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A 30 keV ION BOMBARDMENT APPARATUS FOR THE STUDY OF THE INTERACTION OF LIGHT IONS WITH SURFACES

G. M. McCRACKEN
J. H. C. MAPLE
H. H. H. WATSON

Culham Laboratory,

Culham, Abingdon, Berkshire

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A 30 keV ION BOMBARDMENT APPARATUS FOR THE STUDY OF THE INTERACTION OF LIGHT IONS WITH SURFACES

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A B S T R A C T

An ultra high vacuum system is described which was designed to study particles emitted from surfaces under bombardment by hydrogen ions. Stress has been laid on obtaining pressures $\sim 10^{-9}$ Torr in the presence of the ion beam. The beam is mass analysed and its energy is variable from 5 to 30 keV. A maximum current of 300 μA over 0.1 cm^2 is obtainable at the target, so that a beam/neutral gas bombardment ratio of better than 1000 : 1 can be achieved.

A target mounting turntable is described which allows up to six targets to be bombarded in turn. A quadrupole mass filter is incorporated in the target chamber as a residual gas analyser and as a means of identifying gas species resulting from the bombardment of surfaces.

UKAEA Research Group
Culham Laboratory,
Nr. Abingdon,
Berks.

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C O N T E N T S

1. INTRODUCTION

One of the difficulties involved in nuclear fusion research is the contamination of hot plasmas by impurities from the wall. Even when an attempt is made to magnetically confine the plasma, there is always some drift of charged particles across the magnetic field and the escape of fast neutrals which can bombard the wall of the vacuum chamber. There are three main processes whereby secondary particles can be emitted from surfaces under bombardment by fast ions or atoms:

(1) Sputtering
(2) Desorption of gaseous impurities
(3) Re-emission of incident ions

Some information is already available on the sputtering effects of hydrogen[1] but very little information is available about either of the other two processes. The present apparatus, Fig.1, was designed to investigate these processes and to determine under what conditions plasma contamination could be minimized.

2. VACUUM SYSTEM

In undertaking surface experiments of this kind one of the most important problems is that of maintaining surface cleanliness. In many sputtering investigations using heavy ions, clean surfaces are maintained at relatively high background gas pressures by using large ion current densities[2]. In the case of light ions like hydrogen and helium where the sputtering coefficient can be in the range 0.01 to 0.05 this is not feasible, and a much lower background pressure must be maintained. Balancing the difficulties of producing a high beam current density and a sufficiently low pressure in the presence of the beam, it was decided to use a beam current of ~ 100 μA over 0.1 cm^2. In this case a background pressure of 3×10^{-9} Torr would give a beam/neutral gas ratio of 1000/1. The problem of surface contamination by hydrocarbons of high molecular weight, and the probability of their conversion to carboniferous layers under ion bombardment, leads to the criterion of a partial pressure of $< 10^{-10}$ Torr for these impurities. Even lower hydrocarbon partial pressures must be maintained if initial conditions during pulsed bombardment are to be studied. For these reasons a vacuum system was built which was bakeable from source to target chamber. Getter ion pumps were used wherever possible and the use of oil diffusion pumps and elastomer seals was entirely eliminated.

The vacuum walls were made of 18/8/1 titanium stabilized stainless steel which was internally electropolished in the ultra high vacuum regions and elsewhere vapour blasted with alumina powder.

Differential Pumping

The ion source used was of the R.F. discharge type, principally because of its simplicity and reliability and the large proportion of H_1^+ ions obtainable from it. Because this source is operated at pressures ~ 10^{-2} Torr a four stage differential pumping system was designed to maintain the target chamber at $< 10^{-9}$ Torr when the source was operating. The arrangement is shown in Fig.2. The main gas load is taken by a mercury diffusion pump with a nominal speed of 2450 ℓ/sec for hydrogen when operating with its liquid nitrogen trap. This pump will maintain a pressure of 2×10^{-6} Torr (N_2 equivalent) when the source

is operating at 2×10^{-2} Torr. It is backed by a 2 inch mercury diffusion pump and a rotary pump, with a liquid nitrogen trap designed for viscous flow between the two.

In order to minimize gas flow from the einzle lens chamber into the analyser tube the beam is focused and directed through a 5 mm. dia. hole. Beyond this point the system is pumped by sputter ion pumps. Two ion pumps each of 270 ℓ/sec for hydrogen are placed on either side of the analyser to cope with the load due to neutral gas from the einzle lens chamber and to those ions which are deflected into the vacuum walls by the magnet. They are able to maintain a pressure of $\sim 1 \times 10^{-8}$ Torr in the quadrupole lens chamber with the source at 2×10^{-2} Torr but without the beam, and $\sim 10^{-7}$ Torr in the presence of the beam. A second differential pumping port is arranged between the quadrupole lens and the target chamber with a conductance of 30 ℓ/sec for hydrogen.

Target Chamber

A beam current of 100 μA protons is equivalent to 10^{-5} Torr litres/sec if it is all converted into gas, and hence constitutes the major gas load in the target chamber. In order to provide the necessary pumping speed to keep a pressure of $< 10^{-9}$ Torr a titanium sublimation pump was designed as an integral part of the target chamber. The substrate consists of a hollow cylinder 30.5 cm I.D., 35.5 cm O.D. and 38 cm long which is filled with liquid nitrogen. Titanium is evaporated from a Ti 15% Mo hair-pin filament 2 mm dia. and 25 cm long supported along the axis of the cylinder[3]. Batch evaporation is used, covering the inside liquid nitrogen cooled surface with an average of $\sim 5\mu$ gm/cm^2 of titanium at a time. The pumping speed has been measured by comparing it with an orifice of known conductance[4]. An appreciable degree of additional pumping appears to be due to physical adsorption on the cooled stainless steel not covered by titanium. A total speed of up to 50,000 ℓ/sec was measured immediately after firing the getter; this falls to ~ 15,000 ℓ/sec when a total of 0.5 Torr litres of hydrogen have been absorbed. This means that with the gas load due to 100 μA beam adequate pumping speed can be maintained for about 10 hours. An ion pump of 270 ℓ/sec for hydrogen is used to supplement the sublimation pump for those gases, particularly the rare gases and methane, which do not react chemically with titanium.

For the initial pumping of the system and during bakeout the target chamber is pumped by a small mercury diffusion pump which is afterwards valved off by a 2 inch bakeable metal valve. After initial assembly, the whole apparatus was baked to 400°C for 48 hours. Following subsequent exposures to air, ultimate pressures of $1\text{-}2 \times 10^{-10}$ Torr are obtained in the target chamber after baking it to 250°C for 36 hours.

Demountable Vacuum Joints

Since a large number of demountable joints were necessary on the system (varying in size from 2.5 - 40 cm. bore) the choice of a reliable seal geometry was of great importance. At the time of the original design of the apparatus, the most thoroughly tested type of seal for which adequate design information was available appeared to be the double corner seal developed for the Model C Stellarator[5].

In this type of seal gold wire gaskets are used, and cantilever bending of the flanges produced by compression of the sealing gasket is prevented by the use of a second

similar gasket, located outside the pitch circle of the bolts. The radial width and thickness of the flanges and the necessity for high bolting torques are minimized by using close pitched bolts of small diameter. These are made from a high creep strength austenitic alloy, AD 776, having an expansion coefficient close to that of the stainless steel flanges. It has been found important to stress relieve all flanges after welding and before final machining. Turned finishes up to 30 μ ins C.L.A. surface roughness have been found adequate for successful seals.

Whilst this type of seal has indeed been found to be extremely reliable and trouble free, operational experience has shown that for flanges having bores less than about 10 cm diameter, the complication and expense of making provision for the outer balancing gasket appears to be unnecessary. The gold wire gaskets have been found to be very satisfactory being easily made by fluxless welding, easily removed from the flange after use, and insensitive to the choice of bakeout temperature. In operational cost they compare favourably to flat copper gaskets, (which become increasingly uneconomical as the size increases) because of the high recovery value of scrap gold.

Gas Inlet System

In order to calibrate the mass filter in the target chamber the earlier gas inlet system[4] has been modified to allow absolute measurement of the inlet pressure with a mercury manometer. Hydrogen is introduced into the system through a palladium leak and flows into the target chamber through a porous plug made of sintered silicon carbide. Porous plugs having accurately known conductances in the range $5 \times 10^{-7} - 5 \times 10^{-6}$ ℓ/sec for hydrogen have been used, so that by using inlet pressures of a few Torr, flow rates between 10^{-4} and 10^{-7} Torr litres/sec can be determined. The gas inlet system can be isolated from the target chamber by means of a 5 cm bakeable valve.

3. ION BEAM SOURCE AND OPTICS

The R.F. ion source is based on the designs of Thonemann[6], Moak et al[7], and Barnfield et al[8]. The beam is extracted through an aluminium canal 2 mm. diameter and 1 cm long. Typical operating conditions are:

Extraction Potential	5 kV
Discharge current	2.5 mA
Total Extracted current	1.0 mA
Total R.F. power output	250 watts
Frequency	25 Mc/s

The R.F. power is provided by a simple self excited oscillator using an 813 valve. Power for the source supplies is provided via a 1 kVA, 50 kV isolating transformer. A diagram of the source and accelerating sections is shown schematically in Fig.3.

The physical design used by earlier workers has been modified in order to make the source fully bakeable (Fig.4). The source bottle (1) was made of kodial glass sealed directly to a kovar tube (2), which was in turn welded to a kovar flange (3). The silica disc (4) which shields the canal is as used in ref. (8) and is located in a step ground in the open end of the bottle. The stainless steel bellows (5) locates the aluminium

canal (6) against the silica disc and takes up the differential expansion when baking. The shield (7) was included to eliminate sparking from the plasma past the silica disc by working in a lower range of the Paschen curve.

Hydrogen is introduced from a silver-palladium diffusion thimble via a small hole in the source flange. It diffuses in past the silica disc and is pumped out of the source via the canal. The palladium leak assembly is made from two kovar cups (8) separated by a glass insulator. The palladium silver tube (9) is brazed into it with a nickel gold braze and supported at one end with a flexible silver lead (10) which carries the current used to heat the tube and takes up the expansion due to heating. Hydrogen flows through the assembly via the Kovar tubes (11).

Accelerating and Focusing System

The two main focusing elements in the system are an accelerating gap and a unipotential lens (Fig.3). As the focal length of the accelerating gap changes with applied voltage the focal length of the unipotential lens[9] is also changed to refocus the beam on the exit hole of the primary focusing system. Above a certain voltage the accelerating lens becomes too strong. This effect has been lessened by accelerating in two stages allowing with the present arrangement focusing to be achieved up to an energy of 30 keV.

After focusing by the unipotential lens the beam may be deflected by electrostatic deflection plates to align it with the first diaphragm. This enables compensation to be made for inaccuracies of mechanical alignment and the effect of stray fields.

Analysing Magnet

From the first diaphragm the beam diverges as it enters the sector magnetic field used for selecting the particular ion mass required.

The design was based on the theory due to Cross[10]. By allowing the beam to enter the magnetic field at an angle with respect to the normal of the pole face boundary, the beam is deflected towards the median plane thus providing focusing both in the plane of the pole face and at right angles to it. This increases the total transmission of the beam and reduces the amount of gas produced by loss of beam in the analyser tube. An asymmetric design was chosen with small ℓ^1 (see Fig.5) (which provides a high transmission for a given magnet gap) and a large ℓ^{11} to allow plenty of room for differential pumping between magnet and target chamber, and to allow room for a quadrupole electrostatic lens to correct the astigmatism due to the magnet focusing. The design parameters (Fig.5) chosen are

$$\ell^1/R = 1 \qquad \varepsilon_1 = 57^o$$

$$\ell^{11}/R = 4 \qquad \varepsilon_2 = 34^o\ 30'$$

$$R = 20 \text{ cm.} \quad \Phi = 45^o$$

The magnetic field allows 30 keV ions of up to mass 16 to be focused.

The magnet itself was mounted on a trolley which allowed it to be withdrawn while the vacuum system was baked and also allowed for adjustments of the angle of the sector in both horizontal and vertical planes. Small adjustments were also allowed by bellows

connecting the analyser tube to the primary focusing system and to the target chamber end.

The operation of the analyser was checked first by observation of the image produced by the fluorescence of the beam on a glass plate. An approximately circular image was obtained 2-4 cm in diameter. The adjustment of the magnet was not critical within a few degrees but the focusing of the einzle lens and the beam centering adjustment were critical especially to get a maximum transmission. The transmission was measured by having a moveable Faraday cup system which could be swung immediately in front of the entrance aperture to the magnet thus collecting the total ion beam entering the analyser tube. The ion currents due to the separated components emerging from the magnet were collected by a second Faraday cup system. The transmission was defined as the sum of the H_1^+ H_2^+ H_3^+ ions emerging from the magnet as a percentage of the total beam entering. This varied from 40% at 10 keV to 70% at 30 keV total beam energy. The increase in transmission at high energies is due to the changing focal length of the accelerating lens and the resulting decrease in beam divergence from the first aperture. Other ions from the source appearing at masses 18 and 28 were considerably less than 1% of the total beam current.

Quadrupole Lens

Because of the large magnification of the magnetic field focusing arrangement, a short focal length lens was used to focus the beam to a small spot in order to get a large current density. In order to compensate for the astigmatism of the magnetic field a double electrostatic quadrupole lens pair similar to that described by Barnfield et al[8] was used. The beam can be focussed to a spot approximately 2mm dia. at 30 keV. With the maximum current available of 300 μA of H_1^+ ions this provides a current density of > 5 mA/cm^2.

4. TARGET MOUNTING ASSEMBLY

In order to allow a number of targets to be put successively into the beam a target turntable was built with six target positions (Fig.6). A metal-ceramic bearing combination was used in order to overcome the problem of friction in ultra high vacuum without employing a lubricant which might contaminate the surfaces under investigation. The turntable was mounted on a molybdenum shaft which turned in boron nitride bearings. Initially a polished silica bearing was used but after a short period of satisfactory operation the bearing gradually seized up due to powdering of the silica. Boron nitride was found to be a much more satisfactory bearing material. It has an expansion coefficient close to molybdenum and has good vacuum properties at high temperatures. It also has a graphite-like structure giving a low coefficient of friction even in vacuum. Bearings of boron nitride normally last several months of daily use before excessive play necessitates new bearings. The turntable is rotated by a "wobble stick" mounted in a stainless steel bellows which paddles it around. Holes for this purpose are drilled in the circumference of the turntable at 10^o intervals.

The targets themselves normally consist of foils 2 × 1.25 × 0.025 cm which are supported by molybdenum split tubes on the end of 1.0 mm dia. molybdenum rods. The rods are positioned by small blocks which are electrically insulated from the turntable by ceramic spacers (Fig.6). The targets can be heated by electron bombardment from a tungsten

filament mounted immediately behind them which allows refractory metals to be heated to ~ 2000°K. One target position is normally occupied by a SnO-coated pyrex plate which is used as a screen for focusing the beam. Provision is made for mounting auxiliary electrodes and the usual precautions are taken to suppress electrons in the ion beam and secondary electrons produced at the target when measuring ion currents.

The target chamber is constructed with a 28.5 cm O.D. flange on top, from which the target turntable and all other equipment associated with any particular experiment may be mounted before being put in the vacuum system. On this flange are also mounted an ion gauge and a mass filter with a nude ion source, which acts as a residual gas analyser and a detector for the products of the ion surface reactions.

5. PERFORMANCE

Experimental work so far undertaken has been concentrated on the measurement of re-emission or reflection of ion beams from surfaces. When a target is bombarded the pressure rise in the surrounding volume is a measure of the number of ions and neutrals re-emitted from the surface. For total pressure rise measurements the target is surrounded by a small chamber connected to the outer target chamber by a known conductance, and the yield of gas molecules per incident ion has been measured as a function of energy for a number of metal targets[11]. To demonstrate the capabilities of the apparatus the results of using a high current beam are shown (Fig.7), in which molybdenum is bombarded with 220 μA of H_1^+ ions at 30 keV. The target is heated by the beam to a temperature of ~ 900°C, and the initial burst of gas is due to thermal desorption of hydrogen acquired since the target had last been cleaned. Experiments on thermal release of trapped ions[12] indicate that with molybdenum saturation occurs at ~ 10^{17} ions/cm^2 so that under the above conditions close to 100% of the incident ion beam is released as gas. It is notable that even with this gas load the pressure in the outer chamber rises to only 6×10^{-10} Torr (nitrogen equivalent). By removing the inner chamber, targets can thus be maintained at pressures of $< 10^{-9}$ Torr while under ion bombardment. This means that experiments on sputtering or the reflection of ions and neutrals from surfaces by direct particle techniques, can be undertaken with the very favourable ratio of beam to background gas bombardment of > 1000 : 1. In the present experimental arrangement the high pumping speed of the sublimation pump minimizes gas flow into the inner chamber and thus keeps the pumping speed of the orifice effectively constant.

Typical data obtained from the mass filter is shown in Fig.8. A10 keV H_1^+ ion beam of 30 μA was allowed to bombard an undegassed copper surface and the gas desorbed by the beam was mass analysed. The scanning speed was 0.4 amu/msec. and as shown in Fig.8 the main gas species produced as result of the bombardment were CO_2 , CO and CH_4 . After 15 seconds the height of the hydrogen peak has increased until it corresponds to ~ 100% re-emission of the incident ions. Similar experiments on thoroughly degassed molybdenum targets show impurities less than 1% of the hydrogen partial pressure.

ACKNOWLEDGEMENTS

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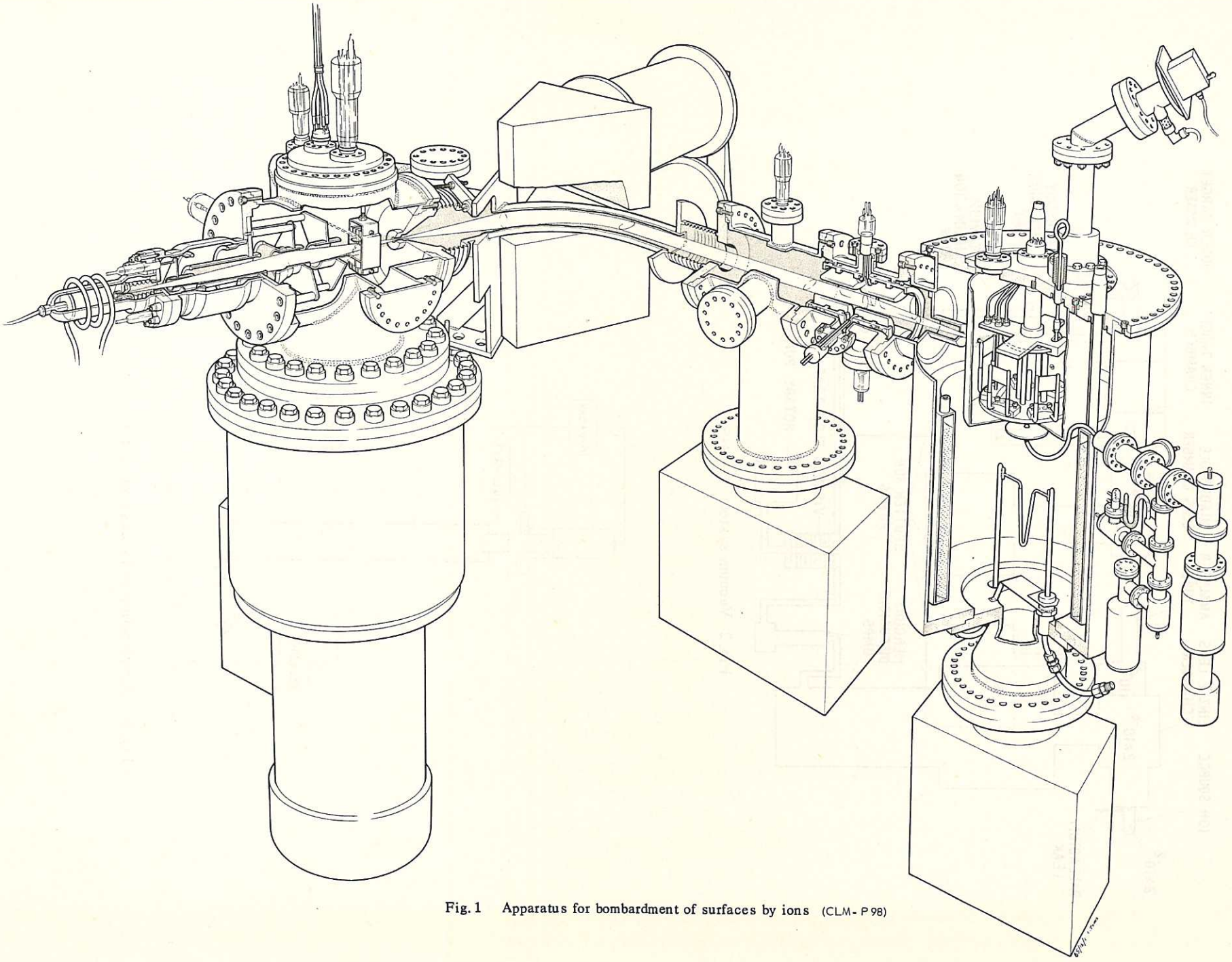

Fig. 1 Apparatus for bombardment of surfaces by ions (CLM-P 98)

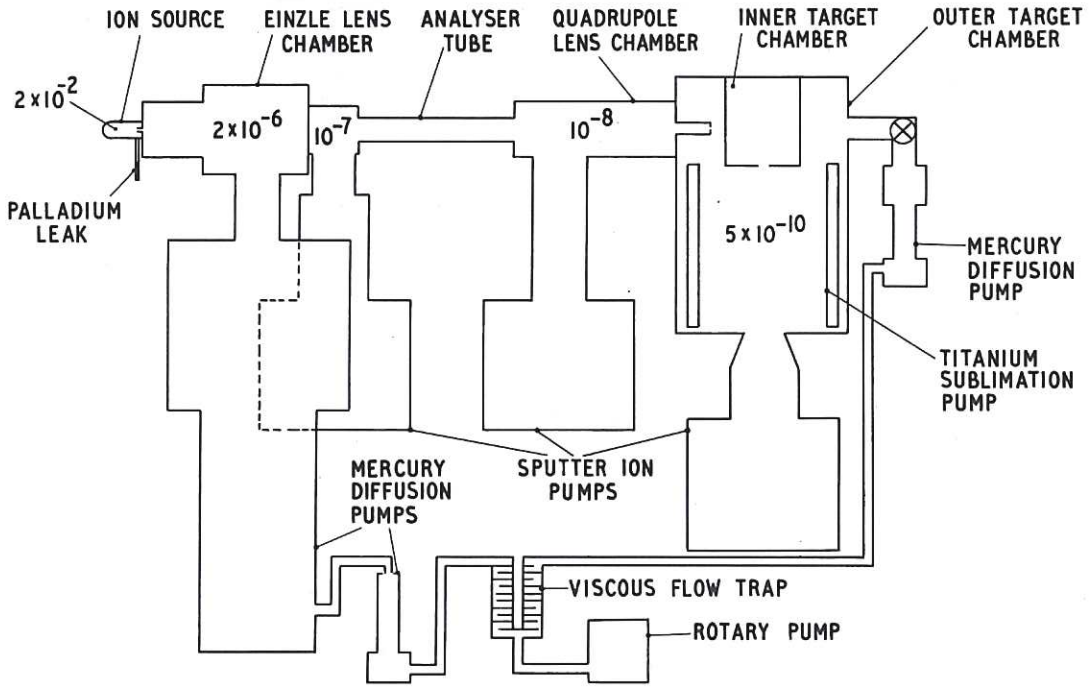

Fig. 2 Vacuum system (CLM-P 98)

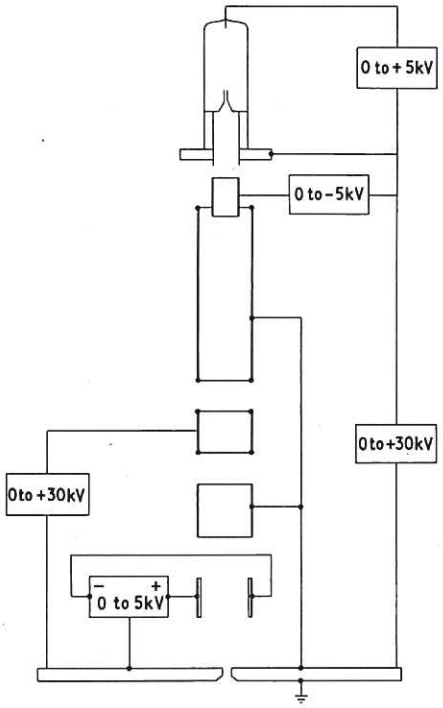

Fig. 3 Accelerating and focussing section (CLM-P 98)

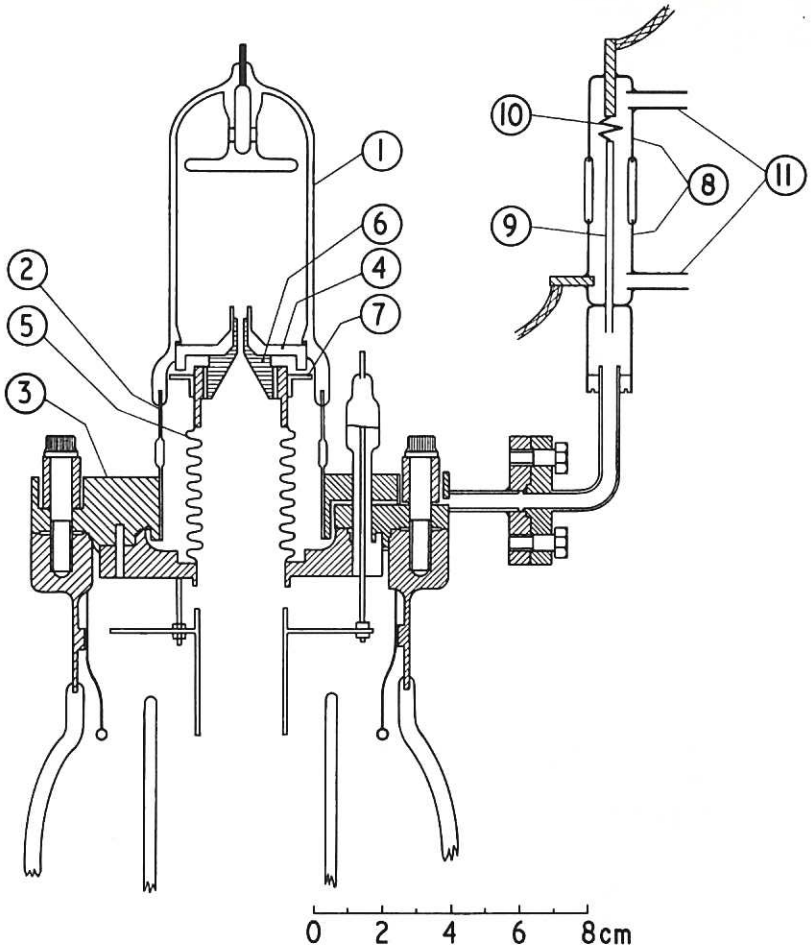

Fig. 4 Ion source and palladium leak (CLM-P98)

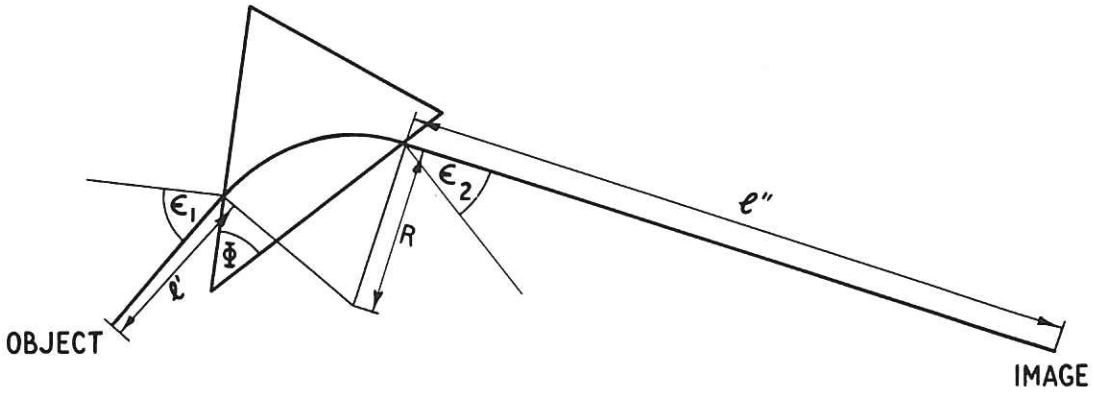

Fig. 5 Magnetic sector field design (CLM-P98)

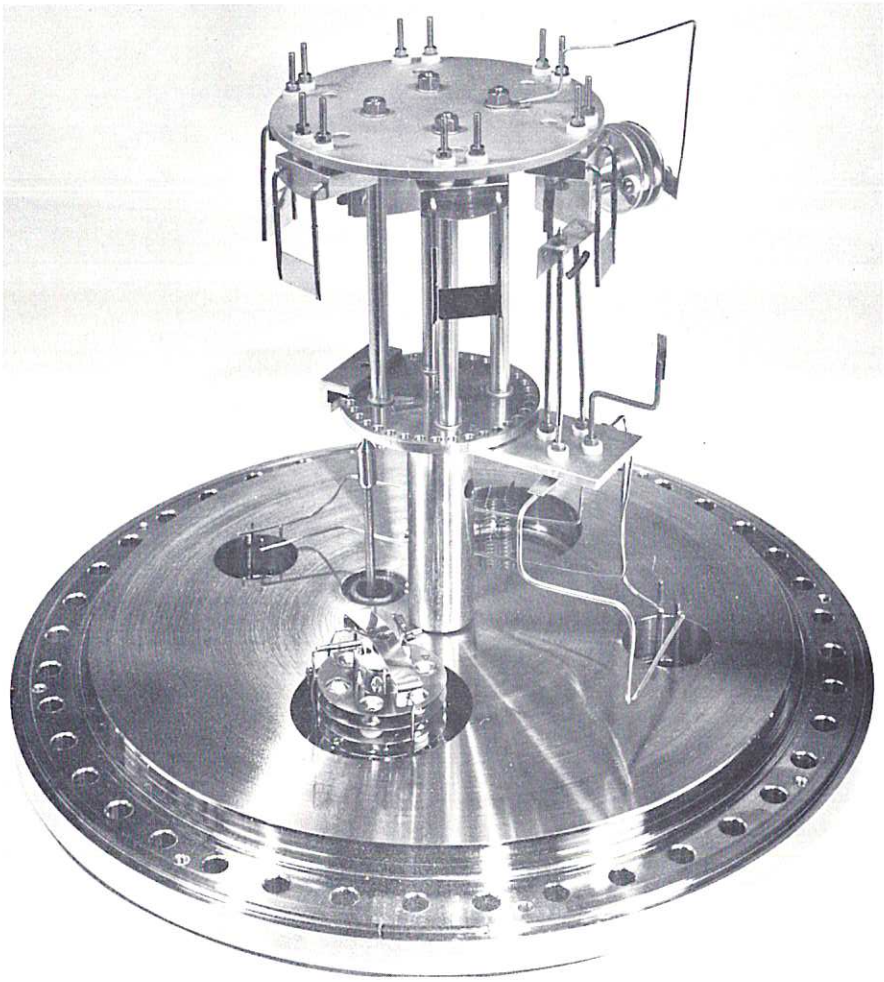

Fig. 6 Target turntable mounted on flange (CLM-P98)

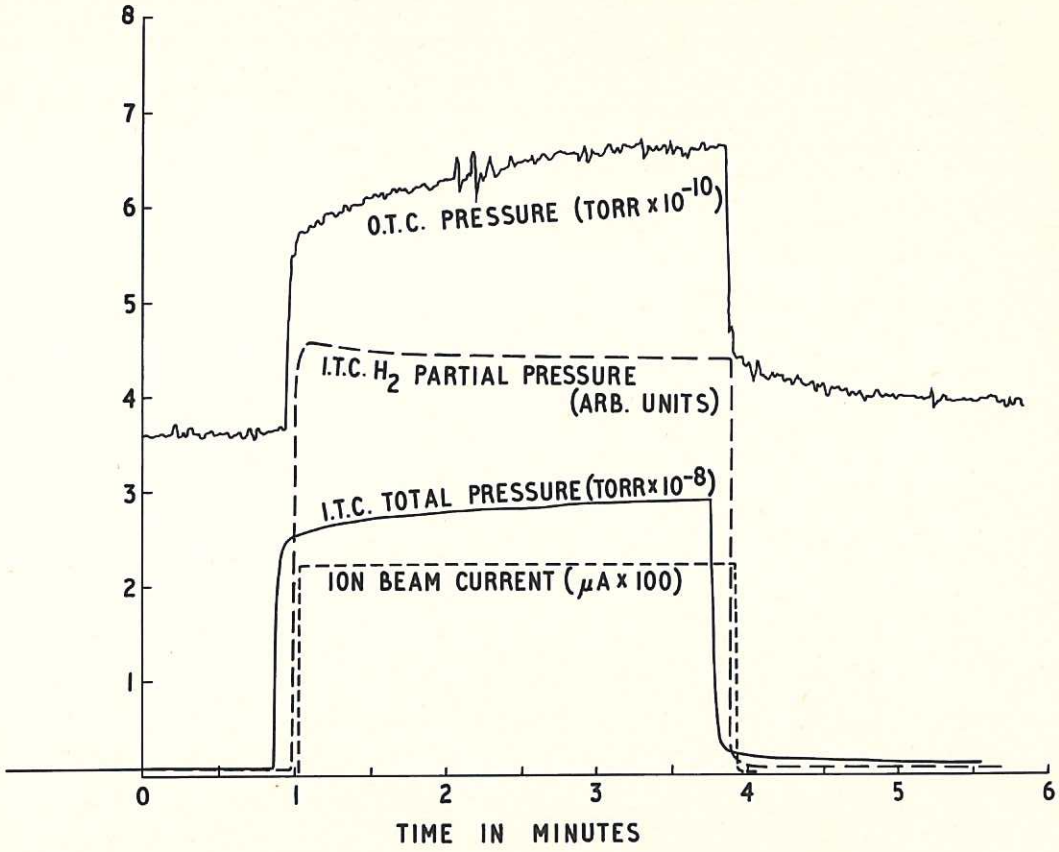

Fig. 7 (CLM-P 98)

Pressure measurements during bombardment of a molybdenum surface by a 30 keV H_1^+ ion beam. Pressures are nitrogen equivalent. O.T.C. : Outer target chamber. I.T.C. : Inner target chamber

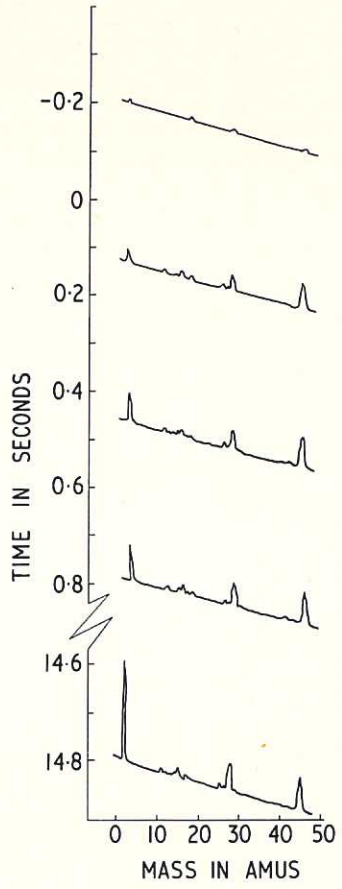

Fig. 8 (CLM-P 98)

Mass spectra using fast scanning quadrupole mass filter