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A modified electron bombardment type ion source for the study of solids

S V GHAISAS, S M CHAUDHARI, M R BHIDAY, V N BHORASKAR
Department of Physics, University of Poona, Poona 411 007, India

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Abstract. A modified electron bombardment type ion source suitable for use with mass spectrometer is described. Ion formation occurs throughout a relatively large volume in the ionisation box, since no magnetic field is used to collimate the ionising electrons. A sensitivity of 2×10^{-5} amp/torr is obtained for an ion extraction energy of 2 keV and 200 mass resolution. Trajectory tracing has been used to study the operation of the ion source. Capability of the ion source to analyse solid samples in microgram quantity was tested by studying evaporation of BaO from tungsten.

Keywords. Electron bombardment; ion source; ion current; sensitivity; mass spectrometer.

1. Introduction

Mass spectrometer is an important tool for analysing samples in microgram quantities. For this purpose it is necessary to choose an efficient ion source or a detector. An attempt to increase the detector efficiency often retards the signal-to-noise ratio and critical vacuum conditions are to be satisfied when an electron multiplier type detector is employed. A majority of electron bombardment type ion sources presently in use are not efficient because of the number of small slits used to collimate the ion beam, which excludes perhaps 90% of the ions produced in the ionisation chamber.

Different types of high transmission ion sources have been developed for mass spectrometers (Giese 1959; Kinzer and Carr 1959; Melton 1966) but these ion sources have rather complex structure. The present paper describes the design and operation of a modified electron bombardment type ion source to study sample in microgram quantity. This ion source is simple in design and rigid in construction and has a high extraction efficiency, with minimum sacrifice in the resolution of the mass spectrometer.

2. Constructional details

The ion source fabricated out of nonmagnetic stainless steel is shown schematically in figure 1. The whole assembly is mounted on four rigid rods fixed on a 12·7 mm thick stainless steel base flange. The ionisation box (between the plates B_1, B_2)

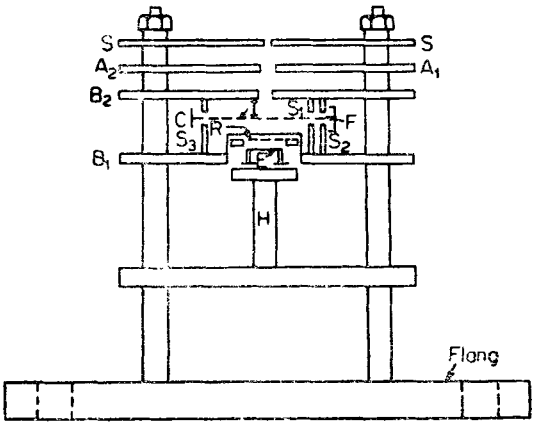

Figure 1. Ion source for the study of evaporation of solids.

has internal dimensions 1·6 cm (long), 1·5 cm (broad) and 1 cm (high). On one side of this box a tungsten filament F, with its glass mount is fitted. The electrons emitted from this filament are accelerated and focussed by an assembly of two slits S_1 (0·4 mm) and S_2 (1·0 mm). At an accelerating voltage of about 100 V, the electron current (trap current) reaching the collector C is about 120 microampere, which is about 15% of the total emission. The trap current is stabilised by an electronic circuit similar to the one described by Caldecourt (1951).

The ion repeller R in the form of stainless steel mesh (made from 0·1 mm wire with mesh hole size 0·01 mm^2) of dimension 1·8 cm long and 1·6 cm broad is kept 1 mm outside the ionisation box. Its purpose is to keep away thermal positive ions from entering into the ionisation box during evaporation of solid sample and also to increase the efficiency of ion extraction. The side of the ionisation box facing the ion repeller is kept sufficiently open. The assembly E, having arrangements for heating the solid sample and temperature measurement, is kept just 2 mm below the ion repeller. The ion extraction system consists of two half plates A_1—A_2 and the source slit S. The two half plates A_1, A_2 are fixed at 2 mm distance above the ionisation box and a further 2 mm above these plates the source slit S(0·2 mm) is fitted. The slit S is kept permanently at ground potential, allowing easy way to extract ions upto 2 keV. To avoid mass discrimination (Coggeshall 1962), no magnetic field is used to collimate the ionising electrons. By removing the ion repeller, the system can be used as a thermal ion source.

3. Results and discussion

A 90° sector mass spectrometer with 23 cm mean radius was used to study the characteristics of the ion source. A mass spectrum of residual gases at pressure 5×10^{-7} torr in the ion source was recorded with a Faraday cup (with 0·5 mm slit), coupled to an electrometer. A portion of the spectrum upto mass number 44 is shown in figure 2. The resolution of the mass spectrometer obtained was 200 at 5% peak height. The potentials required to obtain maximum ion current for any mass value, at a given pressure, are shown in table 1.

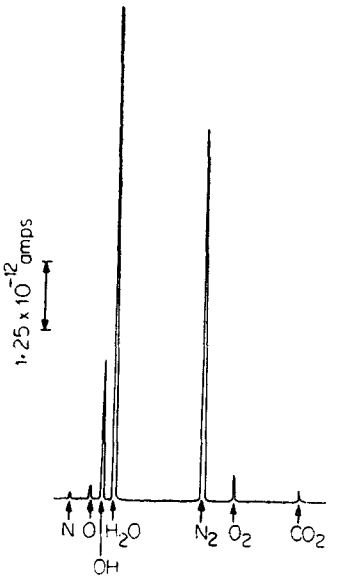

Figure 2. Mass spectrum of air.

Table 1. Relative potentials (volts)

Ionisation box (B_1, B_2)	V_0
Ion repeller (R)	V_0+6
Half plates (A_1, A_2)	$V_0/2$
Slit (S)	Zero (ground potential)

To determine the sensitivity of the ion source, a vacuum $\approx 10^{-7}$ torr was obtained in the mass spectrometer system and the ion source was flushed with nitrogen gas. Trap current was adjusted to 120 microampere at a nitrogen gas pressure of 2×10^{-5} torr in the ion source. The recorded ion current for N_2 ions was 4×10^{-10} amp. This shows that the ion source sensitivity is 2×10^{-5} amp/torr and is about twenty times greater than the conventional ion source under similar conditions (Fusafumi 1970). This relatively high sensitivity results from following considerations:

(i) The electron beam (ionisation region) is very near the ion extraction slit, increasing the ion extraction efficiency (Werner 1974).

(ii) Extraction slit of the ionisation box and half plates are sufficiently wide. This allows large fraction of the ions to be focussed at the final slit S (Melton 1966).

(iii) The potential difference between half plates and extraction slit is 1000 V. In this situation the extraction region increases (Werner 1974).

Further, on the basis of the ion source geometry and the relative voltages (table 1) paraxial trajectories are plotted as shown in figure 3. It shows that the ions are focussed at the source slit S with reduction in magnification (≈ 10). This supports the fact that this particular design furnishes a higher ion extraction efficiency.

Variation in the ion current (sensitivity) with repeller and accelerating voltages is also studied and the results are shown in figures 4 and 5 respectively. Figure 4 shows

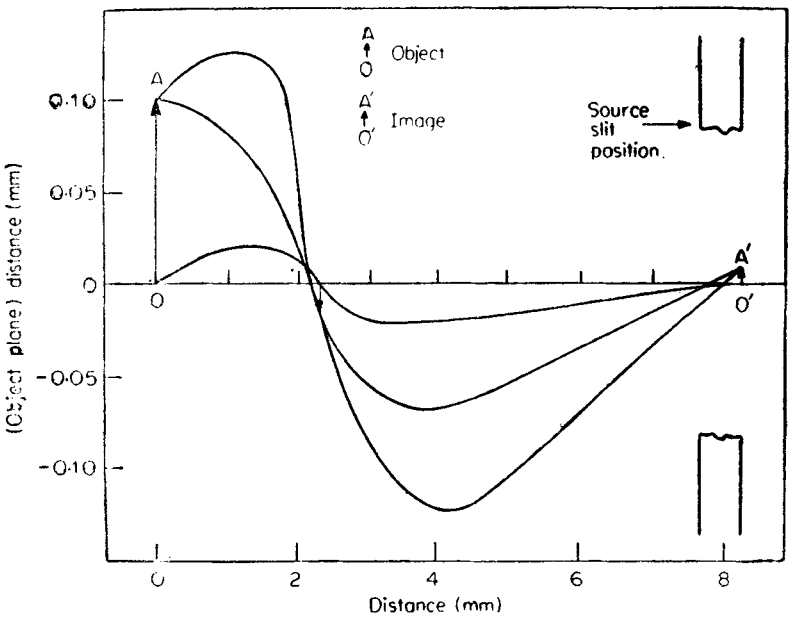

Figure 3. Ion optics of ion source.

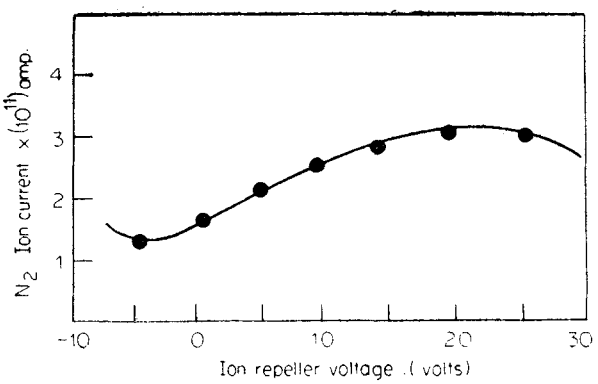

Figure 4. Ion source characteristics for constant accelerating voltage (2 kV).

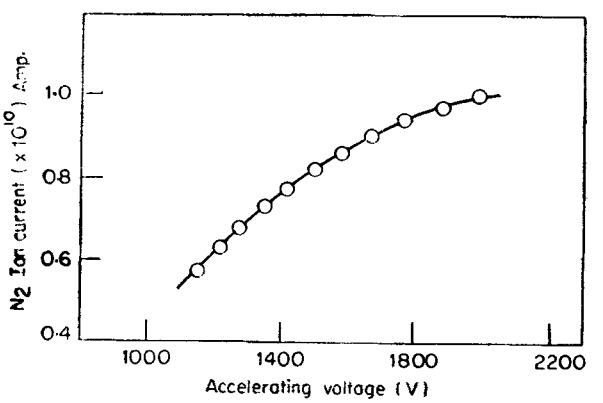

Figure 5. Ion source characteristics for constant repeller voltage (6 V).

that the ion current increases with repeller voltage, however, it results in the reduction of resolution. A proper compromise between the ion current (sensitivity) and the resolution was obtained using 6 V repeller voltage. This provided 2×10^{-5} amp/torr sensitivity at 200 mass resolution. For the same order of sensitivity, other workers (Fusafumi Nakao 1970; Giese 1959; Melton 1966) had obtained mass resolution in the range of 60 to 150. Figure 4 further shows that even for a negative repeller voltage, the ion current does exist at a measurable level and is due to the penetration of the field from the half plates A_1—A_2 in the ionisation box. Figure 5 shows that the ion current (sensitivity) increases with increase in the accelerating voltage as expected. The maximum extraction voltage used in the present work was 2 kV. A higher value of extraction voltage often causes sparking between the electrodes, particularly at relatively high gas pressure and the operation becomes unstable.

In order to check the utility of this ion source to study solid samples an experiment was conducted. About 10 μg of BaO was coated on the tungsten filament fitted to the support H and evaporated by increasing temperature of the filament. The temperature was measured using a four-probe method (Jones and Langmuir 1927). The variation of Ba ion current with temperature is shown in figure 6. After about 1870° K the Ba ion current decreases because of depletion of the sample. To demonstrate its utility as a thermal ion source 40 μg of $Sr(NO_3)_2$ was evaporated from tungsten filament. The thermal Sr ion current of about 4×10^{-12} amp was obtained for about 15 min.

4. Conclusion

The measurements to study the performance of the modified electron bombardment type ion source have been carried out satisfactorily. The measured value of the sensitivity of the ion source was 2×10^{-5} amp/torr for N_2 at 200 resolution of the mass spectrometer. The ion source is simple in construction and useful for the analysis of solid samples in microgram quantity.

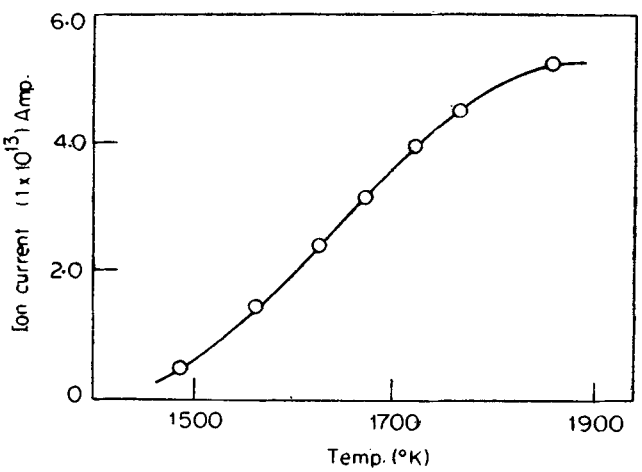

Figure 6. Evaporation of Ba from BaO.

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