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L. MARTON

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FOREWORD

"Ion Beam Technology Applied to Electron Microscopy" by J. Franks deals with the very interesting technique of thinning specimens by ion bombardment in order to make them suitable for electron microscope observation. After a review of the suitable ion sources, the sputtering process is investigated. The article ends with a discussion of applications in both transmission microscopy and surface scanning microscopy.

A. J. Davies examines the state of the art of microprocessors and their use in physics. The extremely rapid growth of this field, and the quasi-experimental reduction in size and cost, may make any review almost obsolete before it is written down. Nevertheless, the basic concepts do not change overnight and that is why this review should be useful to the reader in choosing and using a microprocessor in his own particular application in physics.

In our 19th volume (1964) we presented a review entitled "Endfire Antennae." P. A. Ramsdale has written, under the title "Wire Antennas," what might be considered a sequel to the earlier review. Although he comes to the conclusion that "the time when all forms of wire antenna can be said to be well understood and when useful new variants stop appearing looks to be well into the future," the review contains a very useful survey of how improved gain, greater bandwidth, and reduced size can be traded off against one another.

Metal–oxide–semiconductor field-effect transistors are the subject of a review by P. A. Muls, G. J. Declerck, and R. J. Van Overstraeten. They emphasize the importance of the effect of weak-inversion current on circuit characteristics and on the investigation of the physical properties of the interfaces involved. By using suitable models, theoretical characteristics can be derived and compared with actual performance data.

Our 34th volume (1973) included a review of metal–insulator–semiconductor varactors. The present volume ends with a review of "Modeling of the Transient Response of an MIS Capacitor," by T. W. Collins, J. N. Churchill, F. E. Holmstrom, and A. Moschwitzer. For such modeling the rate equations for hole, electron, and trap occupancy are coupled with Poisson's equation and lead to a computer simulation of the switching transient.

We expect to publish in forthcoming volumes the following reviews:

The Gunn–Hilson Effect	M. P. Shaw and H. Grubin
Solar Physics	L. E. Cram

Digital Filter	S. A. White
High Power Millimeter Radiation from Intense Relativistic Electron Beams	T. C. Marshall and S. P. Schlesinger
Auger Electron Spectroscopy	P. Holloway
Sonar	F. N. Spiess
Electron Attachment and Detachment	R. S. Berry
Electron-Beam-Controlled Lasers	Charles Cason
Amorphous Semiconductors	H. Scher and G. Pfister
Electron Beams in Microfabrication. I and II	P. R. Thornton
Design Automation of Digital Systems. I and II	W. G. Magnuson and Robert J. Smith
Magnetic Liquid Fluid Dynamics	R. E. Rosensweig
Fundamental Analysis of Electron–Atom Collision Processes	H. Kleinpoppen
Electronic Clocks and Watches	A. Gnädinger
Review of Hydromagnetic Shocks and Waves	A. Jaumotte & Hirsch
Beam Waveguides and Guided Propagation	L. Ronchi
Recent Developments in Electron Beam Deflection Systems	E. F. Ritz, Jr.
Seeing with Sound	A. F. Brown
The Edelweiss System	J. Arsac
A Computational Critique of an Algorithm for the Enhancement of Bright Field Electron Microscopy	T. A. Welton
Large Molecules in Space	M. & G. Winnewisser
Recent Advances and Basic Studies of Photoemitters	H. Timan
Application of the Glauber and Eikonal Approximations to Atomic Collisions	F. T. Chan, W. Williamson, G. Foster, aod M. Lieber
Josephson Effect Electronics	M. Nisenoff
Signal Processing with CCDs and SAWs	W. W. Brodersen and R. M. White
Flicker Noise	A. van der Ziel
Present State of High Voltage Electron Microscopy	B. Jouffrey
Noise Fluctuations in Semiconductor Laser and LED Light Sources	H. Melchior
X-Ray Laser Research	Ch. Cason and M. Scully
Ellipsometric Studies of Surfaces	A. V. Rzhanov
Medical Diagnosis by Nuclear Magnetism	G. J. Béné
Energy Losses in Electron Microscopy	B. Jouffrey
The Impact of Integrated Electronics in Medicine	J. D. Meindl
Design Theory in Quadrupole Mass Spectrometry	D. Dawson
Ionic Photodetachment and Photodissociation	T. M. Miller
Electron Interference Phenomena	M. C. Li
Electron Storage Rings	D. Trines
Radiation Damage in Semiconductors	N. D. Wilsey
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Particle Beam Fusion	A. J. Toepfer
Resonant Multiphoton Processes	P. P. Lambropoulos
Magnetic Reconnection Experiments	P. J. Baum
Cyclotron Resonance Devices	R. S. Symons and H. R. Jorg

The Biological Effects of Microwaves	H. Fröhlich
Advances in Infrared Light Sources	Ch. Timmermann
Heavy Doping Effects in Silicon	R. Van Overstraeten
Spectroscopy of Electrons from High Energy Atomic Collisions	D. Berényi
Solid Surfaces Analysis	M. H. Higatsbergar
Surface Analysis Using Charged Particle Beams	F. P. Viehböck and F. Rüdenauer
Low Energy Atomic Beam Spectroscopy	E. M. Hörl and E. Semerad
Sputtering	G. H. Wehner
Reliability	H. Wilde
Photovoltaic Effect	R. H. Bube
Electron Irradiation Effect in MOS Systems	J. N. Churchill, F. E. Hofmstrom and T. W. Collins
Light Valve Technology	J. Grinberg
High Power Lasers	V. N. Smiley
Impurities in Tokamaks	K. Bol
Visualization of Single Heavy Atoms with the Electron Microscope	J. S. Wall
Spin Polarized Low Energy Electron Scattering	D. T. Pierce and R. J. Celotta
Defect Centers in III–V Semiconductors	J. Schneider and V. Kaufmann
Atomic Frequency Standards	C. Audoin
Fiber Optic Communications	G. Siegel
Electron Scattering and Nuclear Structure	G. A. Peterson

Supplementary Volumes:

Image Transmission Systems	W. K. Pratt
High-Voltage and High-Power Applications of Thyristors	G. Karady
Applied Corpuscular Opticw	A. Septier
-coustic Imaging with Electronic Circuits	H. F. Harmuth
Microwave Field Effect Transistors	J. Frey

The advice and guidance of many friends and colleagues were again instrumental in putting together our present volume. Our thanks extend also to the devoted staff of Academic Press. By now they are in the front rank of the friends to whom we are indebted.

L. MARTON
C. MARTON

CONTENTS

Characterization of the MOSFET Operating in Weak Inversion

PAUL A. MULS, GILBERT J. DECLERCK, AND ROGER J. VAN OVERSTRAETEN

Modeling of the Transient Response of an MIS Capacitor

T. W. COLLINS, J. N. CHURCHILL, F. E. HOLMSTROM, AND A. MOSCHWITZER

Ion Beam Technology Applied to Electron Microscopy

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I. Introduction

The high resolution attainable with transmission electron microscopy (TEM), which can provide direct magnification up to 500,000 times, makes this an outstanding technique for examining the microstructure of materials. The thickness of the specimens must be restricted to 100–200 nm, however, in order to avoid undue absorption of the incident electrons. It has therefore been necessary to develop methods for preparing thin specimens of materials that have widely varying mechanical and chemical properties. A detailed review of preparation techniques is given by Goodhew (1972). Soft materials, such as biological specimens, may be prepared by microtoming, although difficulty is sometimes encountered when hard particles are present. For some metals, semiconductors, and other inorganic materials, chemical etching and electrolytic techniques are suitable. In one widely used method, the material is placed in a jet etching tank and the etching process observed through a lens with a light source behind the specimen. When the

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specimen perforates, the etching process is immediately arrested by flushing the specimen with an inhibiting wash. The areas round the perforation are usually sufficiently thin to allow micrographs to be taken. The etch process generally takes 5–10 min. Difficulties arise when materials are not homogeneous; preferential etching may occur, second phases may be leached out, and in semiconductors *p*-type material may etch at a different rate from *n*-type material. Even when a material can be controllably etched, the etchant may form a contaminating layer on the surface.

For materials for which suitable etchants do not exist, such as some glasses, ceramics, and geological specimens, various preparation techniques have been tried. The specimen may be crushed and fine slivers selected, or thin sections may be produced by very careful mechanical polishing (Barber, 1970). These operations require a considerable skill and can generally not be applied to brittle granular materials or materials with voids.

Castaing and Laborie (1953) developed a new preparation technique to avoid the difficulties they encountered when attempting to thin aluminum alloys containing 4% copper by electrolytic polishing. With the electrolytic technique an oxide layer formed and some redeposition of copper occurred, Al_2Cu precipitates etched slower than the matrix and gave rise to a relief structure, and as soon as a hole appeared, the edges dissolved rapidly leaving few thin areas. Instead of electrolytic polishing, they used a two-stage process involving mechanical polishing followed by etching of both faces of the specimen in succession with a parallel beam of 3000 eV ions. The resulting specimen was clear without any oxide film or deposits, the Al_2Cu precipitates thinned evenly with the matrix, the edge of the hole was not attacked at an enhanced rate and the rate of erosion (about 0.05 μm/min) could be controlled.

Later writers found that ion erosion was specially suited to "difficult" materials such as ceramics, glasses, and geological specimens (Bach, 1970a; Barber, 1970). Ion thinning equipment is now widely used in association with the transmission electron microscope in the study of nonbiological materials.

In scanning electron microscopy (SEM) surface features may be obscured as a result of mechanical treatment or chemical contamination. Recent work (Franks, 1977a,b) has shown that surface features can also be rendered clearly visible after ion treatment of the specimen.

II. Production of Ion Beams

A variety of mechanisms exist by which sufficient energy may be imparted to a gaseous atom or molecule to cause ionization. The gas may be ionized thermally, by a high voltage discharge, by a radio frequency field or by electron impact where electrons are derived from a heated filament, or by secondary emission from a cold cathode. These methods of producing ions have given rise to ion sources of various types (Carter and Colligon, 1968). The mechanism selected depends on the purpose for which the sources are to be used. At one end of the scale the requirement may be for beams of low intensity with small energy spreads and at the other end, for sources capable of producing very high intensity beams. For specimen preparation for electron microscopy, the ion source is used as a machining tool, energy spread considerations are of secondary importance.

Cold cathode ion sources of various designs have been used since the work of Castaing (1955a,b, 1956), Castaing and Laborie (1953), and Castaing and Lenoir (1954) in most specimen preparation equipments, because a useful output can be obtained from these sources that can be compact and, when well designed, need relatively little maintenance (there is no filament to burn out). Castaing's source (1955a, 1956) could even be conveniently mounted inside an electron microscope to monitor the thinning process.

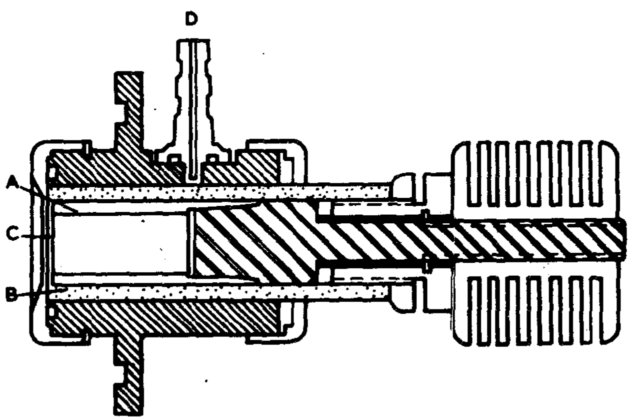

FIG. 1. Hollow anode ion source showing anode A, insulating tube B, cathode C, gas inlet D, and outer case for attachment to bombardment chamber (after Tighe and Hockey, 1969).

The sources favored by Castaing and many later authors (Paulus and Reverchon, 1964; Gentry, 1964; Abrahams *et al.*, 1968; Barber, 1970; Holland *et al.*, 1971; Heuer *et al.*, 1971) were developments of a construction originated by Induni (1947), who used it as cold cathode electron source for an electron microscope.

A construction of a positive ion source (Tighe and Hockey, 1969), is shown in Fig. 1. Gas flowed across the end of the insulating tube into the anode–cathode region. A simplified diagram of this hollow anode source is

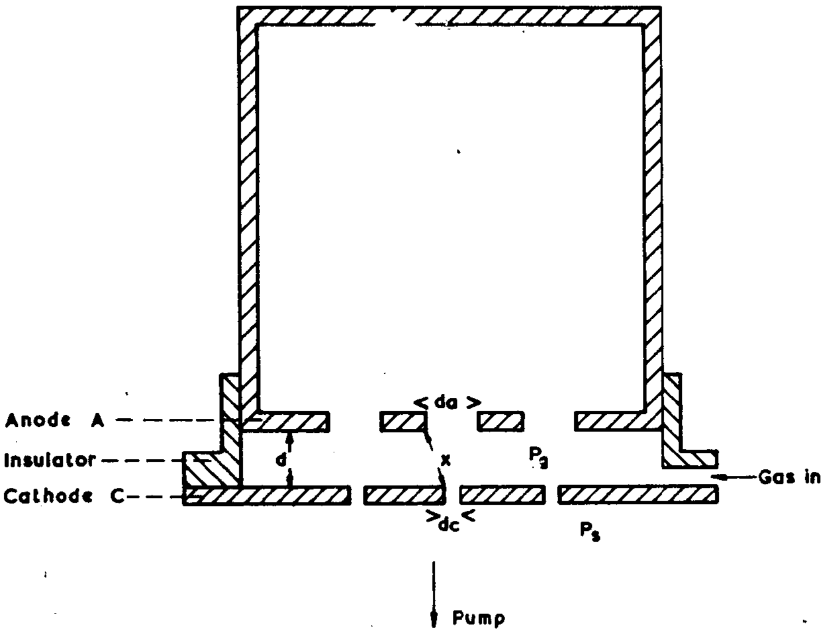

FIG. 2. Schematic diagram of hollow anode ion source. Distance between anode and cathode d, diameter of each anode aperture d_a, diameter of each cathode aperture d_c, P_g gas pressure in discharge region, P_s gas pressure in ion collection region.

shown in Fig. 2. The distance between anode and cathode was about 1.5 mm. According to Paulus and Reverchon (1961) the volume AC bounded by the anode and cathode forms the accelerating region, the distance between anode and cathode being smaller than the mean free path of the ions, thus the ions are all accelerated by almost the same potential drop, and energy losses due to collisions are practically negligible.

The anode was perforated in order to allow electrons from the cathode C to ionize the gas in the hollow anode region. The gas was introduced through a regulating valve to control the pressure within the ionizing chamber to the value required to maintain the intensity of the discharge, depending on the cathode aperture and pumping speed. For a pumping speed of 300 liter/sec, the total open area of cathode must not exceed 4 to 5 mm^2 divided into 25 apertures. Each cathode aperture (diameter d_c) was concentric with a larger anode aperture (diameter d_a). The efficiency of the source, defined by the ratio I_u/I_t, where I_u is the ion current emitted by the source and I_t is the total discharge current, was a maximum when d_a/d_c was about 4, independent of voltage and ion current or whether there were one or more sets of apertures.

This construction with an anode to cathode aperture ratio of 4, has been found most efficient by authors subsequent to Paulus and Reverchon (Gentry, 1964; Abrahams *et al.*, 1968; Barber, 1970; Holland *et al.*, 1971; Heuer *et al.*, 1971) and is also used in commercial equipment produced in France, the UK and the USA (details of manufacturers are given by Heuer *et al.*, 1971).

It is interesting to note, however, that Azam (1964) and later Ward (1971) dispensed with the anode perforations. Instead their anode consisted of an open-ended cylinder as shown in Fig. 3 inside a closely spaced cathode cylinder, the distance between the walls of the cylinder being such that at the operating pressure (10^{-1} Torr) the discharge potential was on the left-hand branch of the Paschen curve, above the applied anode potential. The cathode end plate contained a grid that could be plane, concave (as shown in the diagram) for producing a focused beam, or convex for a diverging beam. The gas pressure was chosen such that the discharge area covered the area of the grid but no more.

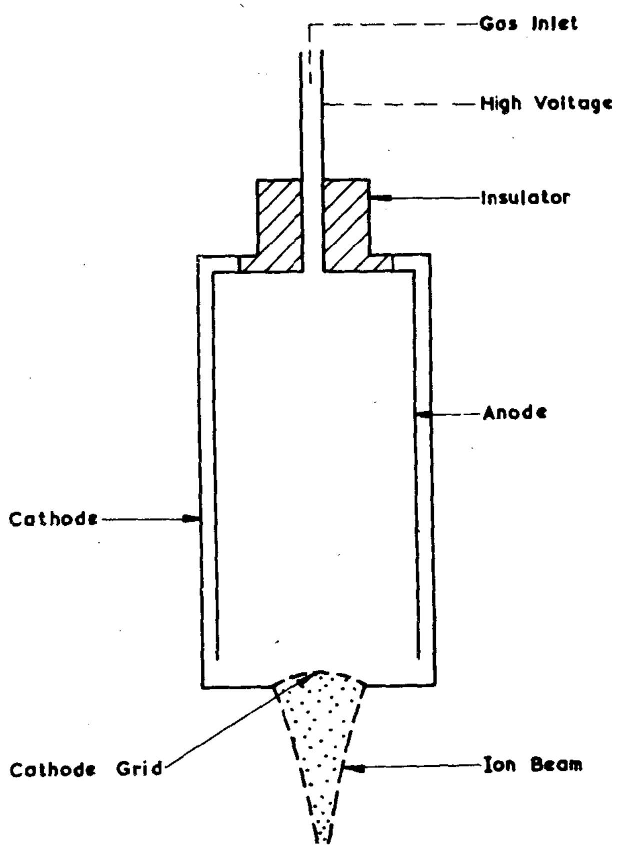

FIG. 3. Hollow anode source configuration (after Ward, 1971).

Holland *et al.* (1971) studied the operation of the Paulus and Reverchon source (1961) in some detail. Referring again to Fig. 2, the source was operated with an argon flow rate of 0.1 Torr liter sec^{-1}, the pressure P_g in the discharge region was about 0.4 Torr and the pressure P_s in the collection region was about 0.2 mTorr.

Figure 4 is a plot of the current flowing in the main discharge as a function of the voltage applied to the electrodes. The ion current at 6 kV, with the discharge current less than 1 mA was about 100 μA, giving an average current density of $\sim 300\ \mu A\ cm^{-2}$. To find the effect of the volume enclosed by the anode cylinder on the ion source operation, a plug was inserted into the anode cylinder, the gas being introduced across the end of the insulators. The anode was therefore in effect solid with short blind holes in the end facing the cathode. The solid plug had no measurable effect on the performance of the source, as shown in Fig. 4. Also, the output current–discharge current ratio remained the same over the pressure and voltage ranges tested.

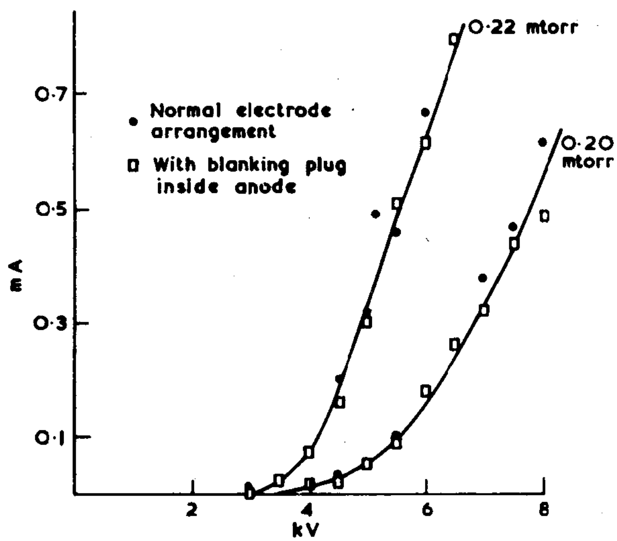

Fig. 4. Current–voltage plots for glow discharge ion source (Holland *et al.*, 1971).

Some measurements were made on the effect of varying the diameters of the anode and cathode apertures from the optimum ratio 4:1 (anode:cathode) with the anode and cathode holes the same size (0.02 in. diameter). The ion output current was negligible, the working chamber pressure increased from 0.2 to 0.5 mTorr and slight increases in either voltage or pressure caused uncontrollable increases in discharge current. With both anode and cathode hole sizes increased to 0.04 in. diameter, ion output currents of

50 μA were obtained, but the working chamber pressure was increased by an order of magnitude (2 mTorr) and the discharge could only run stably for less than a minute. The instability was characterized by a rapid transient increase of discharge current and a cone-shaped discharge extending into the vacuum chamber from one of the cathode holes.

These sources operated under the abnormal glow discharge conditions in which the cathode fall potential is current dependent. Thomson and Thomson (1933) obtained the following relationships for abnormal glow discharge conditions:

$$P_g d_s = A + BP_g j^{-1/2} \tag{1}$$

$$V = F_j^{1/2} P_g^{-1} + E \tag{2}$$

where V is the applied voltage, j the current density, d_s the cathode dark space, and A, B, E, and F are constants. For a discharge to take place, $d_s < d$, the separation of the electrodes. For simplicity, consider $d_s = d$.

For a uniform field between two parallel plates at a separation of d mm, Eqs. (1) and (2) give the corresponding conditions of pressure and density.

For a dark space $d_s = d = 1.5$ mm and $V = 6$ kV, then for argon gas and an aluminum cathode, Thomson and Thomson gave $A = 5.4$, $B = 0.34$, $F = 2.940$, and $E = 240$. The solution of Eqs. (1) and (2) gives $P_g = 0.37$ Torr and $j = 5.44 \times 10^2$ mA cm^{-2}. For a total discharge current of 1 mA, the corresponding active cathode area is therefore only 1.84×10^{-3} cm^2.

Holland considered therefore that for this ion source of several square centimeters in area, the discharge must be localized to account for the relatively low total current measured. Localization must occur in the regions where the ion beam is extracted. The normal distance of 1.5 mm between the anode and cathode plates was taken, to estimate the discharge operating conditions. The discharge path is greatest in the region of the perforations and will be roughly equal to x as shown in Fig. 2. The discharge will be confined to areas where the mean free path is sufficient for it to be sustained, i.e., to the region of the perforations, and this is also a region of intense field concentration.

The pressure of argon in the anode space was measured to be 0.42 Torr, and the mean free path for electron collision at this pressure at 20°C is 1.1 mm. Holland concluded that the volume contained in the cylinder is not necessary to sustain the discharge. The relative dimensions of the holes in the anode and cathode are important, however. As the cathode aperture becomes larger, and therefore x decreases, it becomes increasingly difficult for a discharge to be maintained.

Following the work of Holland *et al.* (1971), a simplified source was constructed by Crockett (1973) with the same electrode configuration as the previous types but from which the hollow anode construction had been omitted (Fig. 5). The anode hole diameter was 4 mm, the cathode hole 1 mm, and the electrode spacing 1.4 mm. Ion currents of up to 0.7 mA for argon and 2 mA for hydrogen were obtained with an applied potential of 8 kV. An analysis of the energy distribution of the ions with a parallel plate

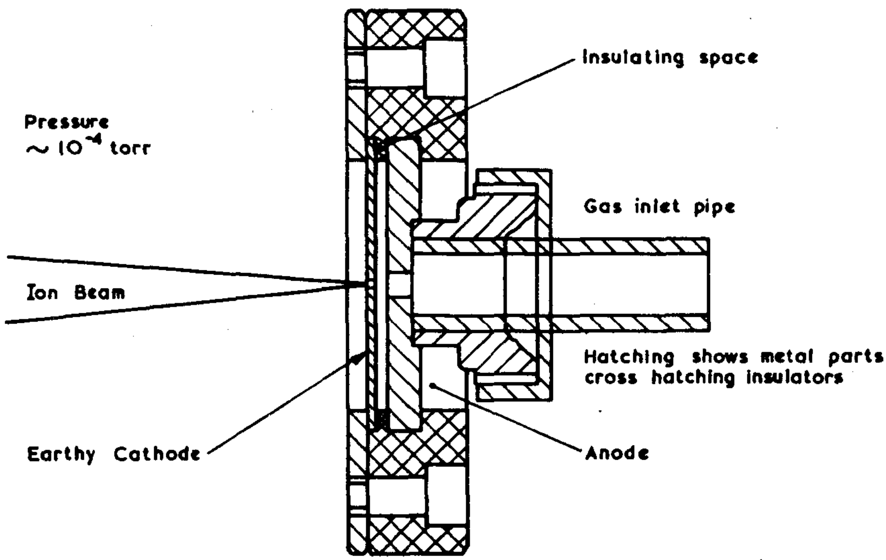

FIG. 5. Glow discharge ion source without hollow anode space (Crockett, 1973).

analyzer yielded two peaks (Fig. 6), the high energy peak occurring at the plasma potential V_c close to the anode potential, and the second peak at about 1/3 of the energy.

In Crockett's proposed mechanism, the electrons necessary to maintain the discharge are liberated from the cathode bombarded by ions and neutral atoms. Most of the electrons are released near the cathode hole through which many of the ions pass, because the ions are focused towards the cathode hole and collide with the sides of the hole at a glancing angle for which the secondary emission coefficient is high. The cathodic electrons accelerate along the field lines with a high probability of reaching the anode before suffering a gas collision. The mean free path of these electrons is estimated to exceed the gap separation of 1.4 mm from consideration of their energy and the interelectrode gas pressure.

An anode hole significantly larger than the cathode allows the high energy cathodic ions to strike the bore of the anode hole so that secondaries are emitted into a very low field. These anodic secondary electrons may