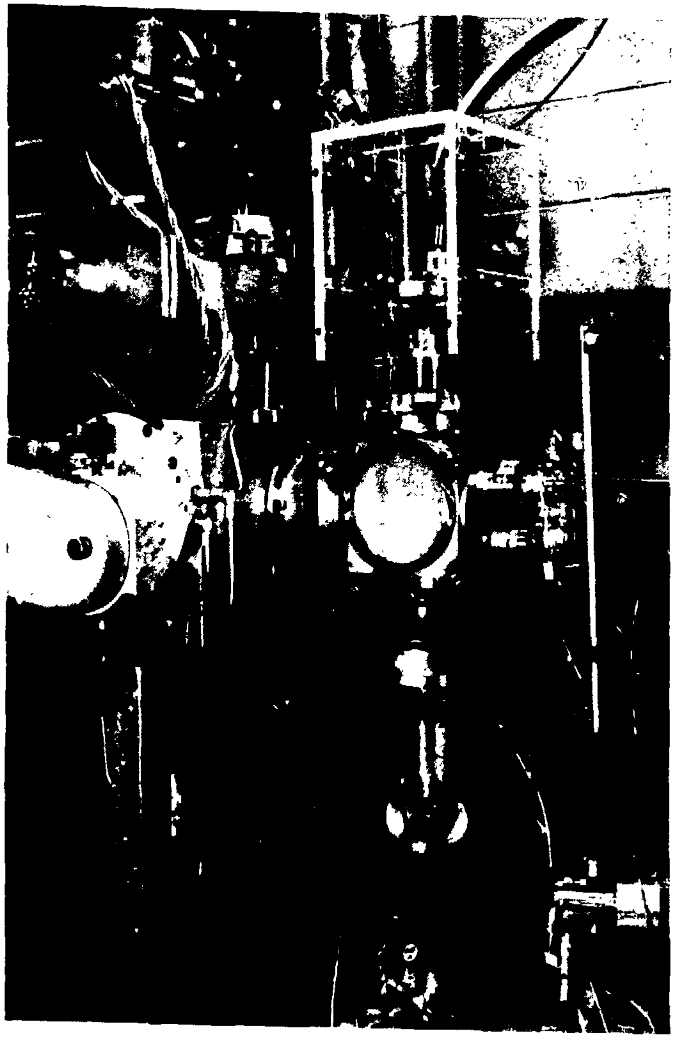

Fig. 1. LEE, the low energy electron bombardment machine.

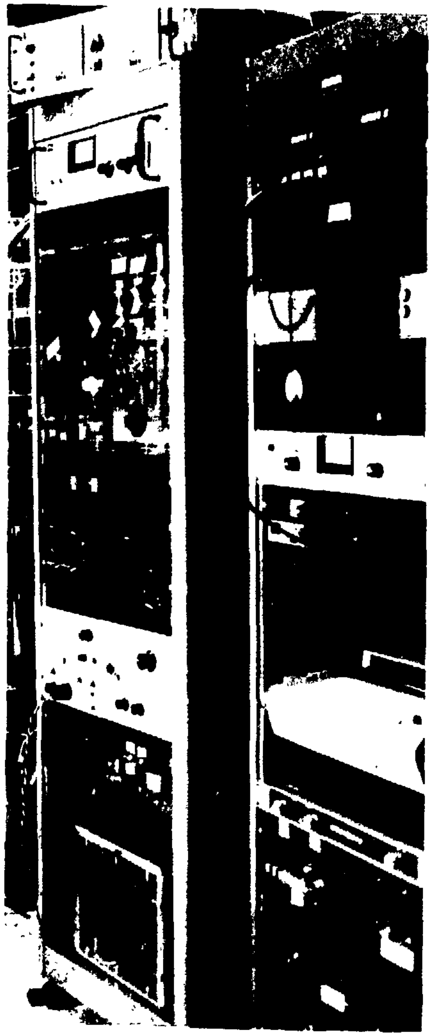

Fig. 2. Relay racks for operating LEE.

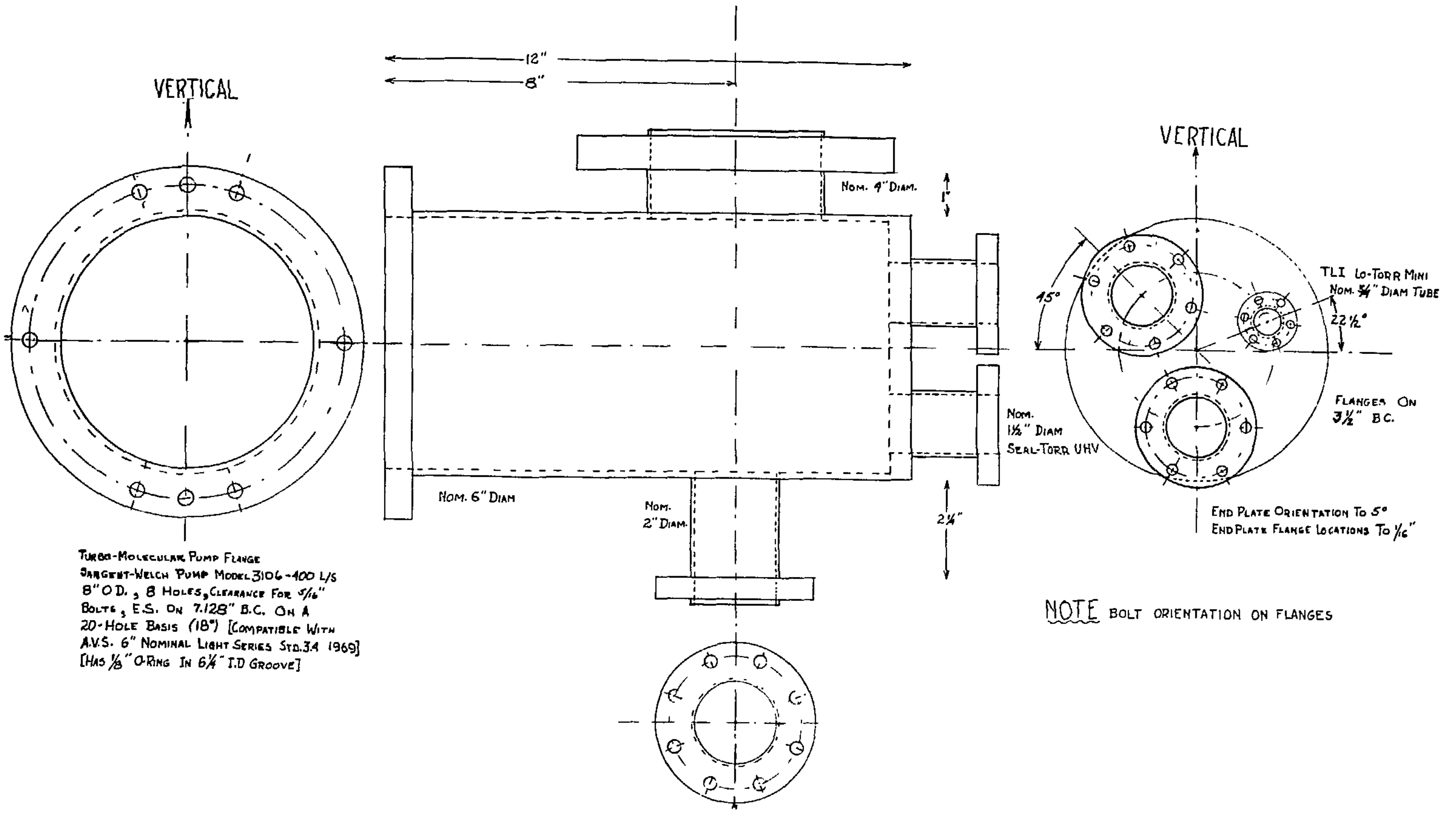

Fig. 3. The main chamber of LEE.

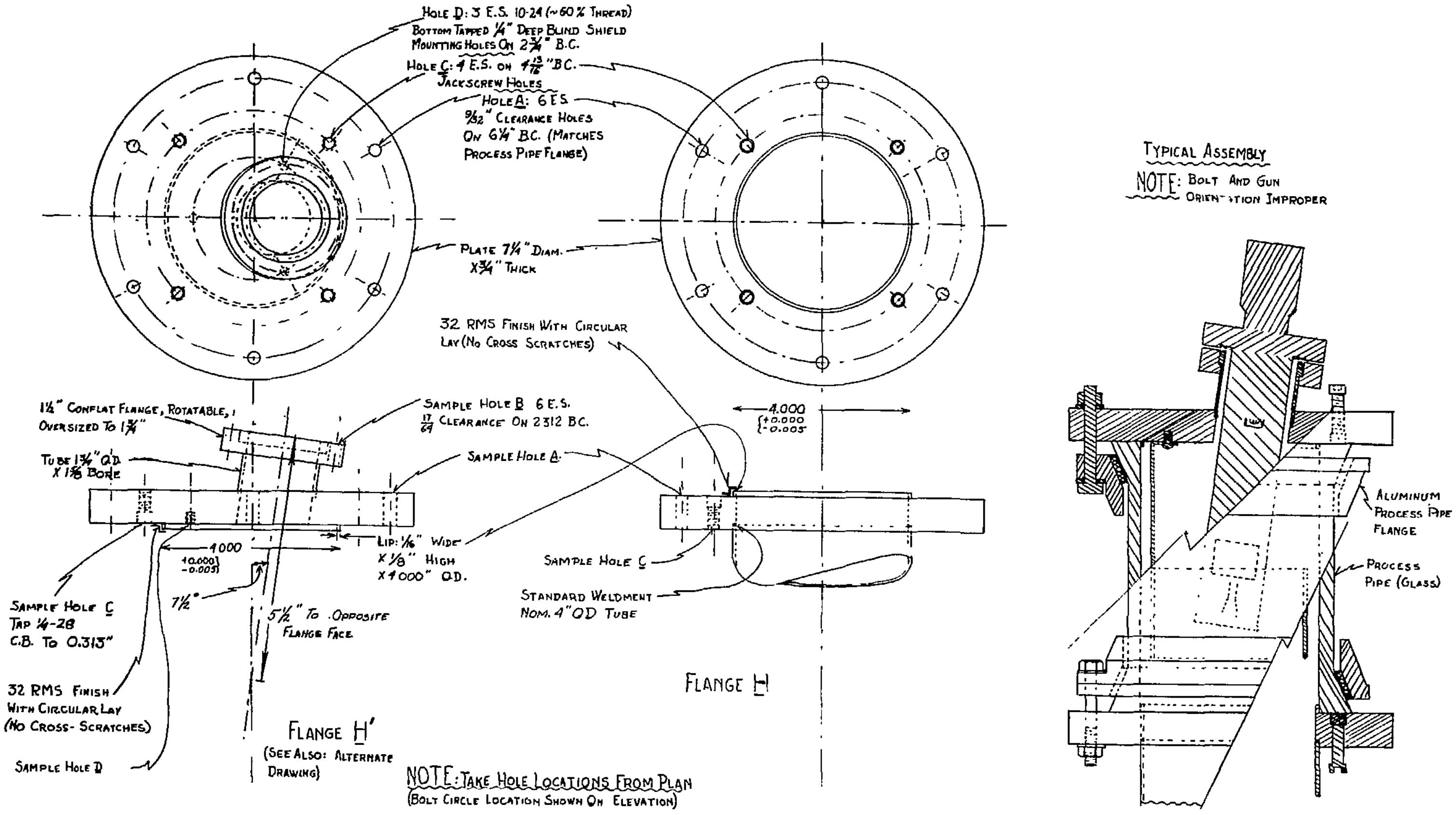

Fig. 4. The large flanges for LEE.

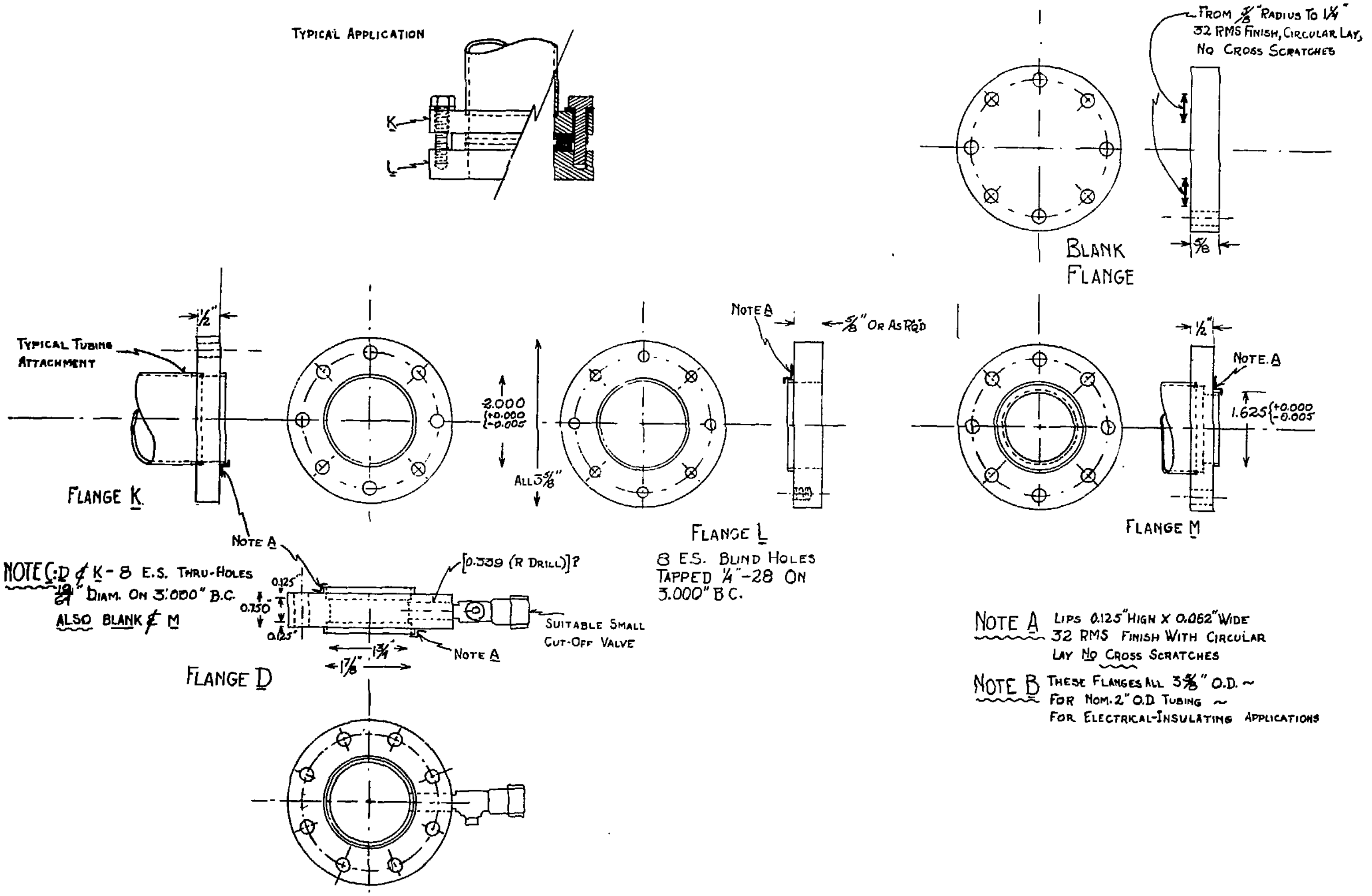

Fig. 5. Small flanges for LEE.

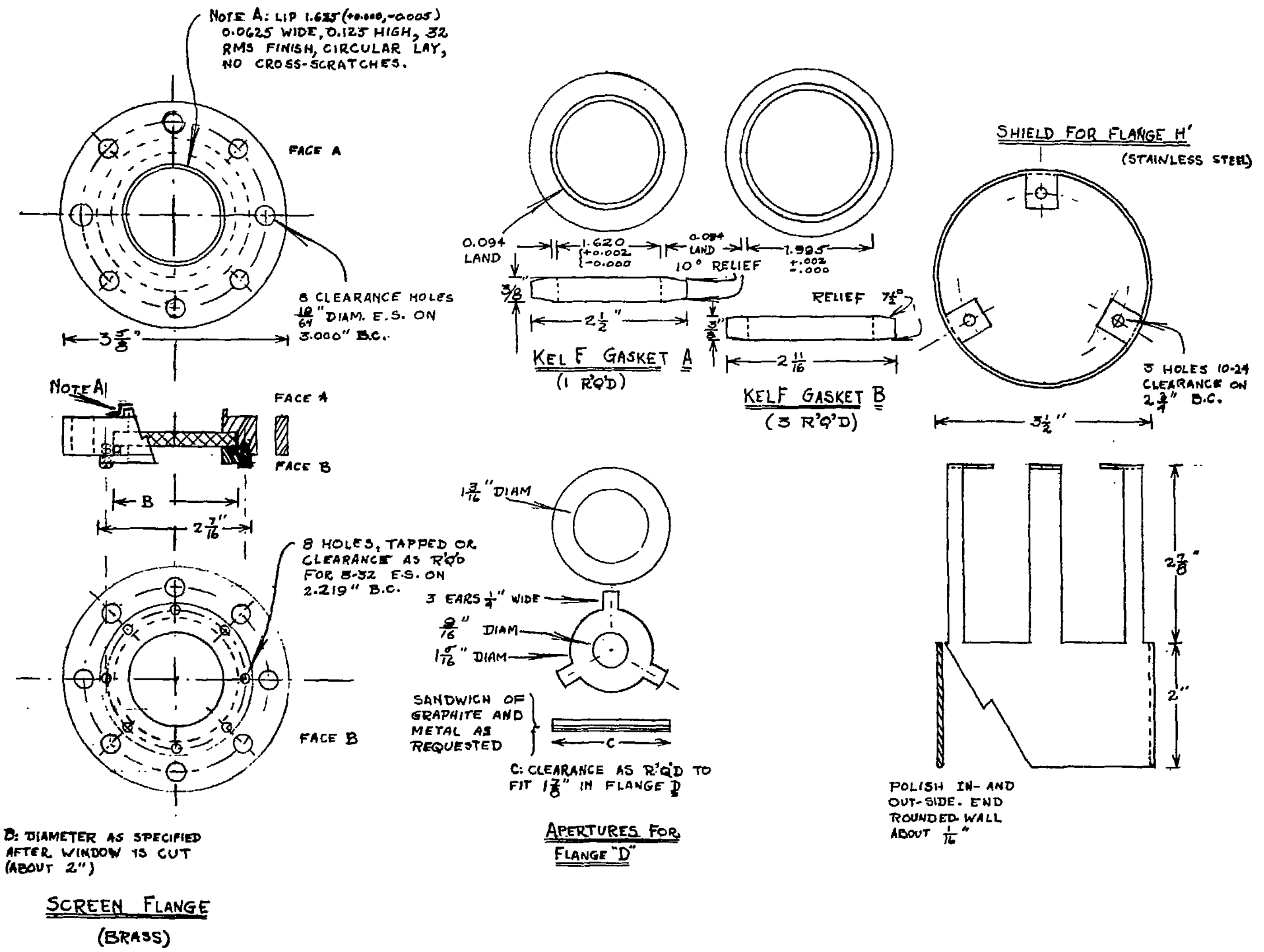

Fig. 6. Miscellaneous parts for LEE.

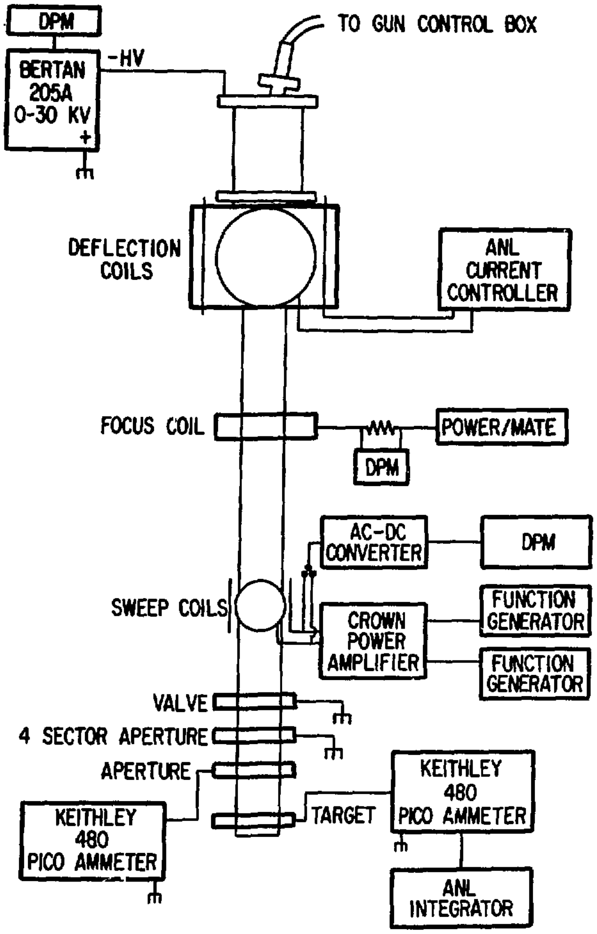

Fig. 7. Reference lay-out of LEE. The parts indicated are:

Electron gun: Varian Auger glancing incidence electron gun 981-2454, 3000 V, 200 μA.

High voltage power supply: Bertan 205A-30N, 30 kV, 500 μA.

Isolation transformer: Del AD3064,40 kV, 250 VA.

Deflection coil power supply: Quad constant current source.

Focus coil power supply: Power/Mate BPA-40D, 0--40 V, 2 A.

Sweep coil power supply: a BK Precision 3010 function generator and a Continental Specialties DM-2 function generator are fed into a Crown D-75 dual channel power amplifier.

Beam current meters: 2 Keithley 480 picoammeters.

Gun power supplies: custom assembled from Power/Mate BPA 100, 10 V, 5 A; Bertan 603-30N, 3000 V, 10 mA; and a 5 kV isolation transformer.

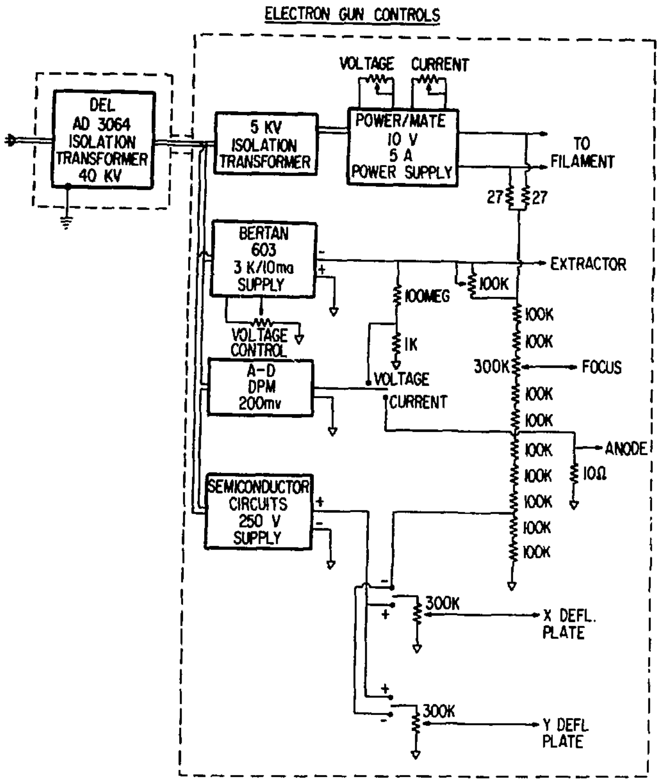

Fig. 8. Electron gun controls.

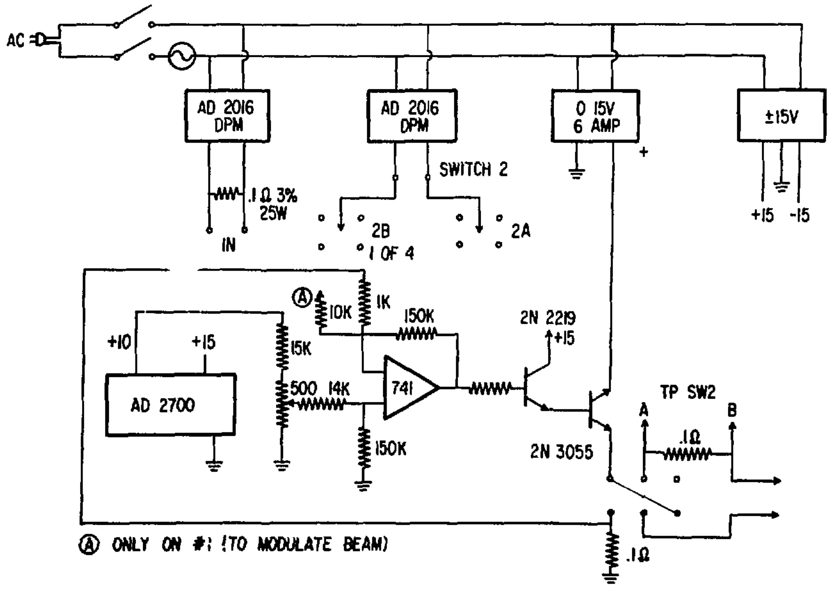

Fig. 9. Upper deflection coil circuits.

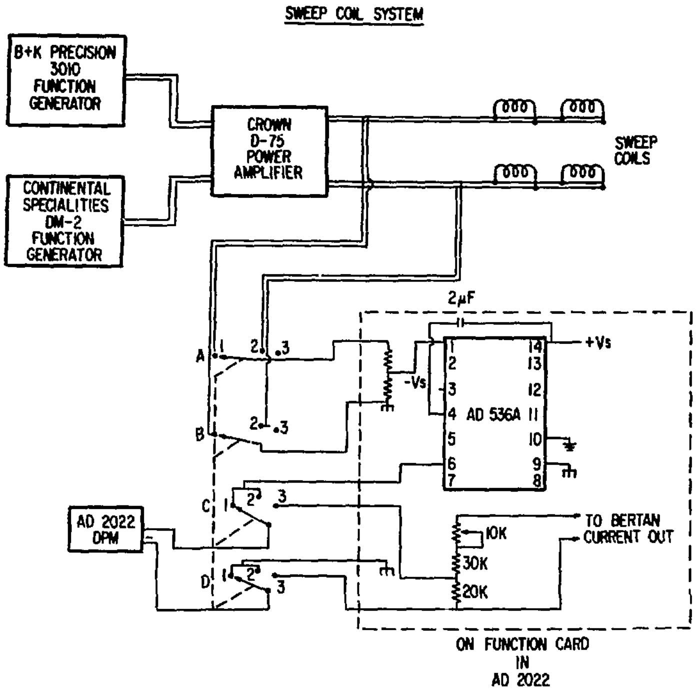

Fig. 10. Lower deflection coil circuits.

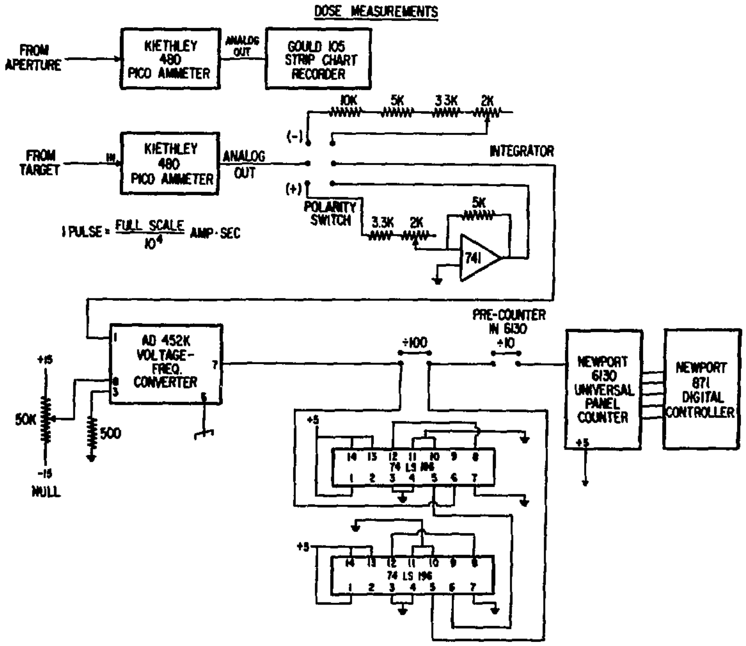

Fig. 11. Target circuits.

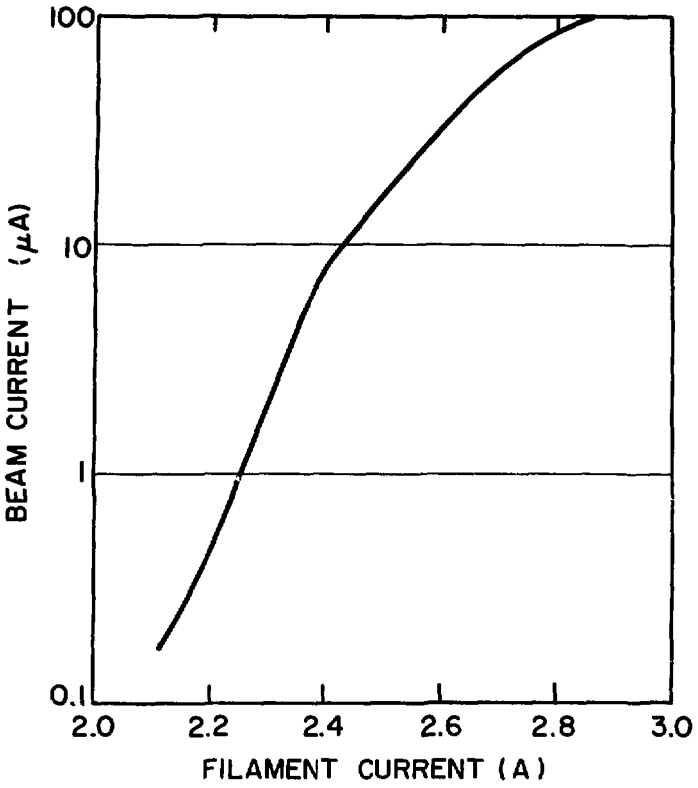

Fig. 12. Filament characteristic.

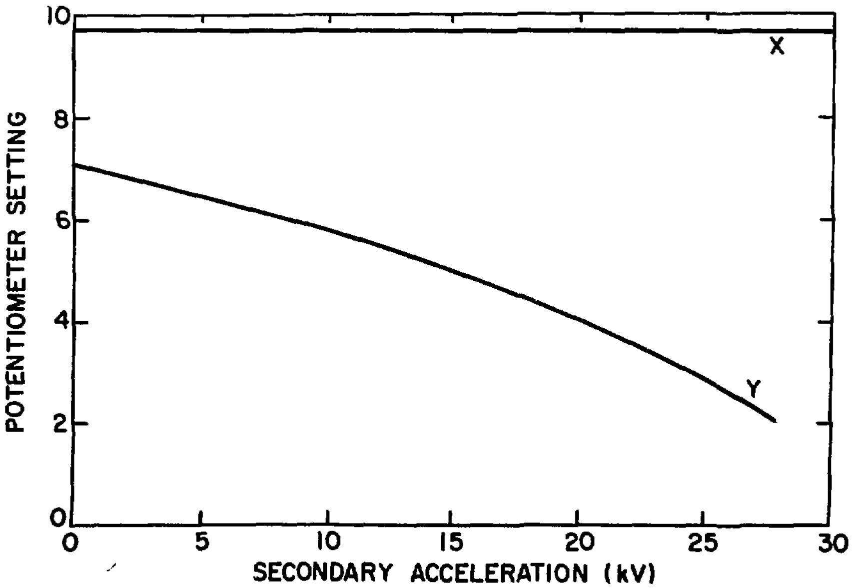

Fig. 13. Deflection plate characteristic.

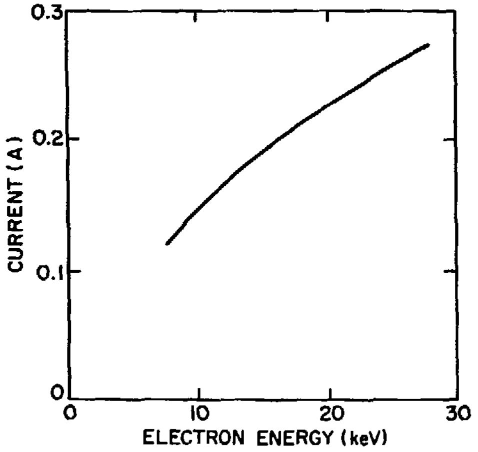

Fig. 14. Focusing coil characteristic.

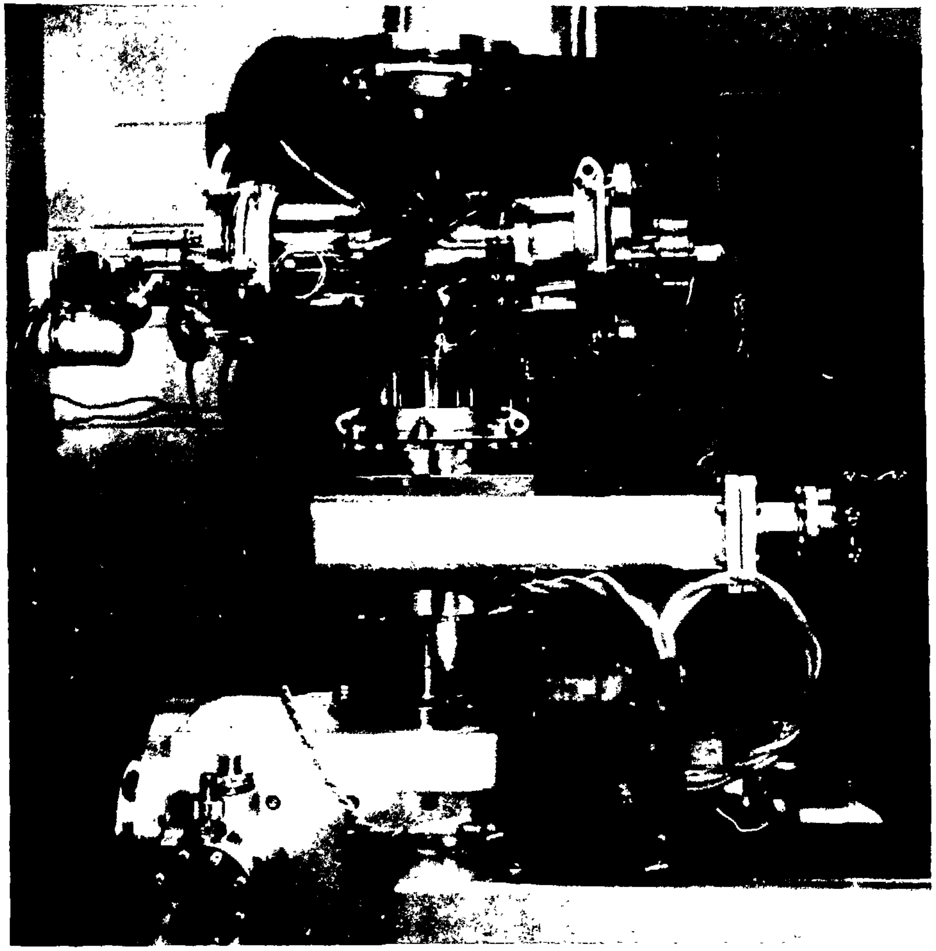

Fig. 15. The coating chamber.