

BEAM DEVELOPMENTS FOR THE HARWELL MICROPROBE SYSTEM

P.M. READ, J.A. COOKSON and G.D. ALTON *

Nuclear Physics Division, Bldg 477, Harwell Laboratory, Harwell, Oxon, OX11 0RA, UK

A consequence of the rapid development of micron and submicron size electronic devices is the diminished applicability of high energy ion microprobes with their present resolution limitations to the study of such components. Although submicron beams have been reported the available beam current is barely sufficiently for PIXE and is not adequate for RBS. This lack of lateral resolution is due to low beam brightness at the microprobe object and aberrations in the focussing elements. As part of a program to address these problems the Harwell microprobe lens has been relocated on a new 5 MV Laddertron accelerator. The increased brightness and improved stability of this facility has so far led to a reduction in beam size from $3 \times 3 \mu\text{m}^2$ to about $2 \times 2 \mu\text{m}^2$.

The feasibility of using a liquid metal ion source has been examined with a view to achieving more substantial increases in brightness. While such sources have brightness approximately 10^5 times greater than conventional gaseous sources the highly divergent nature of the beam presents problems for the beam transport system. The use of a liquid metal source on the accelerator has been successfully demonstrated but it indicates the need for a special low aberration injection lens if brightness is to be maintained.

1. Introduction

Since the development of the Harwell microprobe [1] in 1970 the facility has been extensively used for studies in nuclear science, metallurgy, biology, geology and microelectronics. During this period a beam with dimensions $3 \times 3 \mu\text{m}^2$ and current of 300 pA has been available for proton induced X-ray emission (PIXE) scans. The lower cross-section techniques of Rutherford backscattering spectrometry (RBS) and nuclear reaction analysis (NRA) have typically used rather larger spot sizes with currents of a few nA. In order to increase the capabilities of the microprobe, particularly in relation to the development of micron and submicron size electronic devices, smaller and brighter beam spots are desirable. Attempts to improve lateral resolution, by either decreasing the object slit size or the angular acceptance of the focussing lens, result in a reduction in beam current to a level such that scan times are impractical. This problem is due to a lack of beam brightness at the microprobe object, which is attributable to the intrinsic properties of the ion source, aberrations in the accelerator's various optical elements and the voltage fluctuations of the accelerator. Given sufficient brightness the object slits can be closed down to reduce the first order image size and a modest reduction in the angular acceptance of the microprobe lens can ensure that spherical aberrations, which depend upon the cube of angle, do not dominate. As chromatic aberrations depend only linearly on angular acceptance they would

then be likely to dominate the spot size unless the beam energy spread could be reduced below present values.

A route which could lead to significant improvements in resolution in such facilities is through the use of high brightness sources such as the proton field ionization (FI) source [2] or the liquid metal ion source [3] (LMIS). These sources have reported brightnesses [4,5] of up to 10^5 times greater than the rf ion source currently in use on this facility. However, it is not clear that advantage can be taken of this improved brightness on a high energy microprobe. In the first instance a source must operate in the hostile environment of a high voltage terminal and provide a reliable beam with a reasonable lifetime. Secondly, the accelerator must be able to transmit the high brightness beam to the microprobe system without dramatic degradation through aberrations.

The FI source originally suffered from lifetimes of only about 10 h due to deformation of the field emitter tip by backstreaming negative ions. More recently the use of iridium emitters has increased this lifetime to in excess of 60 h. However, the source output is limited to about 20 nA [5] and attempts to increase it have led to destructive discharges from the emitter tip. This current would be useable in a microprobe provided it were not greatly reduced by angular acceptance defining apertures in the accelerator and that the accelerator could adequately stabilise on the small current. In contrast the LMIS has lifetimes in excess of 1000 h and has the capability to produce tens of μA of beam current. As the source material is metal it does not produce a gas load so that pumping in the high voltage terminal is not necessary. A disadvantage of the source is that the beam

* On temporary attachment from, Physics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.

V. PIXE/MICROPROBES

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

EMB

is highly divergent and will require careful manipulation. Furthermore LMISs are most widely proven for heavy metals such as gallium, iridium and gold which are of little value for PIXE, RBS or NRA. A lithium source, which would fill our RBS requirements, part of the PIXE requirement and permit NRA of hydrogen using the $^1\text{H}(^6\text{Li}, ^3\text{He})^4\text{He}$, $^1\text{H}(^7\text{Li}, ^4\text{He})^4\text{He}$ or $^1\text{H}(^7\text{Li}, \gamma)^8\text{Be}$ reaction, has been reported [6] but because of the metal's chemical activity requires development.

In the present paper we describe progress on a three stage programme of microprobe development at Harwell. The first stage consisted of the relocation of the Harwell microprobe lens from an old 3 MV Van de Graaff to a recently commissioned 5 MV Laddertron accelerator [7]. This move allowed us to take advantage of the improved brightness and high stability of the Laddertron. Stage two is a tuning exercise with an objective of achieving the present state of the art $1\ \mu\text{m}$ spot with a current of 100 pA [8,9], which is mainly used for PIXE analysis of biological specimens. The last stage aims towards submicron beams for RBS and NRA and is presently concentrating on the possible potential of a LMIS.

2. The Laddertron microprobe facility

2.1. Accelerator

The Laddertron is a vertical accelerator with the high voltage terminal supported on four insulating legs constructed from borosilicate glass and stainless steel electrodes. Voltage grading down the column and accelerator tube is provided by metal oxide resistors which zig zag from one equipotential plane to another. The total column resistance of 4.5×10^{10} leads to a high voltage terminal time constant CR of 7 s. Charging is provided by a Laddertron which is constructed from stainless steel rungs and insulating PETP beads. The maximum charging current used to date is 240 μA at a terminal voltage of 5 MV. Power for terminal supplies and controls is provided by an alternator driven by a rotary shaft. The accelerator is stabilised with a conventional feed back loop comprised of a corona probe and either a generating voltmeter or pickoff slits.

A schematic of beam transport in the microprobe facility is shown in fig. 1. Ions are generated in an rf source and typically extracted from a 1.5 mm \varnothing canal at 2 keV. The beam is accelerated to about 20 keV prior to focussing at the gap lens whose function is to match the injection energy to the tube voltage gradient and hence keep the focal length of the tube constant for different terminal voltages. After emerging from the accelerator tube the beam passes round a 90° analysing magnet and is focussed onto the stabiliser slits. First order ion optics of the accelerator calculated using

OPTRYK [10] are shown in fig. 2.

Immediately downstream of the stabilizer slits are a set of steering coils and then the microprobe object slits. A further set of slits defines the angular acceptance of the microprobe lens. This lens, seen in fig. 3, consists of four magnetic quadrupoles of geometric length 180.5 mm, pole gap 38.4 mm and separation 45 mm. The quadrupoles may be excited separately or in coupled modes. The image distance is 210 mm and the object distance, depending upon the number of quadrupoles used, varies from 5785 to 6235 mm.

An estimate of the proton beam brightness at 3 MeV was made using the microprobe object and angular acceptance limiting slits. The object slits defined a 0.5 mm square source and the acceptance slits a divergence half angle of 0.23 mrad. The transmitted beam of 500 nA has a brightness, given by $B = I/(\pi^2 E^2 V)$, where I is current, E is the two dimensional emittance and V is beam energy, of $3\ \mu\text{A}/(\text{mm}^2\ \text{mrad}^2\ \text{MeV})$. Beam current intensity on target has a low frequency peak to peak variation at the 10% level and a 400 Hz ripple of about 5%. The latter is due to the effect of alternator ripple on the ion source extract power supply. The current stability is dramatically better than the old 3 MV accelerator and is quite satisfactory for microprobe applications. The improved dc stability is probably the single most important factor in the threefold improvement in brightness from the value of $1\ \mu\text{A}/\text{mm}^2\ \text{mrad}^2\ \text{MeV}$ obtained with the 3 MV accelerator.

Beam energy spread has been measured using the well-known $^{27}\text{Al}(p, \gamma)^{28}\text{Si}$ resonance at 991.9 keV. The target was a 800 \AA thick freshly evaporated aluminium film on tantalum backing. Beam energy was scanned through the resonance by fixing the accelerator energy slightly above the resonance and applying a variable positive bias to the target. Gamma rays were detected in a sodium iodide detector, amplified and passed through a discriminator with a lower level set at 1.4 MeV. A computer controlled CAMAC system determined the shape of the excitation function averaged over several voltage cycles. Shortly after commissioning the Laddertron the energy spread was measured to be 590 eV fwhm. More recently the power supply defining the energy of the beam injected into the accelerator has been replaced by a high stability supply. The corona needles which had a tip radius of 150 μm have been replaced with needles of tip radius 30 μm . It is known that the blunter needles tend to spark because of the higher electric field required to deliver the same emission current and this can give unstable operation [11]. Since these changes the beam energy spread has been measured to be 440 eV fwhm (see fig. 4). This represents a 40% reduction in energy spread compared to the 3 MV accelerator. In microprobe operation the beam would be more tightly collimated and its energy spread should be lower than the above value.

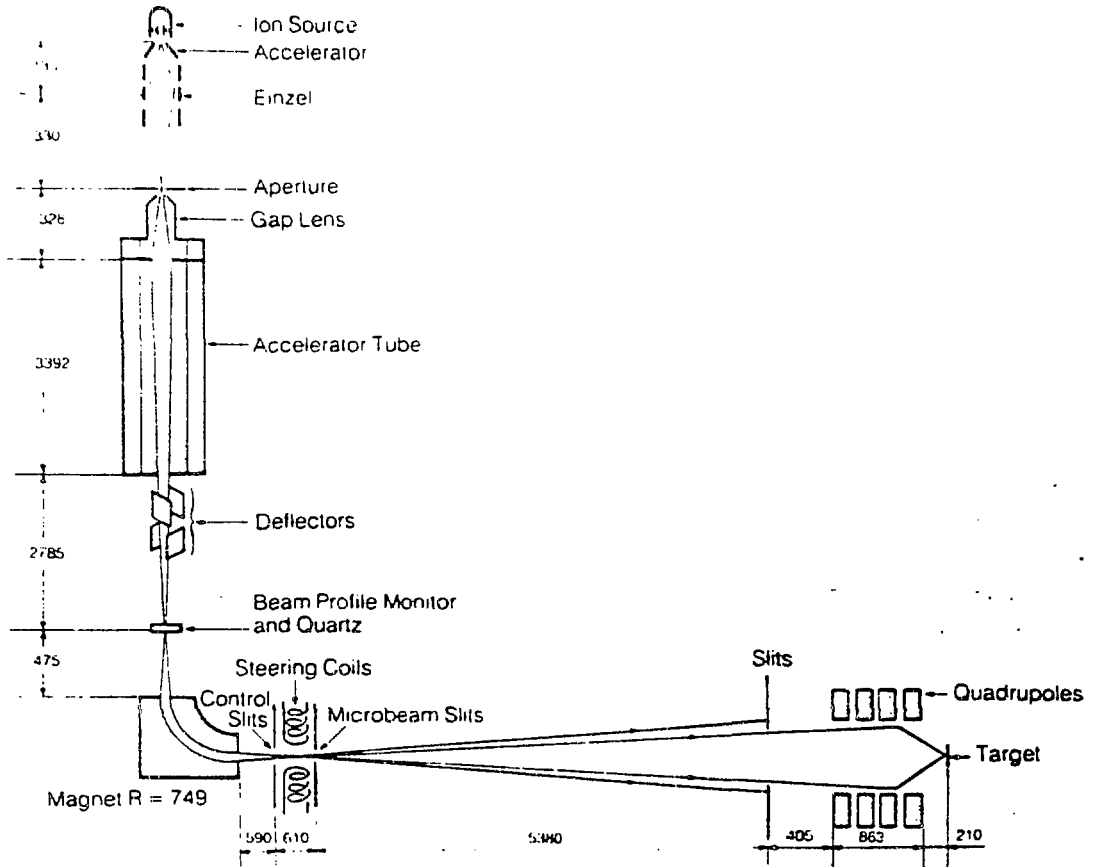


Fig. 1. A schematic diagram of the Laddertron accelerator and microprobe facility.

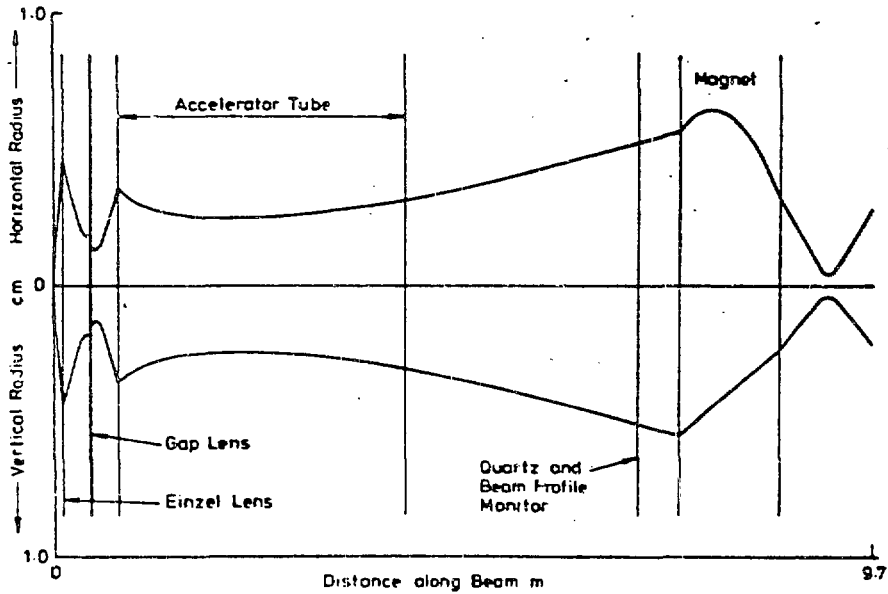


Fig. 2. The first order ion optics of the accelerator at a terminal voltage of 4 MV. The ion source is of the rf type, providing a beam at 20 keV with a virtual source radius of 0.75 mm and half angle of 33 mrad.

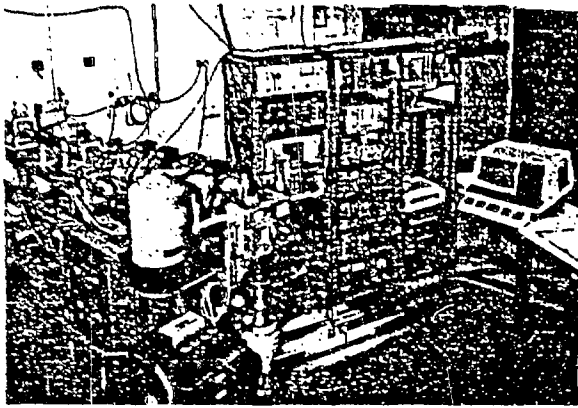


Fig. 3. A photograph of the microprobe facility showing the four quadrupole lens, scattering chamber and control and data acquisition electronics. The angular acceptance limiting slits can be seen slightly upstream of the lens.

2.2. Microprobe image

The first, second and third order imaging properties of the microprobe lens have been calculated using the data of Grime and Watt [12]. They calculate aberration coefficients by numerically solving the equations of motion for a representative set of particles in the beam passing through the quadrupole field and tabulate the results for a variety of lens configurations. The configurations we have considered are: the Russian quadruplet used in the original Harwell microprobe [1] where

Table 1

Parameters of the microprobe arranged in quadruplet, triplet and doublet modes. A quadrupole is described as converging (C) if an ion moving in the horizontal plane is deflected towards the axis and diverging (D) if the particle is deflected away from the axis. Spatial and angular dimensions for the aberrations are in μm and mrad respectively.

	Quadruplet CDCD	Triplet CDC	Doublet CD
Image distance	210 mm	210 mm	210 mm
Object distance	5785 mm	6016 mm	6235 mm
Vertical demagnification	-9.76	-19.7	-43.3
Horizontal demagnification	-9.76	70.0	-6.18
Chromatic aberration coefficients			
$\langle x \theta\delta\rangle$	10.28	-35.16	15.17
$\langle y \phi\delta\rangle$	16.07	101.3	13.16
Spherical aberration coefficients $\times 10^{-3}$			
$\langle x \theta^3\rangle$	-6.97	193	-5.97
$\langle y \phi^3\rangle$	-24.6	-1343	-30.0
$\langle x \theta\phi^2\rangle$	-35.2	348	-64.3
$\langle y \phi\theta^2\rangle$	-33.0	-1290	-9.29

the first and last quadrupoles, and the inner two quadrupoles are excited equally; the Oxford coupled triplet [8] which has the first two quadrupoles equally excited; and the simple doublet. Demagnifications and aberration coefficients are presented in table 1. The

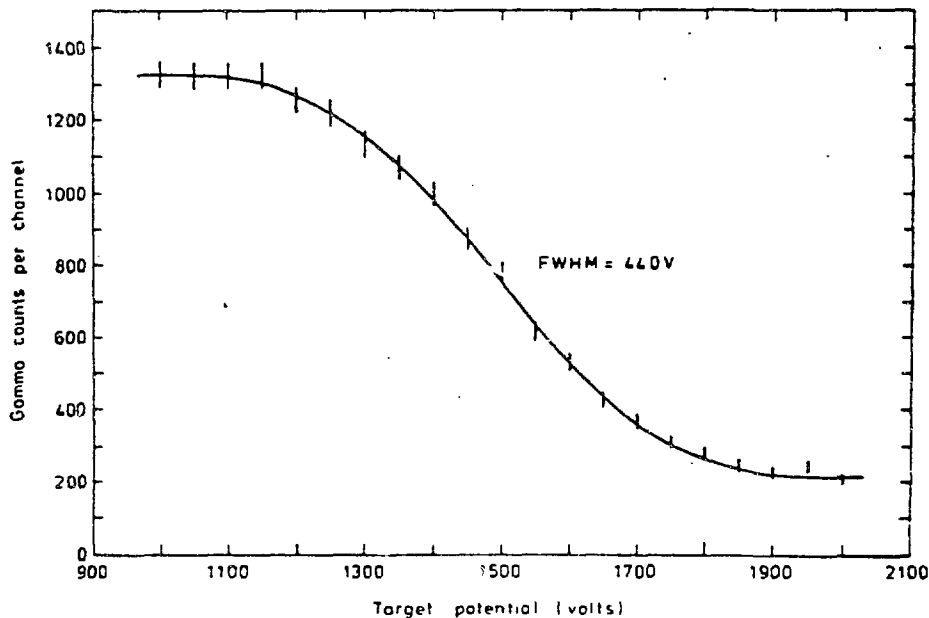


Fig. 4. The energy spread of a proton beam from the accelerator was measured by scanning the beam energy over the $^{27}\text{Al}(p, \gamma)^{28}\text{Si}$ resonance at 991.9 keV. The fwhm energy spread is 440 eV.

Table 2

Contributions to a $1 \mu\text{m}^2$ spot due to first order imaging and chromatic and spherical aberrations. The fractional momentum spread is taken to be $\pm 1.2 \times 10^{-4}$.

	Quadruplet	Triplet	Doublet
Vertical object aperture y (μm)	6.25	12.6	27.7
Horizontal object aperture x (μm)	6.25	44.8	3.95
Vertical divergence ϕ (mrad)	± 0.24	± 0.028	± 0.18
Horizontal divergence ϕ (mrad)	± 0.14	± 0.08	± 0.15
First order image Δx (μm)	0.64	0.64	0.64
Δy (μm)	0.64	0.64	0.64
Chromatic aberrations $2\Delta x_{\text{ch}}$ (μm)	0.59	0.67	0.55
$2\Delta y_{\text{ch}}$ (μm)	0.54	0.68	0.57
Spherical aberrations $2\Delta x_{\text{sph}}$ (μm)	0.52	0.24	0.67
$2\Delta y_{\text{sph}}$ (μm)	0.67	0.52	0.43
Acceptance ($\mu\text{m}^2 \text{mrad}^2$)	5.3	5.1	11.8

quadruplet has a low demagnification and small beam optical aberrations. The image size will therefore be strongly dependent upon the dimensions and quality of

the object slits. For the triplet, demagnification is high and beam optical aberration coefficients are correspondingly high. Parasitic aberrations arising from misalignments, rotations and harmonic contamination of the quadrupole field will also have large coefficients. The characteristics of the doublet configuration are intermediate between the quadruplet and triplet. Contributions to a $1 \mu\text{m}^2$ image for the three quadrupole configurations described above are presented in table 2. The first order image size was fixed at $0.64 \mu\text{m}$ and the angular acceptance of the lens adjusted to give similar contributions from chromatic and spherical aberrations. Taking the acceptance of each system as a figure of merit we find the value for the doublet to be about double that for either the quadruplet or triplet.

To determine the size of the microprobe beam it was first optimized on a quartz viewed by a $\times 100$ microscope. The beam was then scanned across a $12 \mu\text{m}$ wide strip of platinum on silicon substrate and the intensity of Pt L X-rays observed as a function of scan voltage. In tests so far the doublet gave the best result with a beam spot of $2.2 \times 2.2 \mu\text{m}^2$ and current of 150 pA . Another scan across a $8 \mu\text{m}$ wide aluminium strip on silicon substrate indicated a resolution in one direction of $1.4 \mu\text{m}$ (see fig. 5) but the specimen was unsuitable for measurement in the other direction. A more suitable test specimen is being prepared.

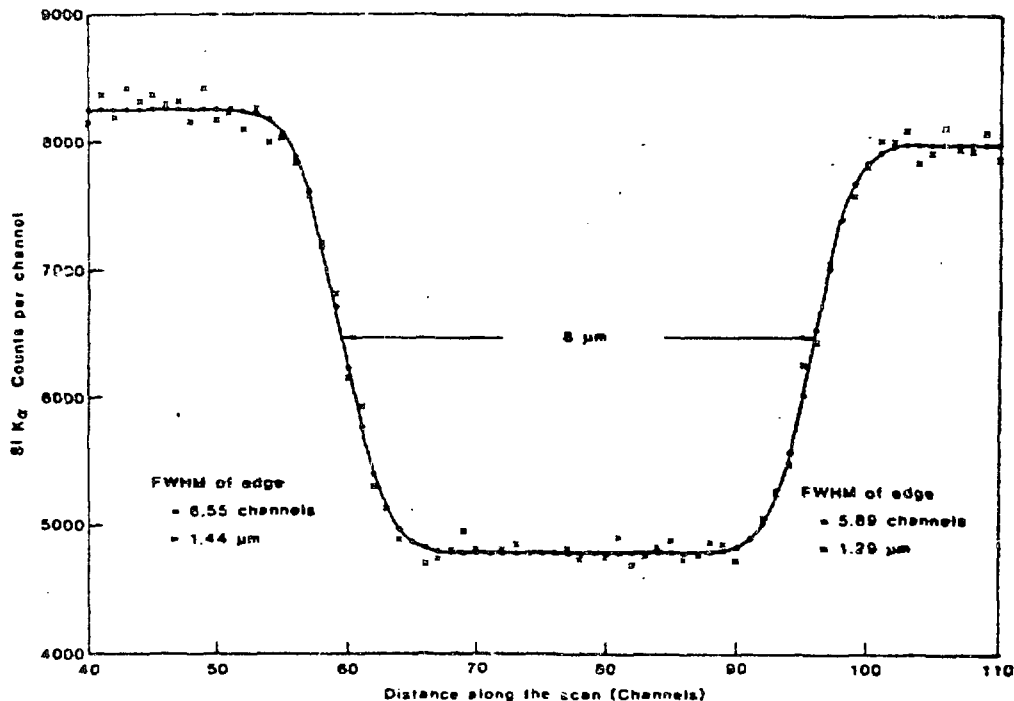


Fig. 5. The silicon K_{α} X-ray yield as a function of scan position across a $8 \mu\text{m}$ wide aluminium strip on silicon substrate; the crosses represent data and the circles fit points.

3. Beam development

3.1. Ion source

This section describes feasibility studies for the use of a LMIS on a high energy accelerator and for ultimately using the beam for microprobe applications. The majority of work has been performed using a gallium source [13] because of its availability from the manufacturers of low energy ion probes. The LMIS consists of a tungsten needle with a tip radius of about $10\ \mu\text{m}$ wetted with gallium and supported in a reservoir tube. It is installed in a source body containing a reservoir heater

and extraction electrode (fig. 6). The heater has been incorporated for subsequent work with lithium but was not used with the gallium source. Ions are extracted from the needle tip by applying between 5 and 6 kV to the extractor. The ion current is controlled by the extraction voltage and the source is stable over long periods with currents at any set level between a few μA and many tens of μA . Immediately after extraction the ions are accelerated to 20 keV and passed through a molybdenum aperture plate to limit the divergence half angle to 150 mrad. A 50 mm diameter gridded einzel lens is located 105 mm from the needle. On the test bench this lens focussed the beam at a set of slits and a

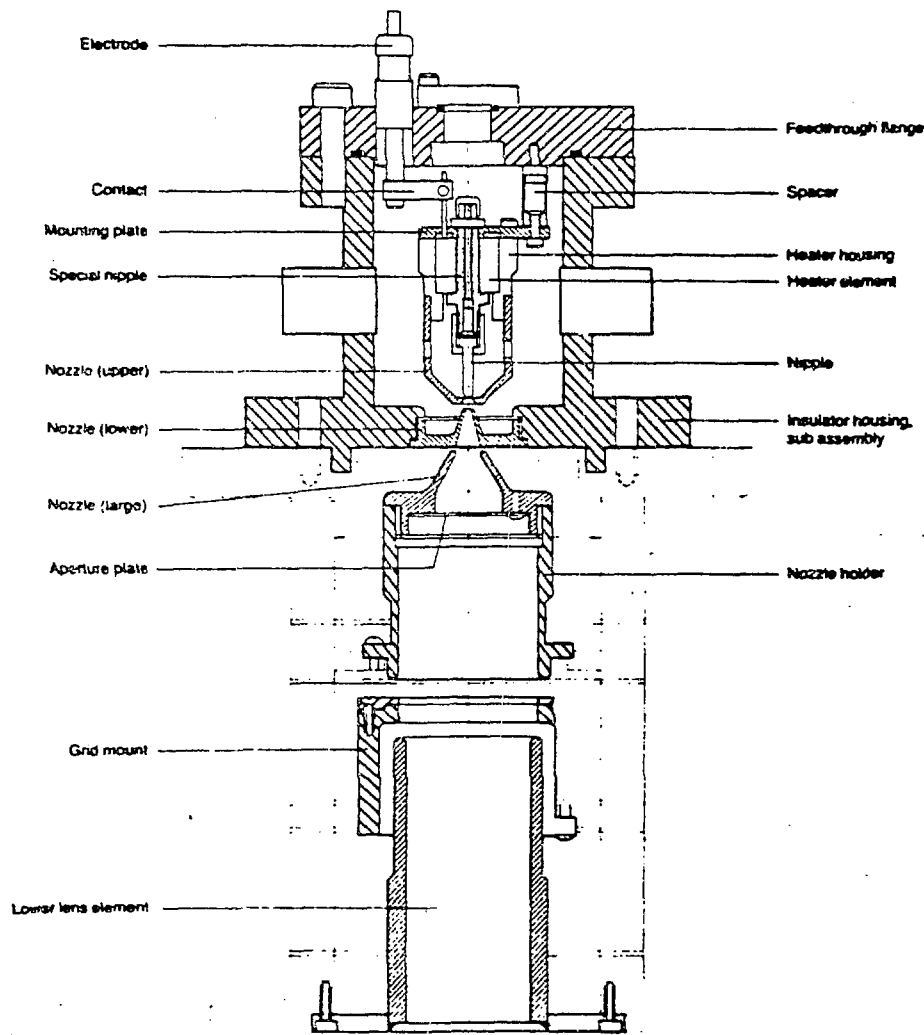


Fig. 6. A schematic of the LMIS housing and focussing lens. The tungsten needle, which is not shown, is located centrally in the nipple and protrudes by approximately 1 mm. In the present studies the nipple was replaced with a commercially available gallium source [13]. The extraction voltage is applied to the needle and the whole source housing sub assembly is floated to the acceleration voltage.

suppressed Faraday cup 350 mm further downstream. With a source current of $45 \mu\text{A}$ the beam passing through a 5 mm square aperture was $2.2 \mu\text{A}$. The theoretical estimates [14] of $0.1 \mu\text{m}$ for the virtual source size indicate that the beam is severely aberrated. To investigate this the acceptance solid angle of the lens was reduced by a factor of four. The beam current passing through the 5 mm square aperture and into the Faraday cup was only marginally reduced to $2.0 \mu\text{A}$. Assuming that at a source output of $45 \mu\text{A}$ the angular intensity is constant up to 150 mrad then in the absence of aberrations the current would be expected to drop by a factor of 4. As this is clearly not the case it indicates that aberrations are a major contribution to the image size. A new focussing system is now being considered with a view to reducing spherical aberration whilst maintaining the beam current at the μA level. The lens should ideally be located close to the needle and have high magnification to reduce aberrations in the accelerator.

A LMIS with the present focussing lens has been installed on the Laddertron accelerator to investigate any transmission or operational problems. The source struck easily and a beam was accelerated to 2 MeV. Observation of the beam emerging from the accelerator indicated it to have a similar size to that from the rf ion source. This is expected because in both cases the beam is an image of the gap lens aperture plate (see fig. 1). Because of the high magnetic rigidity of the beam analysis could only be performed using a 15° magnet with a resolving power of 1 in 50 amu. The combined beam current of the two isotopes of gallium, which were not completely resolved, was $1.7 \mu\text{A}$. Doubly charged gallium was present at 4 nA and contaminant beams of singly and doubly charged oxygen were present at 70 and 10 nA respectively. The magnet did not have sufficient field strength to analyse polyatomic gallium. The results of a secondary ion mass spectrometry (SIMS) depth profile of a test implantation of gallium into aluminium is shown in fig. 7. Implantation was performed at 2 MeV to a dose of 1.4×10^{16} ions cm^{-2} . The mean range is $1.1 \mu\text{m}$.

3.2. Energy spread

The energy spread in the accelerated beam appears at present to be limited by an oscillation in the stabiliser slit differential amplifier. This oscillation may be due to the ion source mechanically vibrating at the alternator frequency of 25 Hz. Whilst such beam movements do not have a dramatic effect on the beam current on target they can alter the small amount of beam being scraped off on the pickup slits by up to 100%. The stabilisation system attempts to correct these beam current changes due to vibration by changing the beam energy and therefore introducing an energy spread. The

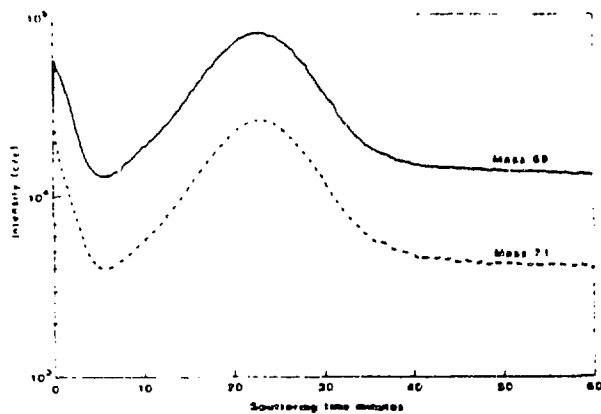


Fig. 7. A SIMS depth profile of a 2 MeV gallium implantation into aluminium. The mean range is $1.1 \mu\text{m}$.

vibration problem is to be addressed by balancing the alternator and its drive shaft and improving the mechanical decoupling of the ion source and accelerator tube from the high voltage terminal and column.

4. Summary

The Harwell microprobe lens has been relocated from an old 3 MV Van de Graaff to a recently commissioned 5 MV Laddertron accelerator. Although the Laddertron uses an rf ion source similar to the Van de Graaff, a threefold increase in brightness in the microprobe line has been achieved. The improvement is due in part to the improved current stability of the Laddertron beam. This brightness improvement, together with a 40% reduction in the energy spread of the accelerated beam, has translated to a spot size of approximately $2 \times 2 \mu\text{m}$ with a current of 150 pA and a best resolution of $1.4 \mu\text{m}$ in one direction. Both these results were obtained with a quadrupole doublet configuration.

Preliminary studies of the feasibility of using a LMIS to increase the brightness of a high energy microprobe have been reported. They indicate that while the beam can be successfully accelerated through the existing optical system the brightness is only comparable to an rf ion source. This degradation of brightness in comparison to reported values is due largely to the spherical aberration of the injector lens. The design of a low aberration lens is being investigated.

The authors are grateful to Dr. P.D. Prewett of IBT Dubilier Limited for providing the gallium LMIS used in these studies and to A.E. Ledbury for carrying out test bench measurements. Work described in this report was undertaken as part of the Underlying Research Programme of the UKAEA.

References

- [1] J.A. Cookson and F.D. Pilling, AERE Report R6300 (1970); J.A. Cookson, A.T.G. Ferguson and F.D. Pilling, *J. Radioanal. Chem.* 12 (1972) 39.
- [2] R. Levi-Setti, *Nucl. Instr. and Meth.* 168 (1980) 139.
- [3] R. Clampitt and D.K. Jefferies, *Nucl. Instr. and Meth.* 149 (1978) 739.
- [4] R.L. Seliger, J.W. Ward, V. Wang and R.L. Kubena, *Appl. Phys. Lett.* 34(5) (1979) 310.
- [5] G.L. Allan, J. Zhu and G.J.F. Legge, *Fourth Aust. Conf. on Nucl. Techs.* (1985) 49.
- [6] R. Clampitt and D.K. Jefferies, *LEIB Salford (1978) Inst. Phys. Conf. Ser.* (1978) no. 38.
- [7] P.M. Read and J.H. Stevens, AERE Report R-11946 (1986); P.M. Read and J.H. Stevens, *Nucl. Instr. and Meth.* A249 (1986) 141.
- [8] F. Watt and G.W. Grime, G.D. Blower and J. Takacs, *IEEE Trans. Nucl. Sci.* NS28 (1981) 1413.
- [9] G.J.F. Legge, D.N. Jarmieson, P.M.J. O'Brien and A.P. Mazzolini, *Nucl. Instr. and Meth.* 197 (1982) 85; G.J.F. Legge, private communication (1985).
- [10] T. Greenway, OPTRYK, Oxford Nuclear Physics Laboratory Internal Report.
- [11] A. Vermeer and B.A. Strasters, *Nucl. Instr. and Meth.* 157 (1978) 427.
- [12] G.W. Grime and F. Watt, *Beam Optics of Quadrupole Probe-forming systems* (Adam Hilger, Bristol, UK, 1984).
- [13] IBT Dubilier Ltd., Abingdon, Oxon, UK.
- [14] J.W. Ward, *J. Vac. Sci. Technol.* B3 (1985) 207.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.