DESIGN OF A LARGE, HIGH RESOLUTION ION MICROPROBE[†]

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Abstract

An ion microprobe intended for the analysis of natural silicates requires both high mass resolution, to separate interferences in the complex secondary ion spectra, and high sensitivity to enable detection of trace elements and accurate measurement of isotope ratios. An instrument designed to have a sensitivity comparable to existing low-resolution machines while operating with a resolution of $M/\Delta M = 10^4$ is now under construction in the Research School of Earth Sciences in Canberra. In order to satisfy the above criteria a large instrument, having a magnet radius of 1m, is considered necessary. Fabrication of the component parts is now approaching completion and the assembly and testing of the system is expected to begin shortly.

1. Introduction

Preliminary plans to construct an ion microprobe at the Australian National University in Canberra were presented at the SIMS Workshop held at Gaithersburg in 1974. At that time it was recognised that for chemical and isotopic studies of geological specimens both high sensitivity and high mass resolution would be wanted. The stated design goal was to combine a resolution of 10,000 with sensitivity comparable with that of existing low resolution ion microprobes. It was demonstrated that in order to achieve this combined objective the instrument should have as large a secondary mass analyser as possible. Since that meeting work on SHRIMP (Sensitive High Resolution Ion MicroProbe) has progressed to the point where fabrication of many of the components has been completed and assembly has begun. It is now considered appropriate to present a more detailed report on the design of the new ion microprobe.

In the description which follows, the instrument is considered as made up of a number of parts or systems, each having a particular function; the primary column, the secondary extraction system, the sample handling system, the secondary analyser and the magnet control and data handling system. We shall begin by considering the primary column.

2. The Primary Ion Source and Focusing System

A commercial duoplasmatron (General Ionex Model 358) capable of generating both negative and positive ions was chosen as the primary ion source and will be modified to operate with a hollow cathode discharge in place of the standard filament. An extraction gap and power supply provided up to 30KV accelerating potential for the primary beam are also included.

The primary focusing system consists of two condenser lenses, each intended to operate with a magnification of 1/5, giving a magnification of 1/25 for the whole system. When the duoplasmatron is operated with a 0.1 mm aperture it is reasonable to expect an apparent source spot 0.025 to 0.050 mm in diameter to act as object for the system [1]. Neglecting aberrations, this would provide a demagnified primary spot approximately 0.001 to 0.002 mm in diameter at the target. A symmetric tube type einzel lens has been used for the first condenser lens. The second or final condenser lens is an integral part of the secondary extraction geometry and, as such, is discussed further in that section. Although the unusual form of this lens prevents an accurate estimate of the coefficient of spherical aberration, rough calculations suggest that, with apertures limiting aberrations to a few microns microns, primary beam currents of the order of 10^{-10} to 10^{-9} A can be achieved.

No attempt has been made in the initial design to incorporate mass separation of the primary beam but it is intended that a Wien filter be added at a later date.

Differential pumping of the primary column is provided by a pair of 400 l/s triode ion pumps suitable for pumping both active and inert gases. It is intended to operate initially with oxygen and bombard with O^- but other primary species may be substituted.

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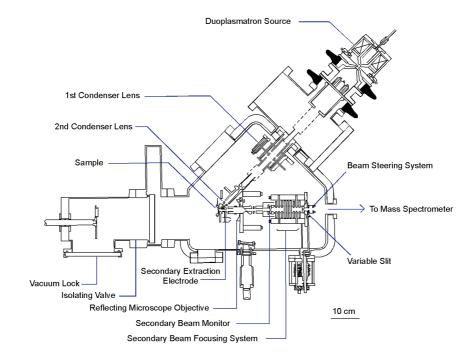
Figure 1. Schematic of SHRIMP source chamber and primary column.

3. The Secondary Extraction and Focusing System

The function of this system is to transmit the largest possible fraction of the sputtered ions from the specimen surface into the secondary mass analyser. The design cannot, however, be considered in isolation from that of the final primary beam condenser lens nor of the arrangement for viewing the sample during bombardment.

Consider first the relation between the incident primary beam, the direction of extraction of secondary ions and the sample surface. It is desirable for the secondary beam to travel along the normal to the sample surface in order to minimize energy discrimination and because this should correspond to the mean axis of emission. At the same time it would be advantageous for the primary beam to strike the surface from a direction as close as possible to the perpendicular. Liebl [2] has described an arrangement permitting simultaneous bombardment and extraction along the normal to the surface provided that the two beams have opposite polarity and that the energy of the secondary ions is much less than that of the primary beam. This approach has not been followed here because it was felt that high extraction potentials (up to 25 KV) would increase extraction efficiency and that, in view of the variety of applications possible for this instrument, the option of using any combination of positive or negative primary and secondary beams should be retained. Hence a compromise solution has been adopted in which extraction is normal to the sample surface while primary bombardment is at an angle of 45°. Figure 1 illustrates the layout of the source region.

The electrode configuration for the final primary condenser lens combines a short working distance (20 mm) with a large bore to reduce spherical aberration. In addition, the form of the einzel lens has been made asymmetric with the smallest (6 mm) bore diameter nearest the target. It has been shown [1] that the spherical aberration coefficient for an asymmetric lens is smaller than for a comparable symmetric lens.

The proposed application of this instrument to geological analyses leads to the need for observation of the sample surface under high magnification, preferably using either transmitted or reflected light. This requirement has been satisfied by the use of a Schwarzchild type aplanatic, anastigmatic objective [3] consisting of two spherical mirrors (Figure 2) and described by the following parameters:
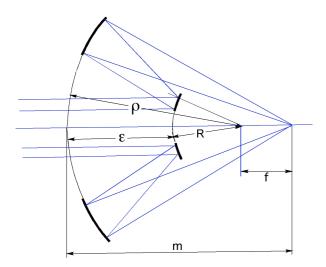
$$f = 17.5 \ mm$$

$$m = \left(2 + \sqrt{5}\right)f = 74.13 \ mm$$

$$\varepsilon = 2f = 35.0 \ mm$$

$$\rho = \left(1 + \sqrt{5}\right)f = 56.63 \ mm$$

$$R = \left(\sqrt{5} - 1\right)f = 21.63 \ mm$$

Analysis of the function of the secondary extraction and focussing system may be done in terms of the phase pace concepts of beam transport theory [4-6]. As Ruedenauer [7] has pointed out, a comparison of estimated secondary beam emittance with instrument acceptance suggests that existing instruments are transmitting only a small fraction of the sputtered ions. The design of the system described here has therefore been carried out within the framework of beam transport theory. It should be noted, however, that the above estimates of beam emittance [7] are almost

Figure 2. Schwarzchild lens system for SHRIMP reflected-light viewing optics.

certainly optimistic in two respects. First, the charge distribution within the secondary emittance is unlikely to be uniform but will depend on both angle of secondary emission and intensity of primary bombardment and, secondly, the spherical aberration in the initial stages of extraction leads to a non-linear distortion [8] which increases the effective emittance area. In view of these comments the phase space calculations have only been carried out in an approximate form.

As will be discussed more fully below, the secondary mass analyser has an acceptance in the XY plane (the plane of deflection in the analyser) of

$$A_{max} = 8.8 \times 10^{-4} \text{ mm-rad}$$

This is defined by the analyser entrance slit width of .044 mm and divergence half-angle of 0.01r, for a theoretical mass resolution of 10,000. Since these figures assume perfect double focussing, it was felt that an entrance slit width of .03 mm was more realistic. Assuming a primary beam diameter of 0.01 mm, therefore, a magnification of 3× is needed in the extraction and focussing system. This system (figure 3) consists of three sections; the extraction system, the intermediate lens and the phase space matching system.

The first of these, the extraction system, may be thought of as a pair of two electrode tube lenses [9]. Simple ray tracing indicates that if these are adjusted so that the sample lies in the front focal plane of the first lens, then an image will be formed in the back focal plane of the second (point A). The magnification will be approximately 1× and the emittance will be approximately rectangular. The intermediate lens serves simply to transfer the beam again with unit magnification to form another image at point B. The beam matching system is made up of three symmetrical slit einzel lenses [10], two active in the XY plane and one in the XZ plane. Having two lenses provides a variable magnification in the XY plane, from 3× to 1/3×, for use with different primary spot diameters. A single lens is considered sufficient in the XZ plane since the acceptance of the secondary analyser is larger in this case. To first order, the phase space transformations produced by the intermediate lens and the matching system should leave the emittance approximately rectangular. Figure 4 shows a theoretically possible first order matching between beam emittance and instrument acceptance in both YY' and ZZ' planes assuming a 0.01 mm primary spot, 25 kV accelerating potential and a 0.03 mm analyser entrance slit width. The emittances are shown as having indefinite extent in the direction corresponding to the angle of emission from the sample, since there is no theoretical upper limit to the transverse energy. However, the density of particles drops off in the indicated direction, since dN/dE decreases with increasing energy beyond the limits defined by the acceptance. It is possible to calculate the transverse energy corresponding to this theoretical cut-of since, to first order, the emittance defined by all particles having transverse energy less than E_T is

$$\varepsilon_y = 2d\sqrt{\frac{E_T}{E_a}}$$

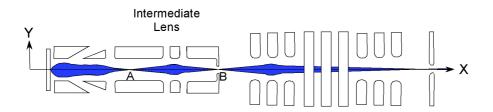where d is the diameter of the primary spot (0.01 mm) and E_a is the energy of the beam after acceleration (25 KeV).

Figure 3. Secondary-ion extraction system. A and B indicate focal positions of the lens system. X and Y show co-ordinate system.
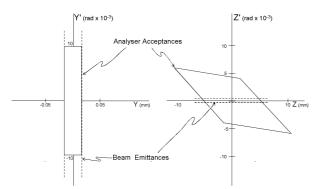
Figure 4. Emittance of secondary ions from source chamber and acceptance of mass analyser.

If the emittance is limited by the acceptance as shown in Figure 4 then

$$2d\sqrt{\frac{E_T}{E_a}} = A_y = 6 \times 10^{-4} \, mm - rad$$

or

$$E_r = E_a \left(\frac{A_y}{2d}\right)^2 \text{ and } E_T = 22.5 \ eV$$

The above figure represents the maximum initial transverse energy, which an ideal matching system would transmit and it is recognised that aberrations will limit the extraction efficiency actually achieved. Computer simulation of potentials and trajectory plotting demonstrates, as might be expected, that the dominant effect will occur in the first stages of extraction, where the ions are moving slowly and at large angles to the ion optical axis. Perhaps further intensive work on the problem will reveal a better arrangement although, as mentioned above, the electrode shapes are severely constrained by the primary condenser and the microscope objective. In order to facilitate ion optical experimentation the first two extraction electrodes can be removed through the vacuum lock. This also enables them to be cleaned more readily.

4. Sample Handling System

Samples can be inserted through a vacuum lock, with one sample mount inside the source region while another is pumping down in the lock. Each mount can accommodate a single specimen up to 25 mm in diameter, or possibly several smaller ones.

Translational motion along all three axes has been provided; up to 3 mm in the X direction (normal to the sample surface) and 25 mm in the Y and Z directions. These transverse movements are driven by micrometers fitted with position encoders giving a digital read–out of position accurate to ± 0.002 mm.

5. Secondary Analyser

A double focussing mass spectrometer is necessary to achieve high mass resolution in the analysis of the secondary ions. The layout chosen (Figure 5) is one given by Matsuda [11], using a combination of a cylindrical electrostatic analyser, an electrostatic quadrupole lens and a homogeneous magnetic field to eliminate any significant second order aberrations. As given in table 5 of reference 11, the parameters defining the geometry of the analyser are shown in Table 1.

Table 1 Ion optical parameters

$l'_e = 1.290$	$d_{eq} = 0.15$	$r_m = 1.0$
$\varnothing_e = 85°$	$L = 0.22$	$\varepsilon' = 15°$
$r_e = 1.272$	$K_o = 1.91$	$\varepsilon'' = 0$
$Q_1 = -4.0$	$d_{qm} = 1.129$	$l''_m = 0.894$
$Q_2 = 0$		

The performance of this design has been simulated using a computer program by Kaiser [12], modified to include electrostatic quadrupole lenses. This program includes fringing field integrals to second order only, whereas Matsuda's calculations include third order integrals. This difference is not serious, as the simulation confirms Matsuda's conclusions. The exact values of the integrals are likely to differ slightly from those assumed by Matsuda in any case, since slight modifications to design parameters have been made (for example, the magnet gap width is 0.04 r_m, instead of 0.06 r_m). By adjusting parameters such as K_o, d_{qm}, 1'$_e$, 1"$_m$ it should be possible to achieve double focussing with only a small contribution to beam width due to aberrations sine the aberration coefficients do not change rapidly with small variations in the geometry of the instrument [11]. In order to combine high mass resolution with sensitivity, the dimensions of the machine have been made as large as was considered practical. The radius of curvature of the magnet was therefore set at 1000 mm, with other dimensions in proportion. This gives a theoretical resolution of MΔ/M = 10,000 with the source entrance slit set at 0.044 mm.

A feature of this mass spectrometer is the existence of an intermediate image between the electrostatic analyser, which permits the sharp definition of energy limits. The advantage of such an arrangement has been pointed out by Steele et al. [13]. A slit with a width variable from 0 to 6 mm (corresponding to an energy band width of 150 eV in 25 KeV) has been provided at this image point.

The analyser entrance and collector slits are adjustable from 0 to 2 mm and can be rotated about the beam axis to permit correction of misalignment of slits or beam. In addition, collimating slits limit the half angle of divergence of the beam entering the analyser to a maximum of 0.01 rad.
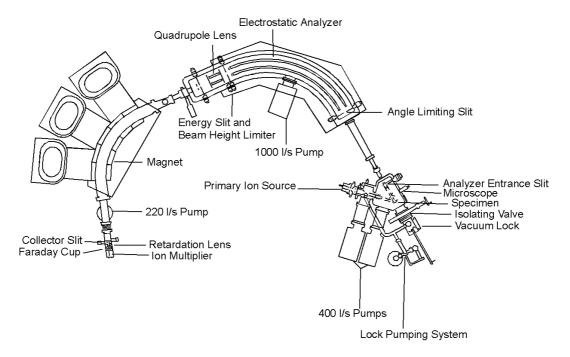
Figure 5. Schematic of SHRIMP layout.

The mechanical design of SHRIMP is such that the magnet is fixed while metal bellows in the vacuum housing allow the positions of electrostatic analyser, the source and the collector to be adjusted. For this purpose the collector, in particular, has been mounted on a carriage permitting translation in two directions plus rotation in the horizontal plane about the collector slit.

Because rapid, precise, switching from one peak top to another is considered necessary either for isotopic measurements or for measuring the abundances of a large number of trace elements, special attention has been paid to the design of the analyser magnet. The yoke segments have been laminated from 12 mm low carbon steel and the polepieces from 2 mm soft ion sheet. Purcell filter gaps separate the polepieces from the yokes. Extra insulation has been applied to the coil windings to prevent damage during switching transients. Field sensing is by Hall effect probe (modified Varian Fieldial) and the magnet switching is controlled by an on-line computer. Manual control is available as an alternative. It is expected that full response to a field step of 10% will be achieved in approximately one second, despite the large mass of the magnet (approx. 7 tons). Details of the system will be published separately.

Two modes of ion detection have been provided in the collector, a Faraday cup and an ion multiplier. The Faraday cup has been included because large beams may be obtained, particularly when the instrument is being operated in a low resolution mode with very wide slits. Located on the beam path in front of the ion counter, the Faraday cup has a sliding bottom, which can be retracted to allow the beam to pass into the ion counter, a Balzers SEV 117.

An additional feature of the collector is the incorporation of a retardation lens for the suppression of low energy scattered ions [6,14,15]. Such suppression is of particular importance in SHRIMP because of the long beam path and the multiplicity of species, which would lead to severe problems of 'trails' from a major component interfering with a trace element or a low-abundance isotope. A design based on that of Kaiser and Stevens [15] was selected as most suitable for use with high voltages.

6. Magnet Control and Data Collection

A Hewlett-Packard 2116B computer is on-line to SHRIMP to control the magnet switching and data collection as well as initial data reduction. The computer is interfaced to a Princeton 801 graphic display terminal and Tektronix 4632 video hard copy unit for operator control and data display purposes. Development of the software is in the hands of Mr D. Kerr of the Bureau of Mineral Resources in Canberra.

7. Conclusions

Construction is now well advanced and it is expected that SHRIMP will soon be in operation and that it should provide a powerful analytical tool in the field of earth sciences. It is presently envisaged that the initial applications will be primarily isotopic but study of the problems of elemental abundance analysis, particularly for trace components in silicates, will be included. An instrument with the potential capabilities of SHRIMP should have important contributions to

make to the understanding of the processes involved in sputtering and secondary ion formation.

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